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On alternative cost-effective approach to determining high concentrations of mobile heavy metals (on example of studies of environmental damage from mining and metallurgical waste)

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Abstract. This article discusses a new, simple, and inexpensive method for determining high concentrations of water-soluble forms of heavy metals using a non-destructive chemical analytical method – an X-ray fluorescence analysis. Unlike standard approaches, which employ labor-intensive and expensive acid digestion techniques (inductively coupled plasma atomic emission spectrograhy, inductively coupled plasma mass spectrometry, and atomic absorption spectroscopy) to measure pollutant concentrations in solution (requiring analysis of aqueous extracts), this method studies changes in concentrations in the solid residue of samples on filters at specific time intervals that enables the use of a more cost-effective X-ray fluorescence analysis method. The effectiveness of this method is demonstrated on example of the study of copper and lead migration in man-made waste generated by the combined effects of the mining and metallurgical industries at one of the facilities in the Irkutsk region. The concentrations determined using X-ray fluorescence analysis and inductively coupled plasma atomic emission spectrograhy are compared. The study results are shown to be completely comparable, while the labor intensity and cost of the proposed approach, as well as the qualification requirements for the personnel performing sample preparation and analysis, are significantly lower. The proposed method allows to assess the potential for toxic agent migration under various conditions – from short-term exposure to precipitation to prolonged exposure to water. The visualization results presented enable rapid categorization of samples based on the leaching behavior of water-soluble forms.

Keywords: mobile forms of metals, migration, X-ray fluorescence analysis, inductively coupled plasma atomic emission spectrograhy, mining and metallurgical waste, pollution

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Научная статья
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Об альтернативном экономичном подходе к определению высоких концентраций подвижных форм тяжелых металлов (на примере исследований экологического вреда отходов горно-металлургических предприятий)

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Резюме. В статье рассматривается новая простая и недорогая методика определения высоких концентраций водорастворимых форм тяжелых металлов с помощью неразрушающего химико-аналитического метода – рентгенофлуоресцентного анализа. Особенность методики в том, что, в отличие от стандартных подходов, в которых с помощью трудоемких и дорогостоящих методик с кислотным разложением проб (атомно-эмиссионной спектрометрии с индуктивно-связанной плазмой, масс-спектрометрии с индуктивно-связанной плазмой, атомно-абсорбционной спектрометрии) исследуются концентрации поллютантов, перешедшие в раствор (что требует анализа водных вытяжек), в данном случае изучаются изменения концентраций в твердом остатке проб на фильтрах через

определенные интервалы времени, что позволяет применять для анализа более экономичный метод рентгенофлуоресцентного анализа. Эффективность методики показана на примере исследований миграции меди и свинца в техногенных отходах, сформированных комплексным воздействием горноперерабатывающей и металлургической промышленности на одном из объектов в Иркутской области. Приведено сопоставление определения концентраций с помощью рентгенофлуоресцентного анализа и атомно-эмиссионной спектрометрии с индуктивно-связанной плазмой. Показано, что результаты исследований абсолютно сопоставимы, при этом трудоемкость и стоимость предлагаемого подхода, а также требования к квалификации персонала, выполняющего пробоподготовку и анализ, существенно ниже. Предлагаемый способ позволяет оценить возможность миграции токсикантов в различных условиях – от краткосрочного воздействия атмосферных осадков до продолжительных воздействий воды. Представленные результаты визуализации позволяют быстро категорировать пробы по особенностям протекания процесса вымывания водорастворимых форм.

Ключевые слова: подвижные формы металлов, миграция, рентгенофлуоресцентный анализ, атомно-эмиссионная спектрометрия с индуктивно-связанной плазмой, отходы горно-металлургических предприятий, загрязнение

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Introduction

Currently, the environmental component is becoming increasingly important in the Russian mining industry. This applies both to assessing the background state of the environment in new areas, and to the assessment and remediation of historical waste from mining, metallurgical, and similar industries, which can be considered both as objects of environmental damage and as technogenic deposits. Serious technogenic pollution of environmental components by such facilities is often caused by extremely high concentrations of various toxicants in these components. The high level of responsibility for environmental protection or for the assessment of resources/reserves in such facilities equally requires the use of accurate and reliable analytical methods for chemical analysis. However, it should be noted that the development of the modern analytical base used in the mining and geological industry in recent decades has mainly been aimed at increasing the sensitivity of methods at the lower limit of detection, as this is precisely what is necessary for studying the natural distribution of various chemical elements in the earth's crust, soil, rocks, and so on. Semi-quantitative methods have been replaced by methods such as inductively coupled plasma atomic emission spectrometry (ICP-AES), atomic absorption spectrometry, and mass spectrometry, which make it possible to detect even the smallest concentrations of substances at the level of hundredths of ppm (mg/kg) and to study the corresponding variations in the natural geochemical background in natural objects.

