Abstract. The materials of long-term research of the features of the content and distribution of fluorine in southern chernozems are highlighted. The purpose of this work is to investigate the effects of irrigation and chemical meliorants on the fluorine accumulation in soils and crops. To achieve this goal, the following tasks were set: to determine the content of fluorine in soils and plants in conditions of irrigation and application of phosphorus fertilizers (phosphogypsum); to carry out ecological assessment of southern chernozems in terms of accumulation and migration of fluorine in soil, plant resistance to fluorine accumulation and translocation. The research was conducted within the irrigation areas of Odessa region. Bulk and movable forms of fluorine were determined by the potentiometric method in the southern chernozems of different reclamation state (non-irrigated, irrigated and irrigated reclaimed). The content of movable fluorine in agricultural plants was determined by the author’s method. It was established that the contest of bulk fluorine is the highest in the non-irrigated chernozems, but does not exceed the MPC. In irrigation, the number of bulk forms of fluorine decreases due to their dissolution and migration from the top layer to the depth of the soil profile. The content of soluble forms of fluorine increases significantly in all components of agrolandscape under the influence of irrigation and especially the introduction of phosphogypsum. It was determined that the combined application of phosphorous fertilizers and manure leads to a decrease of the fluorine content in soils, lysimetric waters and agricultural plants. The degree of mobility of fluorine compounds in the studied chernozems during irrigation and especially the introduction of phosphogypsum increases. The level of fluorine content in lysimetric waters and the coefficient of water migration correlate with its content in soils. A positive correlation was found between the fluorine content in the roots of maize, oats, wheat ear and soil while its absence between the content of fluorine in the stems and leaves. The introduction of mineral and organic fertilizers together enhances the ability of plants to absorb fluorine from chernozem soils. The active contamination rate for all tested plants is higher than one, which can cause their inhibition. When resuming irrigation of chernozem soils, there will be a need for increased doses of mineral, including phosphorus fertilizers. Despite the low solubility of fluorine compounds, conducted research has shown that irrigation and application of phosphogypsum significantly increases the soluble forms of fluorine, which can adversely affect the pollution of agrolandscapes and public health.

Key words: fluorine, southern chernozem, agricultural plants, ecological assessment

Agroecological assessment of fluorine in soils and agricultural plants of steppe landscapes of Odessa region

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Introduction. In modern conditions of declining environmental quality, the environmental approach of geographical research becomes a priority when studying both individual components of the nature and the environment in general (Topchiyev et al., 2019). Soil is the most stable component of the environment, in which not only the nutrients of the plant, but also the toxic chemicals accumulate. One of these phytotoxic trace elements is fluorine. Fluoride accumulation in plants depends on the presence of its movable forms in the environment (and especially soil) and the individual characteristics of plant organisms. Fluorine and its compounds are characterized by high chemical activity and are highly toxic (1 hazard class) to soil biota and plants. Excessive content of fluorine in soils is manifested in the change of its physicochemical properties, decrease in fertility, damage to plants, decrease in yield, diseases of animals and humans.

The systematic introduction of mineral fertilizers and chemical meliorants which are used to increase the soil fertility is inevitably related to the introduction of contaminants into the soil. Despite the sharp decrease in the volumes and rates of application of mineral and organic fertilizers in recent decades, the degree of contamination of the irrigated chernozems in southern Ukraine remains high, which is caused by the consequences of the previous long-term chemical melioration of agriculture, the features of lands use and the properties of the chernozem soils.

Fluorine and its compounds have a narrow range of physiologically optimal content. Both insufficient and excess fluorine content can adversely affect living organisms. It is very difficult to determine safe levels of fluorine in soils and crops (Trigub, 2014). The contradiction of numerous studies and opinions of scientists at a level of safe fluorine content in the components of the environment due to the presence of various factors which define toxicity of fluorine and the lack of comprehensive analysis of environmental and hygienic aspects of pollution of the biosphere generally (Trigub and Lyashkova, 2018).

The relevance of the author’s study of fluorine compounds in the steppe landscapes of the region is due to the properties of trace elements accumulate in plants and through the trophic chains adversely affect human health.

The purpose of the study is to investigate the effects of irrigation and chemical meliorants on the fluorine accumulation in soils and crops.

To achieve the goal, the following tasks were set:

1. To determine the content of fluorine in soils and plants in conditions of irrigation and melioration of them by phosphogypsum; to carry out ecological assessment of southern chernozem with indicators of accumulation and migration of fluorine in soil, resistance of plants and their organs to fluorine accumulation.

