IRSTI 87.00.00 https://doi.org/10.26577/EJE20258432

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MONITORING OF DDT IN SOILS NEAR FORMER STOREHOUSES IN THE TALGAR REGION OF THE ALMATY OBLAST

The main aim of this research was to study soils from five villages located near former warehouses of banned and obsolete pesticides to determine their contents of DDT and its metabolite. Superficial soils were sampled from five villages (Kyzylkairat, Beskainar, Belbulak, Amankeldy and Yenbekshi) in the Talgar region (Almaty oblast) and their contents of organochlorine pesticides were investigated. Physical and chemicals parameters of soils were determined including dry matter, water content, ash, organic matter and pH. Monitoring of soil samples were performed by gas chromatography mass-spectrometry (GC-MS). Beskainar and Kyzylkairat agricultural soils exceeded MRLs for DDTs according to regulations in Kazakhstan, Russia and EU by more than 200 times. The concentrations of DDT found in the other studied soils showed medium levels respecting normative regulations. Nevertheless, concentrations tended to increase in comparison to previously published data of these places.

Keywords: soil, organochlorine pesticides, monitoring, DUST, DDT, contamination.

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Алматы облысы Талғар ауданындағы пестицидтер сақталған қоймалар маңындағы топырақтарда ДДТ мониторингісі

Бұл мақаладағы зерттеудің басты мақсаты тыйым салынған және ескірген хлорорганикалық пестицидтердің бұрынғы қоймаларына жақын орналасқан бес ауылдың топырағын зерттеу, ДДТ және оның метаболиттерінің құрамын анықтау. Алматы облысы, Талғар ауданының бес ауылының (Қызылқайрат, Бесқайнар, Белбұлақ, Аманкелді және Еңбекші) жер үсті топырақтарының үлгілері алынды және олардағы хлорорганикалық пестицидтердің концентрациясы зерттелді. Сонымен қатар алынған сынамаларда келесі физико-химиялық көрсеткіштер анықталды: құрғақ заттар, су, күл, органикалық заттар және pH. Топырақ сынамаларындағы ДДТ және оның метаболиттерінің концентрациясы газды хроматография – масс-спектрометриялық әдіс (GC-MS) арқылы анықталды. Бесқайнар және Қызылқайрат ауылдарының топырақтарындағы ДДТ мөлшері Қазақстан, Ресей және ЕО стандарттары бойынша шектік концентрациядан 200 еседен асып түсті. Басқа зерттелген ауылдардағы топырақтарда табылған ДДТ концентрациясы шектік көрсеткішке жақын болды. Зерттелген нысандардарда табылған хлорорганикалық пестицидтердің концентрациясы өте жоғары және де бұл ауылдар Қазақстанның ең ірі қалаларының бірі – Алматыға жақын орналасқан. Сондықтан да алынған мәліметтер осы ауылдардағы хлорорганикалық пестицидтермен ластануды басқару, ластанған топырақтарды «тазарту» шұғыл және маңызды іс-шараларды қабылдау үшін үкіметтің назарына ұсынуға пайдаланылады.

Түйін сөздер: топырақ, хлорорганикалық пестицидтер, мониторинг, ДУСТ, ДДТ, ластану.

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Мониторинг ДДТ в почвах вблизи бывших складов в Талгарском районе Алматинской области

Основная цель данного исследования заключалась в изучении почв из пяти поселков, расположенных недалеко от бывших складов запрещенных и устаревших пестицидов, для определения содержания в них ДДТ и его метаболитов. Были взяты образцы поверхностных почв в пяти поселках (Кызылкайрат, Бескайнар, Белбулак, Аманкельды и Енбекши) Талгарского района Алматинской области и исследовано содержание в них вышеуказанных хлорорганических пестицидов. Были определены физико-химические параметры почв, включая сухое вещество, содержание воды, золы, органического вещества и pH. Анализ на содержание хлороганических пестицидов в образцах почвы проводился методом газовой хроматографии с масс-спектрометрическим детектированием (ГХ-МС). В сельскохозяйственных почвах поселков Бескайнара и Кызылкайрата ПДК для ДДТ и их метаболитов превышали нормативы Казахстана, России и ЕС более чем в 200 раз. Концентрации ДДТ, обнаруженные в других исследованных почвах (Бельбулак, Аманкельды и Енбекши) не превысили ПДК, но были близки к пороговому значению. Полученные результаты будут использованы для информирования правительства РК с целью принятия безотлагательных мер для управления загрязнением пестицидами на исследуемых территориях, расположенных в непосредственной близости от одного из крупнейших городов Казахстана – Алматы и, следовательно, оказывающих влияние на большое количество жителей.