At the same time, when it comes to pollution, technogenically disturbed areas, and especially the direct sources of such hazardous impact in the form of waste and technogenic surface formations, for example, from objects of accumulated damage, there is a need to reliably and accurately measure very high concentrations, hundreds and thousands of times higher than both the geochemical background and the standard values for content. The aforementioned modern methods using acid digestion – ICP-AES or atomic absorption spectrometry – are highly sensitive methods that give good results in low determination ranges, but are quite strictly limited by their upper limit. For example, according to the methodology ERD F 16.2.2:2.3.71-2011[1] for the study of various natural objects by spectral methods, including mobile forms of metals in soil samples, the upper limits for the main heavy metal pollutants are limited to tenths of a percent (only for iron is the upper limit 10 %), even considering significant dilutions of tens or hundreds of times. Such ranges often cover most research

[1] *Environmental Regulatory Document Federal 16.2.2:2.3.71-2011. Quantitative chemical analysis of soils. Methodology for measuring mass fractions of metals in sewage sludge, bottom sediments, and plant samples by spectral methods.* Moscow; 2011, 45 p.

tasks, but only if it does not involve large-scale technogenic pollution of environmental objects with toxicant concentrations already measured in percent. In such situations, significant multiple dilutions of samples are used. This makes it possible to detect the actual content of elements in the sample, but such actions lead to high measurement errors. Thus, it turns out that complex and expensive measurements do not provide either high reliability and information content, nor high efficiency and cost-effectiveness of using these methods.

At the same time, in recent years, significant progress has been observed in the field of non-destructive analysis methods. The non-destructive testing method – X-ray fluorescence analysis (XRF), used to determine the total content of elements, allows working in different modes with solid samples, both with low concentrations of the determined elements ("geochemical modes" are now implemented in the latest generations of X-ray fluorescence spectrometers from most leading manufacturers) [1], and with high concentrations (often referred to as "mining modes"). The XRF method is fast, cost-effective, and does not require complex sample preparation, other than grinding and, in some cases, pressing. At the same time, it is often believed that the XRF method is rather semi-quantitative and does not provide high measurement accuracy. A large number of modern publications demonstrate the capabilities of modern XRF implementations in cases of geochemical/geological exploration, core studies, and other tasks of determining total concentrations, and from these publications it often becomes obvious that for many purposes, methods with acid digestion are no longer needed [2–6].

This work, however, addresses a somewhat different range of issues. In many research and practical tasks, there is a need to determine water-soluble forms of elements, for example, to simulate the leaching of toxic components by atmospheric precipitation [7–9], to model migration into soils [10–11], into plants [12, 13], to study migration into water bodies [14], to solve medical-ecological [15], and many other problems [16]. Furthermore, there is a need to calculate the percentage ratio of water-soluble and poorly soluble forms in order to assess the actual amount of elements migrating into environmental components relative to the total mass of technogenic formations with known total toxicant content. The authors propose a new method for determining water-soluble forms of elements; its effectiveness will be examined in a case study typical for the mining and metallurgical industry – assessing the state of the environment contaminated with a number of typical heavy metals extracted from ores and their processing products from a number of historical mining, metallurgical, and chemical industries.

This work presents an alternative, low-cost option for determining the ratio of water-soluble and poorly soluble forms of heavy metals in natural-technogenic objects, suitable for research work.

The task at hand is to completely switch to a non-destructive testing method for assessing not only total, but also water-soluble forms.

As the object of the study, 16 samples of technogenic soil (technozem) were selected from the most contaminated areas of the industrial zone we surveyed in the city of Svirsk, Irkutsk Oblast. This area is contaminated due to the activities of the former "Angarsk Metallurgical Plant", which processed sulfide-containing arsenic ores, and the "Vostsibelement" battery production plant (the main pollutant is lead) (Fig. 1).