The issues of fluorine translocation from soil to plants are quite fully covered in the literature (Pickering, 1985; Elrashidi and Lindsay, 1986; Trigub and Poznyak, 2008; Jha et al., 2009; Mourad et al., 2009; Tayibi et al., 2009; Perez – Lopez et al., 2010; Smidt et al., 2011; Tandelov, 2012; Lakshmi et al., 2016). However, the analysis of previous studies shows that a single opinion on the accumulation of fluorine in plants depending on its content in the soil, especially in the reclamation of phosphogypsum, has not been developed and is now controversial. The accumulation of fluorine in soils and plants during the application of mineral fertilizers was considered in the works of Kudzin and Pashova (1970), Piotrowska and Miacek (1975), Thompson et al. (1979), Pashova (1980), Baranovsky and Pankrutskaya (1992), Kabata-Pendias and Pendas (1989), Loganathan et al. (2001), Weinstein and Davidson (2004), Tandelov (2012), Trigub, Poznyak (2014), and others. Systematic application of phosphorus fertilizers and phosphogypsum in large doses causes an increase in fluorine content in chernozem soils (Trigub and Poznyak, 2014). The question is whether plants are enriched with fluorine from soils which contain it in large quantities? Kabata-Pendias and Pendas (1989) tend to conclude that the fluorine content in plants does not depend on its content in soils. However, this conclusion is not sufficiently substantiated, as it concerns the results of determinations of sparingly soluble (gross) forms of fluorine. According to Holevas (1960), some tropical soils may contain organic fluorine compounds, which are easily available for plants and highly toxic for animals. According to Piotrowska and Miacek (1975), Thompson et al. (1979), Pashova (1980), Trigub and Poznyak (2008), Mourad et al. (2009), Tandelov (2012) enrichment...
of soils with fluorine-containing mineral fertilizers leads to an increase in its content in crop yields. The relationship between the quantitative content of fluorine in soils and plants is defined in the works of Haidouti et al. (1993), Davis (1995), Tandelov (2012), Trigub and Poznyak (2014). In climate changing conditions and the urgent need for irrigation of soils of the steppe zone of Ukraine, the problem of fluorine pollution of chernozem soils and agricultural plants is relevant and needs further study.

**Materials and methods of research.** The following methods are applied in the work: field, laboratory, measuring, calculation and comparative, data graphical display. The studies were conducted using techniques and methodological approaches certified and standardized in Ukraine (Metodicheskiye ukazaniya…., 1975; Yakist gruntu…, 2004; Baliuk et al., 2013).

Studies on the influence of irrigation and phosphogypsum application on the fluorine content in chernozem soils and agricultural plants were carried out within the irrigation arrays of Odessa region (Fig. 1). In 1993-1995, soil scientists of Odessa University under the leadership of I.M. Gogolev established an experimental network of long-term soil and ecological monitoring sites on irrigation
areas (IA) of Odessa region. The plots differ in landscape and reclamation conditions and genetic and reclamation and production properties of the original (before irrigation) chernozem soils, the duration of the period of more or less intensive and systematic irrigation and the subsequent period of its cessation and extensification of agriculture in the last 25 years. In modern conditions, regular (or even periodic) watering is carried out on an area of not more than 10-20 times, up to 30% of irrigation developed lands in previous years. However, even today, monitoring sites annually conduct research on the evolution of soils and lands under conditions of irrigation, as well as the cessation and post-irrigation evolution. Monitoring studies also include studies of fluorine in agrolandscapes of irrigation areas (Chornozemy’ masy’ iv ......, 2016).

Soil contamination can be controlled by the content of both bulk and movable elements. The vast majority of fluorine in soils (up to 95%) is in the form of poorly soluble compounds (Perelman, 1989). Although, it is mostly affected by soil properties and living organisms, such as water-soluble forms of fluorine.

Studies on the effects of irrigation and phosphogypsum introduction on fluorine content in chernozem soils and agricultural plants were conducted within the irrigation areas of Odesa region. In soil samples, the bulk and active (acid-soluble and water-soluble) fluorine were determined using potentiometric method, in plants, the active forms of fluorine were determined by the author’s method (Trigub, 2019).

In order to determine the vertical migration of fluorine in the studied area, lysimeters were installed at depths of 30 and 60 cm. Sampling of lysimetric waters was performed three times per a year – in spring, summer and autumn. The fluorine migration ability (coefficient of water migration) was determined by the ratio of the number of element atoms that passed into the mobile state (water) to the number of its atoms in the soil (Kovda, 1973).