Ключевые слова: почва, хлорорганические пестициды, мониторинг, ДУСТ, ДДТ, загрязнение.

Introduction

According to the results of the inventory of hazardous waste in the EECCA countries (Eastern Europe, the Caucasus and Central Asia) 56 930 tons of pesticide waste and 190 848 tons of other POPs (persistent organic pollutants) are present on the territory of the Republic of Kazakhstan [1]. The pesticides actively used in Kazakhstan in Soviet Union time and accounts approximately 18–20 million ha of the lands [2]. At the same time, there is no official data on pesticide contaminated soil. Soil is the main reservoir for all types of pollutants, including POPs and in particular organochlorine pesticides (OCPs). There are partial scientific studies conducted in Kazakhstan on the content of organochlorine pesticides in the soil and in food. For example, the concentrations of organochlorine pesticides such as p,p'-DDT, p,p'-DDE, p,p'-DDD and hexachlorocyclohexanes (HCHs) from former storehouse in the village Kyzylkairat (Talgar region, Almaty oblast) reported ranges from 1.38 to 11 100 μg kg^{-1} [3], when maximum permissible concentrations (MPC) are limited at 100 μg kg^{-1} in Kazakhstan. Generally, 24 out of 80 warehouses located in Kazakhstan showed pesticide levels in soil higher than MPC [4]. In addition, recent studies reported concentrations of more than 24 different pesticides found in food products of plant and animal origin which located in the vicinity of old warehouses in Talgar rayon of Almaty oblast. Pears, apples, tomatoes, cucumbers, meat and milk are very heavily contaminated with pesticides DDTs, HCHs, aldrin, endrin, dieldrin, which probably accumulated from the contaminated soil along the food chain. [5].

According to the other studies [6, 7] contaminated soil can be the main exposure pathway for free-ranged animals. Livestock such as dairy cows and sheep and chicken could ingest daily up to 10% soil in totally ingested dry matter in normal grazing conditions [8] whereas over 20% of soil has been reported in deteriorated grazing conditions [9, 14]. Therefore, soil could be one of the main vectors of these environmental contaminants to farm animals and in food they produced (milk, meat and eggs) especially when these animals were raised on contaminated areas.

Subsequently consumption of contaminated food would be a possible route of POP exposure for humans, including OCPs. The main studied OCPs in soil of Kazakhstan are DDT, HCH isomers, endosulfans, and the aldrin group. These pesticides were legally used as insecticides till 1983 but illegal use up to recent years [10]. Due to the widespread use of these chemicals and their very high persistence in soil, they can even today be found in environmental

samples. Moreover, even nowadays it is easy to find in the local markets a white household powder in red flasks called "DUST", which consists at 99% of DDT according to preliminary studies. Today, the population of our country uses it currently against insects but does not know the dangerous side effects on human health. Therefore, the issue of monitoring organochlorine pesticides in environment remains relevant even some decades after the withdrawal of their authorization to be used.

We performed a determination of DDT and metabolites in superficial soils near the biggest city of Kazakhstan: Almaty. The main aim of the research was to monitor the concentrations of DDT and its metabolites in soil samples from five villages near former pesticides storehouses close to Almaty city in order to evaluate the risks of contamination and as a consequence of their transfer into food of free-range animals.

Materials

Analytical standards and reagents

Certified reference standards of individual organochlorine pesticides 4,4-DDT (p/n: 31041-100MG), 4,4-DDE (p/n: 35487-100MG) and 4,4-DDD (p/n: 35486-250MG) were purchased from Sigma Aldrich Co. Stock solution of OCP mix and calibration solutions were prepared in n-hexane GC grade. Organic solvents used in this study include acetonitrile and n-hexane with analytical grade. Anhydrous magnesium sulfate was used as drying agent before the evaporation stage.