This object is characterized by an extremely uneven pattern of contamination with very high total contents of priority toxicants [17, 18]. It is important to note that the nature of the contamination and the task itself are quite typical for the mining and metallurgical complex – there is historical contamination of the upper part of the soil to a depth of 1.5–2 meters with a complex of heavy metals, and the waste is located in the open air. Toxicants from them, both with surface runoff and by infiltrating into groundwater, can enter water bodies (in this case, the Angara River, on the bank of which the studied territory is located). Thus, the results of the work have not only general methodological significance, but also a distinctly practical nature.

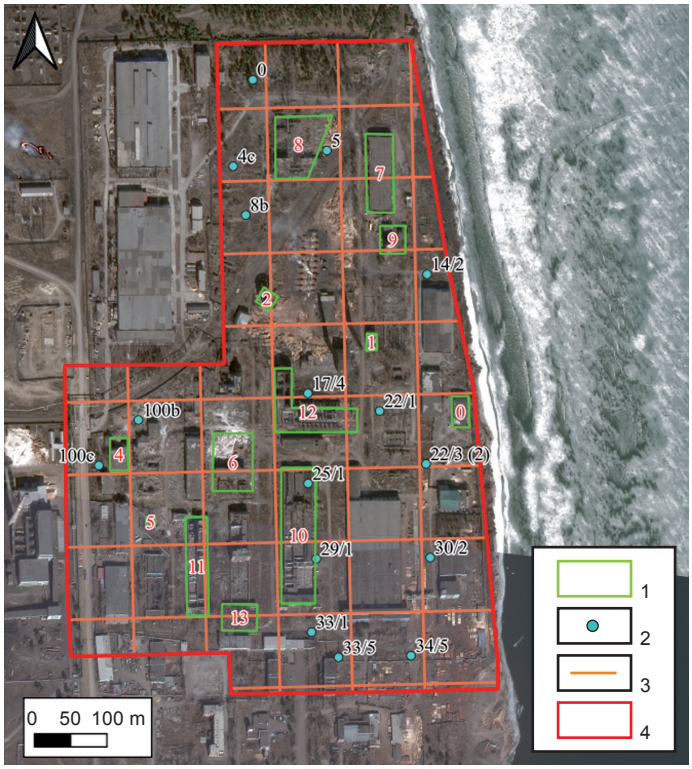

Fig. 1. Schematic sampling map:
1 – the most hazardous enterprises; 2 – sampling points (the sample number is to the upper right of the point); 3 – sampling network; 4 – boundaries of the former industrial site
Рис. 1. Схематическая карта отбора проб:
1 – наиболее опасные предприятия; 2 – точки отбора проб (справа вверху от точки – номер этой пробы); 3 – сеть отбора проб; 4 – границы бывшей промплощадки

Materials and methods

Modern certified methods for determining water-soluble forms involve working with an aqueous extract, and accordingly, using X-ray analysis methods for such a study object is very difficult. The preparation of an aqueous extract involves mixing a sample portion with distilled water in a 1:5 ratio, followed by shaking the resulting suspension on a shaker for 15 minutes. The extract is then filtered through a double “white ribbon” filter, and the elements in the resulting filtrate are determined using ICP-AES, inductively coupled plasma mass spectrometry, or atomic absorption spectrometry with plasma and electrothermal atomization (ERD F 16.2.2:2.3.71-2011[1]).

The total content is determined using concentrated nitric acid and hydrogen peroxide, followed by filtration and measurement of elements in the filtrate (ERD F 16.2.2:2.3.71-2011[1]). The ratio of poorly soluble and water-soluble forms can then be determined. The entire process requires highly qualified specialists, a large consumption of reagents, including state standard samples, and gas consumption, which makes the cost of analysis quite high. Of course, analytical methods can be combined, and the total content can be determined using other, less expensive methods. However, given that the final result for assessing the proportion of water-soluble forms depends on two different types of analysis, it is more appropriate to perform such studies using the same equipment, the same operator, and the same batch of reagents to reduce the likely cumulative error. When it comes to independent research projects, this can become a serious limiting factor, for example, affecting the number of samples collected, or leaving no room for error when planning the experiment. These limitations will certainly significantly reduce the overall quality of the study.