Ecological evaluation of the studied soils and plants was performed according to the indicators of accumulation and migration of fluorine in the soil, resistance of the plant to the accumulation and translocation of fluorine (Ilyin and Stepanova, 1979, Baliuk et al., 2013).

Results of the studies and their discussion. Phosphorus is one of the important elements of root plant nutrition on irrigated chernozems. Phosphorous fertilizers which are used in agricultural production (superphosphate, double superphosphate, precipitate, etc.) always contain more or less biologically toxic fluorine in their composition. There are different opinions about the possibility of soil contamination with fluorine due to irrigation and prolonged use of phosphorus fertilizers.

According to our researches, in the upper horizons of the southern chernozems, the samples of which were collected on the irrigation areas (IA) of the Odessa region of Ukraine (Fig. 1), the bulk fluorine content ranges from 310.0 to 597.0 mg/kg (Fig. 2). The content of bulk fluorine in

![Fig. 2. The content of fluorine in the arable layer of southern chernozem irrigation areas of Odessa region (mg/kg)](image-url)
The high content of soluble forms of fluorine in the upper horizon of southern chernozem, especially in non-irrigated ones, can be explained by the biological accumulation of this element. In the subirrigated horizon, the content of fluorine decreases slightly. The highest concentrations are typical for carbonate accumulation horizons. There is a natural increase in the concentration of active fluorine downwards the profile: at a depth of 140-150 cm it reaches 25-30 mg/kg, which is associated with an increase in its bulk forms. The maximal values of fluorine content are confined to soil-forming rocks.

Researches on fluorine accumulation in soils with the systematic application of fluorinated fertilizers are highlighted in the works of Pashova (1980), Pomazkina (2004), Trigub and Poznyak (2008), Mourad et al. (2009), Brindha et al. (2011), Tandelov (2012).

Versatile researches of the influence of irrigation and gypsum at fluorine content in soil, groundwater and crop products were conducted within the Danube-Dniester IS in the field experiments (Fig. 1). The objects of the study were southern chernozems of warm southern European facies irrigated with low-mineralized waters. In the studies conducted, the main attention was paid to the study of the content of active fluorine, because this form of the microelement is the most toxic to the food chain. Phosphogypsum containing 2% (20000 mg/kg) of total fluorine and 0.3% (3000 mg/kg) of water-soluble fluorine was used for the amelioration of the solentic chernozems in the southern Danube-Dnestrovsk IS. Therefore, with the introduction of 10 t/ha of phosphogypsum, about 30 kg/ha of active fluoride is introduced into the soil, which can cause an increase in its content in the soil.

According to the results of our research, under the influence of irrigation, the concentration of bulk fluorine in the arable layer in the irrigation area decreased by 60 mg/kg, in the subarable layer 72 mg/kg, which can be explained by the gradual dissolution of the bulk forms and prolonged migration of water-soluble fluorine from the upper layer to the bottom of the soil profile. The content of water-soluble fluorine in the arable layer is the lowest in non-irrigated chernozems (Fig. 3). During irrigation, and especially during irrigation and gypsuming, the content of movable forms of fluorine in the arable horizon increases. With the addition of phosphogypsum, in combination with manure, the fluorine content decreases, because the organic matter of manure is capable to form poorly soluble complexes with fluorine.

According to the classification of irrigated soils by the content of water-soluble fluorine (Baliuk et al., 2013), non-irrigated chernozems of the studied area have low level (0-3 mg/kg), irrigated – mainly average level (3-6 mg kg) and during irrigation and introduction of phosphogypsum, fluorine content increases, in some cases reaching a high level (6-9 mg/kg).

Consequently, the introduction of phosphogypsum into irrigated chernozems increases the content of water-soluble and acid-soluble fluorine, but its absolute values remain, at the same time, lower than the level of MPC. Studies conducted confirm that, despite the low solubility of fluorine compounds (2.1 $10^{-3}$ %) in chernozem in the process of irrigation, there is a gradual dissolution occurs, which leads to an increase of the active forms, especially acid-soluble fluorine (almost by 3 times). The increase of movable fluorine concentrations in irrigated soils, compared to their bulk content, indicates an increase in its mobility, migratory capacity, increase of the risk of flow into plants, the possibility of migration into the lower horizons of the soil and groundwater, which requires constant monitoring to prevent negative consequences in soils, groundwater and crop plants.
The dynamics of the content of active fluorine in the southern chernozem of the Danube-Dniester IS is presented in the Fig. 4 and the Fig. 5. As it is clear from the figures, that during irrigation (Fig. 4) and especially during irrigation and gypsumming (Fig. 5), the activity and, consequently, the mobility of fluorine in the upper layer of chernozems increases from year to year, creating a threat of contamination for geochemically dependent landscapes, natural and groundwater, plant products with this element.