Methods

Sampling

Soil samples were collected in 5 villages: ***Kyzylkairat*** (43°17'58.84"N and 77°11'41.53"E), ***Beskainar*** (43°13'16"N and 77°6'51"E), ***Belbulak*** (43°19'26.88"N and 77°06'19.71"E), ***Enbekshi*** (43°19'56.88"N and 77°12'09.71"E) and ***Amankeldy*** (43°17'55.3"N and 77°12'27.89"E) located in Talgar region, at Almaty oblast. These samples represented anuperficial soil with a long-time contamination in field conditions. The sampling sites were chosen due to their proximity to old, abandoned pesticide storage facilities (storehouses). Sampling was performed in May 2020. Superficial soil (0-20 cm deep) was collected and placed directly in a suitable aluminum container. Before analysis, stones, twigs, and other objects with a diameter of more than 2 mm were manually removed. Then, soils were air-dried and sieved over a 2 mm sieve.

Physical and chemicals parameters of soils

Organic matter and pH of soils were analyzed according to GOST 26213-91 and GOST 26423-85 respectively. Determination of water content and dry matter were performed using gravimetric method according to the ISO 11465.

Extraction of OCPs from soil

The extraction of the soil samples was carried out by the method described elsewhere [11] with some modifications. 10 grams of soil were weighed and transferred to 250-mL separatory funnels. Then, 10 mL of acetonitrile was added, and the corked flasks sonicated for 5 min. An additional 10 ml of acetonitrile was added, and the separating flasks closed tightly. The content of the flasks was placed on a horizontal mechanical shaker (Ika-Werke HS 501 Digital) and was set to shake continuously during 30 min at 300 rot/min and allowed to stand for 10 min to sufficiently separate the layers. The supernatants (organic layers) were carefully transferred into 50 mL centrifuge tubes for centrifugation at 3000 rpm for 5 min. After, 10 mL of supernatant collected and passed through the paper filter with anhydrous magnesium sulfate into 250-mL rotary flask. Additional 10 mL of acetonitrile was used to rinse the filter. Combined extract evaporated to dryness and re-dissolved in 1 mL n-hexane. Each soil extract was prepared in triplicate and analyzed by GC-MS. All samples recommended to analyze in the same day with extraction otherwise, the samples must be refrigerated at -20 ºC.

GC-MS parameters

Determination of selected OCPs in soils were performed using a GC-MS (7890B, Agilent Technologies). Briefly, the mass spectrometer was set at a resolution of 10 000, in electron ionization mode (70 eV electron energy) (5975C, Agilent Technologies). Single Ion Monitoring (SIM) was used to record the most abundant signals (235 m/z for DDD and DDT, 246 for DDE). A HP-5ms Ultra Inert (30 m x 250 μm x 0.25 μm) capillary column from Agilent J&W (Agilent Technologies) was used in spitless mode. The GC temperature program for OCP analysis was the following: 120°C (1 min), 40°C min−1 to 220°C (13 min). Total time – 16,5min. Signals were integrated using Mass Hunter (B 07.05.2479).

Data Analysis and Calculations

Determination of pH and the contents of water, dry matter and organic matter were performed and calculated strictly according to the ISO 11465 Soil quality. Determination of dry matter and water

content on a mass basis by gravimetric method, ISO 10694:1995 «Soil quality. Determination of organic and total carbon after dry combustion (elementary analysis)» and ISO 10390:2005 «Soil quality. Determination of pH».

The concentrations of pesticides (C) in the soil samples were calculated using external calibration method. Two calibration solutions with concentrations 25, 50, 100, 250,500 and 1000 µg/L for each compound were prepared in n-hexane and injected to GC-MS. Each calibration point was realized in triplicates. Mean peak areas were calculated and used to build the calibration curves for each studied pesticide.

The linear equations obtained using the calibration curve were used to calculate the concentration of the substance in the extract (C, µg/L), i.e., peak area divided on calibration coefficient. Then, concentration in the extract (µg/L) was converted to concentration of OCPs in the soil mass (µg/g) according to equation 1:

$$C_{soil} = \frac{C_{extract} \times \text{Solvent (L)}}{m_{soil}\,(g)} \qquad (1)$$

Where C_{soil} – concentration of compound in the soil in µg/g, $C_{extract}$ – concentration of compound in the soil extract in µg/L, Solvent (L) – volume of organic solvent that was used, and m_{soil} – mass of soil in gramm.