In the method we propose, the preparation of the collected samples consisted of coning

and quartering, followed by chessboard sampling to create more averaged and representative samples weighing 10 g, with 4 samples per one original sample, totaling 64 subsamples.

Next, to assess the reliability of our analysis results, half of the samples were simultaneously analyzed for total content using a Thermo Scientific 6300duo atomic emission spectrometer and a Sciaps X200 Geo mobile X-ray fluorescence spectrometer in an accredited laboratory of the Siberian School of Geosciences at Irkutsk National Research Technical University (Irkutsk, Russia). The results were processed in accordance with IST 41-08-214-04[2], which is intended for use in laboratories of departments conducting prospecting and exploration, extraction and processing of mineral raw materials, as well as the study of environmental objects. The results are presented in Tables 1 and 2.

Based on the results shown in Tables 1 and 2, the internal operational control for the two measurement methods was deemed satisfactory. Accordingly, the analysis results obtained for the available samples using XRF are in no way inferior to the longer and more costly results obtained using ICP-AES.

Table 1. Comparative results of copper content analysis using inductively coupled plasma atomic emission spectrograhy and X-ray fluorescence analysis methods
Таблица 1. Сравнительные результаты исследования проб на содержание меди методами атомно-эмиссионной спектрометрии с индуктивно-связанной плазмой и рентгенофлуоресцентного анализа

Sample number	ICP-AES		XRF		*Kk = C – Ck*	*Km*	*Kk ≤ Km*
	Content *C,* ppm	Relative measurement error	Content *Ck,* ppm	Relative measurement error			
14/2	157.8	37.8672	151.2	45.36	6.58	59.089	Yes
0	71.7	21.51	81.6	24.48	9.9	32.588	Yes
22/1	92.4	27.726	106.0	31.8	13.58	42.19	Yes
33/5	77.1	23.124	116.2	34.86	39.12	41.832	Yes
29/1	82.2	24.654	97.5	29.25	15.32	38.254	Yes
4c	59.7	17.922	85.6	25.68	25.86	31.315	Yes
8b	51.2	15.354	55.3	16.59	4.12	22.605	Yes
100 B	61.9	18.576	87.8	26.34	25.88	32.231	Yes

Table 2. Comparative results of lead content analysis using inductively coupled plasma atomic emission spectrograhy and X-ray fluorescence analysis methods
Таблица 2. Сравнительные результаты исследования проб на содержание свинца методами атомно-эмиссионной спектрометрии с индуктивно-связанной плазмой и рентгенофлуоресцентного анализа

Sample number	ICP-AES		XRF		*Kk = C – Ck*	*Km*	*Kk ≤ Km*
	Content *C,* ppm	Relative measurement error	Content *Ck,* ppm	Relative measurement error			
14/2	929.6	223.104	1048.6	146.804	119	267.0708	Yes
0	1931.6	309.056	1824.9	255.486	106.7	400.9847	Yes
22/1	1380.2	220.832	1152.5	161.35	227.7	273.497	Yes
33/5	5650	904	6341.5	570.735	691.5	1069.09	Yes
29/1	2254	360.64	2230.9	245.399	23.1	436.2131	Yes
4c	333	79.92	405.9	85.239	72.9	116.8456	Yes
8b	271.8	65.232	312.15	65.5515	40.35	92.47818	Yes
100 B	24060	3849.6	25526	1199.722	1466	4032.214	Yes

[2] *IST 41-08-214-04. Industry Standard. Quality Management of Analytical Work. Internal Laboratory Control of Accuracy (Trueness and Precision) of Quantitative Chemical Analysis Results.* Effective 01.06.2005, 92 p.

Results and discussion

The experiment methodology was as follows: a 10 g sample was placed in a 300 ml container, 100 ml of distilled water was added, mixed thoroughly, and then left for 2, 4, 6, and 8 hours. After each time interval, the sample was filtered through a “white ribbon” filter to separate the sample into solid and liquid phases (Fig. 2). The solid phase is of particular interest. Unlike the traditional method for determining water-soluble forms, which involves analyzing the filtrate, we assess the solid phase to determine the amount of substance that did not transfer into the filtrate, i. e., that remained in poorly soluble compounds in the solid phase.

After filtration, the filters with the residue were dried, transferred into polyethylene bags, and then subjected to further analysis. The experimental results for some samples are presented in Figs. 3, 4 and in Table 3.