As can be seen from the Figures 4 and 5, the irrigation of chernozems leads to redistribution of fluorine in the soil profile and increase the concentration of its active forms in the arable layer. The systematic introduction of phosphorus fertilizers and phosphogypsum in high doses leads to an increase of fluorine content in soils, especially its active forms (water-soluble and acid-soluble).

Among the negative effects of irrigation and the introduction of phosphate fertilizers is the accumulation of fluorine not only in the soil, but also in surface waters. The accumulation of fluorine in groundwater is influenced by the degree of mobility of fluorines (MF), which may increase with additional
hydration (irrigation) and the introduction of fertilizers containing fluorine compounds. The degree of mobility of fluorines is calculated by the ratio of the content of soluble forms of fluorine to their bulk content in the soil (Pomazkina, 2004). Despite the low solubility and mobility of fluorine in chernozem soils, when the phosphogypsum was introduced, the degree of mobility of water-soluble fluorine increased by 2 times, while the acid-soluble fluorine by 1.5 times (Fig. 6).

To determine the intensity of migration of fluorine through a profile, its content was investigated in lysimetric waters. The study of the composition of lysimetric waters allows to determine the degree of anthropogenic influence on soil processes, to identify patterns of movement of contaminants in soils, to evaluate soil as a natural filter for chemical elements and their compounds, as well as to establish their influence on the composition of groundwater, which is formed due to soil runoff (Zeidelman, 2008).
The investigation of the fluorine content in lysimetric waters showed that in case of irrigation (without fertilizing) its contents ranged within 0.13-0.22 mg/dm$^3$, which is by 1.5-3.0 times lower than the fluorine content in irrigation water (0.43 mg/dm$^3$). Thus, field studies of lysimetric waters confirmed the high absorbing capacity of chernozem soils in relation to fluorine compounds contained in irrigation water.

In irrigated areas where phosphogypsum was introduced, the fluorine content in lysimetric waters increased significantly, in some cases reaching over 1 mg/dm$^3$, due to the high migration activity of the microelement element and the additional supply of its compounds with ameliorants (Fig. 7).

The conducted studies are confirmed by the calculations of the fluorine migration activity in the lysimetric waters of experimental sites (during irrigation and introduction of phosphogypsum). The coefficient of water migration of fluorine increased by 2.5-3.5 times during the introduction of phosphogypsum, which can lead to significant contamination of groundwater and its accumulation in plants.

Thus, the degree of mobility of fluorine compounds in the studied chernozem increases with irrigation and especially the introduction of phosphogypsum. The level of fluorine content in lysimetric waters and the coefficient of water migration correlate with its content in soils and can serve as a source of contamination of groundwater and plant products.

Scientists pay considerable attention to the study of fluorine migration in the soil-plant system, especially while applying fluorine-containing fertilizers. However, there is no general opinion regarding the dependence of fluorine content in soils on its content in plants. Natural fluorine is inaccessible to plants, so its content is insignificant. The average fluorine content in plants is 0.1-5.0 mg/kg of dry matter and can range from zero to several hundred milligrams. In areas distant from industrial enterprises, the maximum content of fluorine in plants (depending on the types) is 10-20 mg/kg of dry matter, which makes 0.001-0.002%. Compounds of fluorine entering the soil under conditions of technogenic pollution, unlike natural ones, are easily soluble and can be actively accumulated by plants (Orlov et al., 2002). Due to the high chemical activity and the danger for the health of plants and animals, the content of fluorine in plant products is normalized. The maximal permissible concentrations of fluorine in food of plant origin (bread, vegetables, fruits) is 2.5 mg / kg, in green fodder and hay ~ 20 mg / kg of dry matter. But the general (reserve) content of slow-moving fluorine is normalized. Moving fluoride is dangerous, which is readily stored in living organisms. The introduction of fluorine-containing fertilizers can lead to the accumulation of movable forms of fluorine.