Finally, the concentrations of pesticides in soil were expressed on the dry matter basis (Table 1). Each soil sample and calibration samples were tested in triplicates. Mean concentration and standard deviations were calculated and presented in the tables in comparison to literature data.

Results and discussion

Physical and chemical characteristics of soils

Physical and chemical properties of soils such as pH, dry matter, water content, organic matter and ash are very important because of their influence on DDT bioavailability and mobility in soils. Dry matter is a main carcass of soil which is responsible for accumulation. Consequently, determined pollutants in soil are generally expressed on a dry matter basis.

Results showed that soils from Kyzylkairat, Beskainar, Yenbekshi, Amankeldy and Belbulak have similar physical and chemical properties (Figure 1). This can be explained by the fact that the studied sites are located in the same geographic zone (piedmont) and climate. Despite the similarity of physical and chemical parameters, the content of pesticides found in studied soils differed largely.

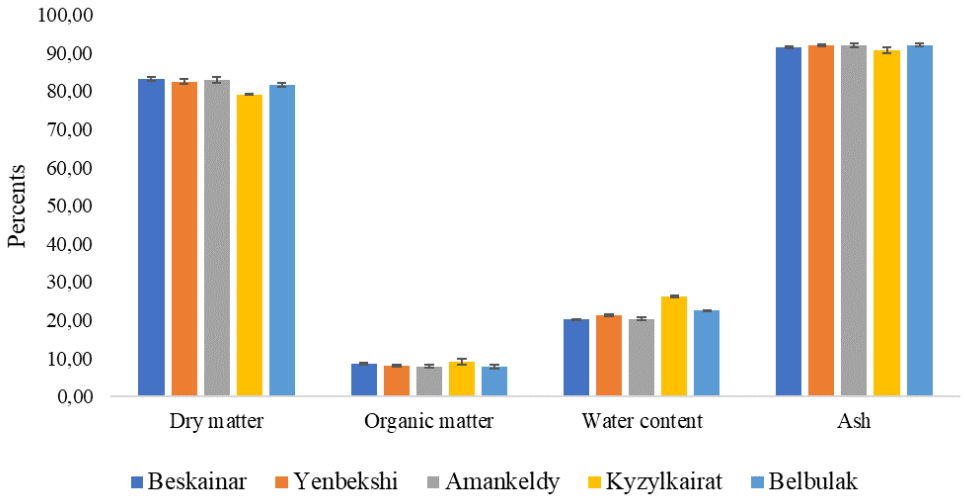

Figure 1 – Physical and chemical characteristics of soils sampled in Beskainar, Yenbekshi, Amankeldy, Kyzylkairat and Belbulak

Determination of organochlorine pesticides residues

Results of organochlorine pesticides DDT, DDE and DDD concentrations were detected in studied soils and compared to the published data (Table 1). Generally, all studies soil samples were contaminated by organochlorine pesticides studied in a notable manner. Results showed that soils from Kyzylkairat and Beskainar have the highest concentrations of DDT, DDE and DDD with 8,5, 11,1, 1,2 mg/kg and 0,1, 1,1 and 0,2 mg/kg dry matter, respectively. The soils from these sites are highly contaminated and overpassed consequently MPC applicable in Kazakhstan. Indeed, some of the metabolites exceeded the threshold of 0,1 mg/kg between ten and even hundred times (table 1).

Comparing the obtained results with the previously published data (2016), DDE probably reflects a metabolic activity in these soils and is cracked from the parental compound of DDT. Nevertheless, the DDT concentration did not vary significantly (Table 1).