This leaching method allows assessing the potential for toxicant migration under various conditions – from short-term exposure to atmospheric precipitation (2-hour leaching) to prolonged floods or inundations (8-hour leaching). Furthermore, this type of visualization (See Figs. 3, 4) enables rapid categorization of samples based on the leaching behavior of water-soluble forms. Thus, the presented figures show that in some samples (from certain areas), the majority of water-soluble forms transfer into the filtrate within the first two hours, and then begin to adsorb back onto the solid surface of the technosol. In other samples from different areas, the maximum amount of water-soluble forms transfers into the filtrate only by the eighth hour of the experiment. This information will subsequently allow

Fig. 2. Sample filtration process
Рис. 2. Процесс фильтрации пробы

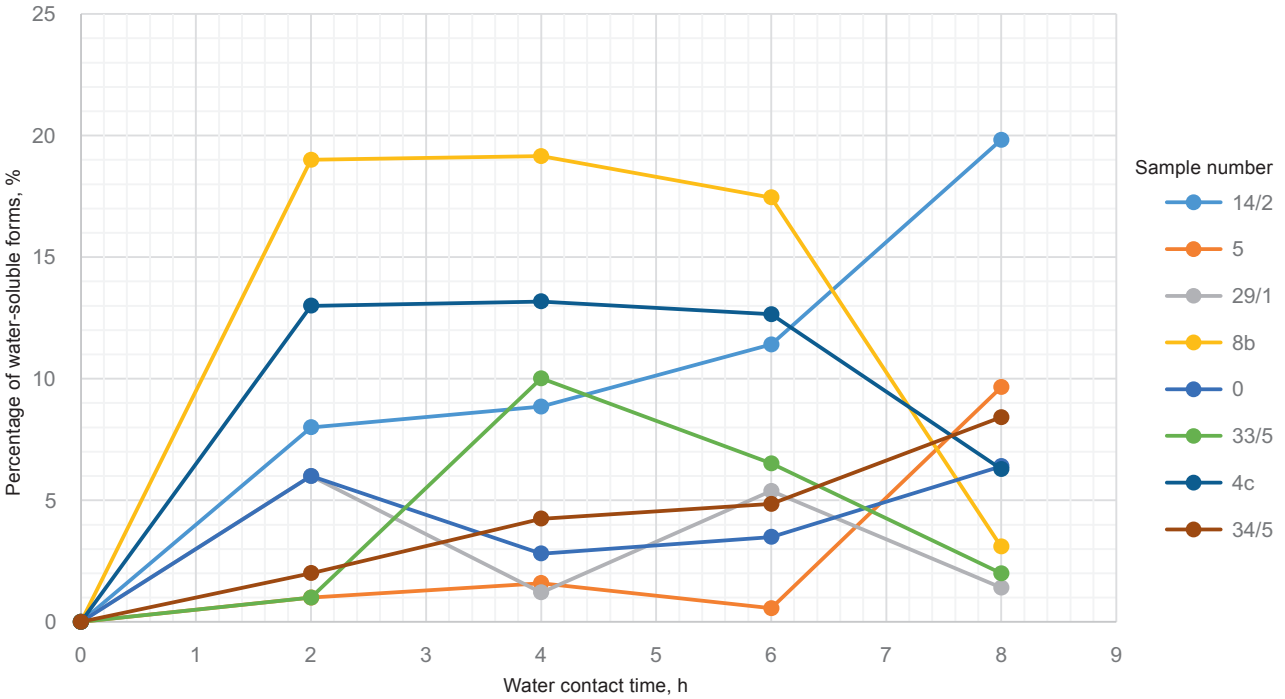

Fig. 3. Graph of changes in the percentage content of water-soluble forms of lead with different duration of contact with water for eight examined samples
Рис. 3. График изменения процентного содержания водорастворимых форм свинца при разной продолжительности контакта с водой для восьми обследованных проб