The available scientific literature on the effect of fluorine on crop yields has considerable controversy. Some scientists note the positive effect of fluorine on plants (Pashova, 1980). According to others (Kabata-Pendias and Pendias, 1989) – fluorine is not...
an essential element for plant development. The high content of fluorine in soils is toxic for plants (Nowak et al., 2000; Rezaei et al., 2017) and impairs their growth and properties (Cui et al., 2011). The negative effects of fluorine on plants are manifested, for example, by chlorosis (yellowing) and necrosis of leaves, as well as a decrease in chlorophyll content, resulting in inhibited plant growth (Weinstein and Davidson, 2004). Found that mineral fertilizers increase the fluorine content in the upper soil horizons and agricultural plants. There is no definite relationship between the concentration of soluble fluorine in soil and their content in plants. According to Kabata-Pendias and Pendias (1989) there is no correlation between the bulk fluorine content in soil and plants. Other scientists (Pashova, 1980; Pomazkina, 2004; Tandelov, 2012) have found that mineral fertilizers increase the fluorine content in the upper soil horizons and agricultural plants.

Fluorine-containing fertilizers are one of the main factors for the contamination of agricultural plants with fluorine (outside of industrial exposure). However, the response of plants to the introduction of increased amounts of fluorine into the soil is not straightforward and requires studies of the patterns of absorption of fluorine compounds by plants. The ability of plants to absorb fluorine from the soil is characterized by a biological absorption coefficient (BAC), which shows the ratio of fluorine content in the soil of the plants to the content in the upper (root) horizon of the soil (Perelman, 1989). It is established that the average value of BAC for cultivated plants is 0.2 (Perelman, 1989). However, these definitions concerned the total content of the microelement and not its movable forms.

Our field researches have shown that while irrigation, the content of movable fluorine in various agricultural plants and their particular organs ranged from 0.06 to 0.20 mg/l. With the introduction of phosphogypsum in irrigated chernozem, the content of fluorine in the sap of plants increased significantly − 0.12-0.74 mg/l. When introduced together with mineral and organic fertilizers, the fluorine content ranged from 0.12 to 0.42 mg/l (Fig. 8). Therefore, the introduction of increased doses of fluorine-containing fertilizers leads to an increase of the movable forms of fluorine in all the agricultural plants and their particular organs. In our opinion, this is due to the flow of fluorine directly to their fertilizers even before absorption by the solid part of the soil. A corresponding pattern was also found in the determination of fluorine in soil samples and lysimetric waters.

The calculations of the biological absorption coefficient of the movable forms of fluorine, that is the level of plants' ability to absorb fluorine from the soil, had sufficient differences between particular agricultural plants and their organs (0.05-0.15). The highest index BAC belongs to the wheat ears − 0.33 as well as the corn and oats − 0.22, which were higher than the average values for cultivated plants. Therefore, the ability of plants to absorb soluble fluorine from the soil while applying fertilizer is high, can affect the quality of crop products.

Studies have shown a positive correlation between the content of fluorine in the roots of maize, oats, wheat ears and soils in the absence of fluorine in the stems and leaves.

BAC in plants grown in areas where fluorinated and organic fertilizers were jointly introduced was the

![Fig. 8. The content of fluorine in soils (mg/kg) and plant cell sap (mg/dm³).](image)
forms in the soil. The content of active fluorine in the "southern chernozems-plants-
fertilizers, meliorants and irrigation water creates an
additional risk of contamination with this element of
agricultural production, it is advisable to monitor
systematically the content of water-soluble fluorine in
irrigated soils and crop products.

3. In order to grow ecologically pure crop products
and prevent toxic effects of the microelement on
organisms of animals and humans, it is recommended
to add fluorine to the list of indicators which are
subject to compulsory control in the certification of
agricultural products, especially its soluble forms.

Conclusions.

Thus, the studies carried out show that:

1. Irrigation and chemical melioration is a
significant factor in the differentiation of the territory
by the content of bulk and water-soluble fluorine in
the southern chernozems. Irrigation and introduction
of phosphogypsum leads to the accumulation of
active fluorine in the “southern chernozems-plants-
groundwater “ system. The content of active fluorine
in the plant sap depends on its content of soluble
forms in the soil.

2. As the flow of fluorine with phosphorous
fertilizers, meliorants and irrigation water creates an
additional risk of contamination with this element of
agricultural production, it is advisable to monitor
systematically the content of water-soluble fluorine in
irrigated soils and crop products.

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