Table 1 – Mean concentrations (C, mg/kg dry matter) and standard deviations (SD) of DDE, DDD and DDT determined in 5 villages near Almaty city

Sampling sites	Pesticides		
	4,4-DDT	4,4-DDE	4,4-DDD
Beskainar: Our study	0,1 ±0,004	**1,1** ±0,04	**0,2** ±0,003
Belbulak:			
Our study	0,02 ±0,001	0,04 ±0,002	0,01 ±0,0003
Global Monitoring Plan 2016	**1,7** ±0,2	**0,9** ±0,02	
Yenbekshi: Our study	0,01 ±0,002	0,02 ±0,001	0,005 ±0,003
Amankeldy: Our study	0,05 ±0,001	0,1 ±0,002	0,02 ±0,002
Kyzylkairat:			
Our study	**8,5** ±0,37	**11,1** ±2,3	**1,2** ±0,06
Global Monitoring Plan 2016	**6,6** ±0,2	**2,1** ±0,05	**1,9** ±0,04
Sailaukhanuly et al., 2016	**0,3** ±0,001	**0,4** ±0,001	**0,4** ±0,001
[1]RK MAC, mg/kg	0,1	0,1	0,1
[2]EAEU MRL, mg/kg			
[3]EU MRL, mg/kg	0,1	-	-
[4]US MRL, mg/kg	0,05	-	-

[1]Republic of Kazakhstan Maximum Allowable Concentrations for DDT and metabolites in soil
[2] Eurasian Economic Union maximum residue limits for pesticides in DDT in agricultural soil
[3] European Union Maximum residual level of DDT in soil
[4]United Stats MRLs United States maximum residue limits for pesticides in DDT in agricultural soil
Each soil sample was analyzed in triplicates
Bold means exceed the MRL levels

Storehouses represent up to today an undoubted danger for the studies areas. Moreover, the presence of DUST and its likely use let fear a continuous diffusion of DDT (Figure 2).

Other earlier monitoring studies showed similar tendency of contaminated levels of DDT and metabolites in Kyzylkairat soil to current study results with exceeding international and national MRLs (Table 1). Unfortunately, there is a lack of existing data of pesticide levels in soils of other studied areas. However, according to literature, local produced fruits (apples, pears), vegetables (tomatoes, cucumber), meat and milk exceeded international and national regulatory MRL in a consequent manner (Djangalina et al., 2020). The local habitants traditionally produce still food for their own consumption which could be highly contaminated by pesticides and therefore represent a threat to human health. Urgent government attention is necessary to manage pesticide contamination in the studied areas located in close proximity to the one of the biggest city of Kazakhstan and therefore with an impact on a large number of inhabitants.

Figure 2 – Photo of commercial mixtures DUST in the Almaty market

OCP transfer to food of animal origin

According to available literature data [13], there are totally 64 abandoned pesticide warehouses in 8 regions of Almaty oblast located at up to 250 km from the largest metropolis – the city of Almaty. About 352 650 kg of obsolete [13], unusable pesticides had been stored in the Almaty oblast. The largest number of pesticides was registered in the Ili region having three pesticide storehouses which had received 107150 kg of obsolete pesticides (Figure 3). These pollutants are extremely resistant to degradation and would accumulate in the human and animal fat. The chronical exposure of the local population even to small doses have shown to contribute to higher frequencies of major pathologies as cancers and reproductive pathologies causing sometimes even the death.

We studied 5 villages (Kyzylkairat, Beskaynar, Belbulak, Amankeldi and Enbekshi) located from 5 to 15 km from the city of Talgar and from 25-50 km to the city of Almaty. These sites showed high contamination level of DDT and metabolites in soils which is used in agricultural purposes. Moreover, these places may widespread such toxic compounds in surrounding fields or gardens by movements of water or soil dust. In addition, commercial mixtures contained DDT are still on the markets of Kazakhstan. This fact raises a sharp question of pesticide transfer from soil to livestock and food of animal origin.

Despite the availability of data of all abandoned pesticide storehouses, there are no information on the levels of pollutants in environmental objects and food products in other regions of the Almaty oblast. Consequently, it is not known what happens to the health of people over time in other rayons who still unwittingly could consume pesticide contaminated food.