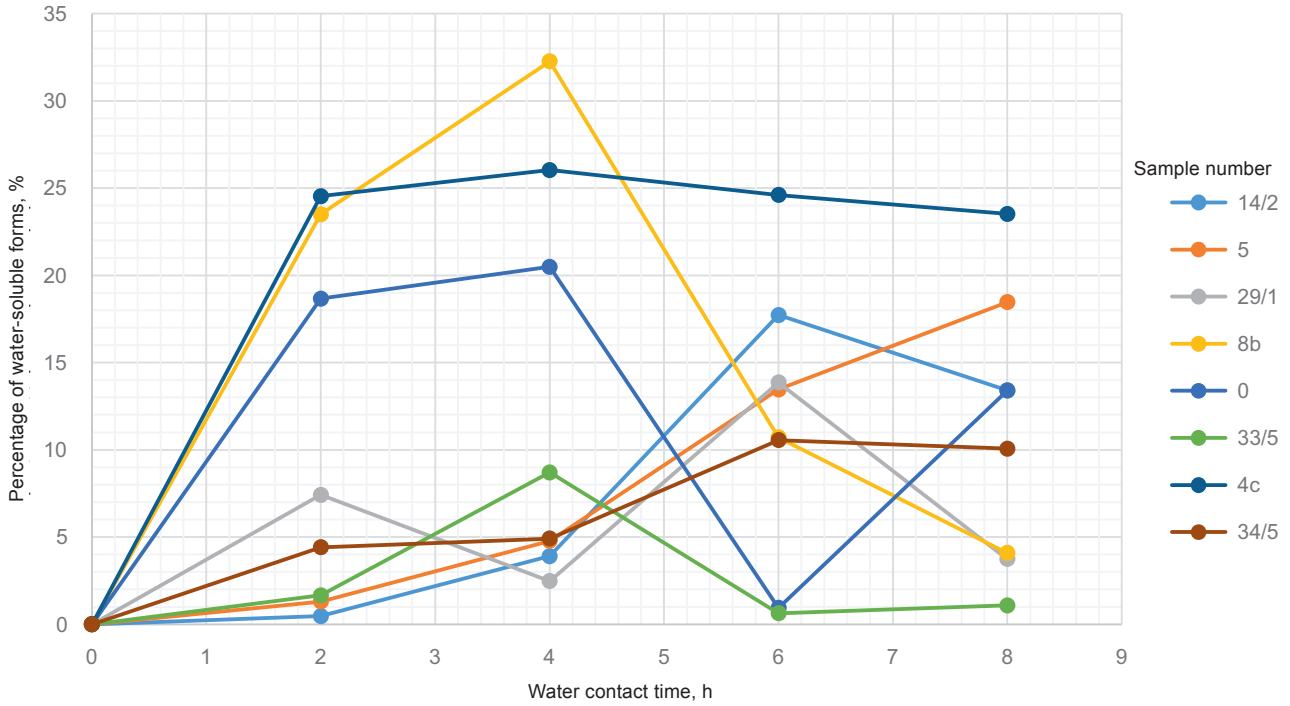

Fig. 4. Graph of changes in the percentage content of water-soluble forms of copper with different duration of contact with water for eight examined samples
Рис. 4. График изменения процентного содержания водорастворимых форм меди при разной продолжительности контакта с водой для восьми обследованных проб

Table 3. Results of sample No. 22/1 washing
Таблица 3. Результаты проведенной отмывки пробы № 22/1

Indicator	Content, ppm	
	lead	copper
Total content	1152.52	106.03
Content in solid residue on filter after 2 h	1030.49	87.55
Content in solid residue on filter after 4 h	944.29	79.71
Content in solid residue on filter after 6 h	927.79	79.71
Content in solid residue on filter after 8 h	1112.37	92.34

for the selection of remediation technologies for the given area, taking into account the specific characteristics of each technogenic site. Such information could not have been obtained using the standard method for determining water-soluble forms with ICP-AES, because in that case we would have obtained only the final result, which, as shown, does not convey the course of a distinctly non-linear process.

Conclusion

The method for determining water-soluble forms presented in the experiment differs from common certified methods in that it analyzes not the filtrate, but the solid residue on the filter. In other words, the percentage of water-soluble forms is calculated relative to the total content of the element in the sample and the amount remaining in the sample after its contact with water. This method does not require complex sample preparation or chemical reagents, and measurements can be performed using a portable X-ray fluorescence analyzer by a trained staff researcher without a degree in chemistry. The cost of such work is minimal, making it extremely relevant for independent research initiatives. The low cost allows for more complex experimental designs by significantly increasing the number of samples, for example, varying the contact time between the solid phase and water.

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