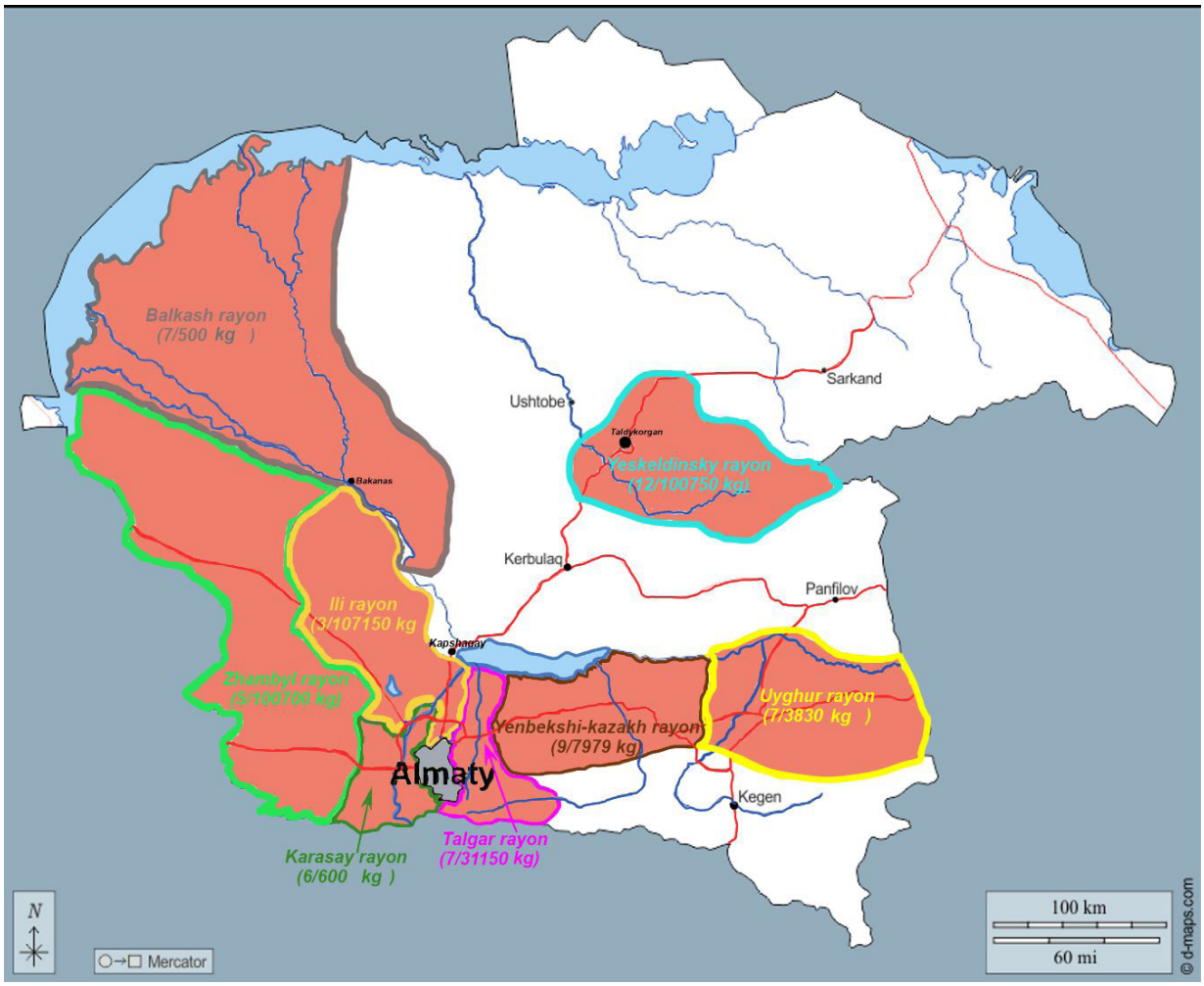

Figure 3 – Map of the number of warehouses / tons of obsolete pesticides in different rayons of Almaty oblast

Conclusion

The long-time storage of pesticides enhanced strong contamination in the studied areas and created a real risk of transfer of organochlorine pesticides from agricultural used soils to food produced on these territories. This represents a high risk for the health of the local inhabitants. Urgent government measures are necessary to manage this risk by corrective means such as identification of the hotspots and sequestration methodologies. Further investigation is required to estimate the real sanitary status of the local food products and to develop appropriate effective technologies to limit the distribution and bioaccumulation of these toxic substances in the environment.

Acknowledgment

This work was supported by the Ministry of Education and Science of the Republic of Kazakhstan, Committee of Science in the frames of research project: AP09057889 «Development of technology to reduce the transfer of persistent organic pollutants from pasture soils to food of animal origin».

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Поступила 03 июля 2025 года
Принята 26 сентября 2025 года