

BASIC PROBLEMS OF GEOCRYOLOGY

**EXPERIMENTAL STUDY OF THE ISOTOPIC FRACTIONATION OF WATER
IN THE PROCESS OF ICE SEGREGATION**

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The paper presents the results of the laboratory research of the isotopic composition of water and ice in dispersed soils. It has been established that isotopic fractionation occurs during soil – water interactions, water migration and ice formation during freezing, depending on the chemical composition of soil and freezing conditions.

Stable water isotopes, isotopic fractionation, porous water, water migration, segregated ice

PROBLEM STATEMENT

A great interest in the isotopic composition of ground ice is primarily associated with the successful use of ice cores isotope data in the paleoclimatic reconstructions. Given that the formation of ice bodies from solid precipitates with their isotopic composition dictated by the climatic conditions of the related period of time is in some measure comparable to the formation of ground ice, with atmospheric precipitation also involved in its formation, the formation of ice in soils and conditions for its conservation represent by themselves more complicated processes [Lacelle, 2011]. Despite the discussed complications, great progress has been made in the study of polygonal wedge ice, with the challenges and achievements discussed in detail by Yu.K. Vasilchuk in his monograph [2006]. It should be noted that fairly good results have been obtained due to the accurately established mechanism of polygonal ice wedges formation, mainly from snowmelt water. The isotopic data on other types of ground ice have also been published [Archangelov et al., 1986; Vasilchuk, 1992; Konyakhin et al., 1996; Dereviagin et al., 2003; Streletskaya et al., 2012; Dereviagin et al., 2013] and are available, providing insights into the formation processes, those that are either not clear (massive ground ice), or they are known, but isotopic composition of the water that formed it (segregated ice) is unknown. However, the principles applied to data interpretation on their isotopic composition are generally similar to those applicable to polygonal wedge ice.

Beginning with E.G. Karpov [1986], almost all researchers have observed a heavier isotopic composi-

tion of segregated ice as compared to other types of ground ice. A heavier isotopic composition is interpreted in different ways, but always in the context of changes in the isotopic composition of water caused by its interactions with atmosphere. For example, F. Michel [2011] showed in his paper that the isotopic composition of segregated ice getting heavier with depth is associated with the thermal optimum warming, which time period coincides with the age of lacustrine sediments that provided segregated ice for the investigations. Other authors link this increase in isotopic weight with the evaporation of snow cover prior to entrance of melt waters into the soil [Konishchev et al., 2003], and after it as well [Hagedorn et al., 2010], or with the evaporation of segregated ice following its formation [Mikhalev et al., 2012], which can cause changes in the isotopic composition of ice in the active layer, but that of ice lying deeper remains unaffected.

It is traditionally assumed in the permafrost studies, that the mechanism of ice formation (ice-cement and segregated ice) includes migration of moisture to the freezing front, to be followed by ice segregation [Yershov, 2002], which is not considered in the context of the formation of the texture-forming ice isotopic composition, although it has long deserved a special attention. In turn, moisture migration in the course of freezing is inseparable from the presence of bound water in soils, but this category of water has not yet been analyzed as part of the problem discussed herewith, given a paucity of studies of the isotopic composition of bound water.

BACKGROUND TO THE PROBLEM

In the light of the theoretical assumptions, the isotopic composition of water is determined by ground water – organo-mineral skeleton interactions, mass transfer and phase transitions. As is well known, the properties of bound water, when affected by ions, atoms and molecules that form the soil skeleton surface differ from bulk water properties, which allows for a possibility of any other molecules content therein, including heavy isotopes of hydrogen and oxygen. To some extent, this suggestion is supported by the empirical data presented in the paper by Yu.A. Fedorov [1999].

He showed that a significant isotope fractionation of hydrogen and oxygen takes place in the “adsorbed and constitutional water of clay minerals – water of the sedimentary basin” system. It was found that tightly bound water of clay minerals, at least, the one localized in the monolayer is isotopically lighter by hydrogen and heavier by oxygen, as compared to water in isotopic equilibrium with the clay minerals. In this case, the difference in δD can reach -160% (for sericite) and $+28\%$ in $\delta^{18}O$ (for hydrargillite) compared to the SMOW standard. The fractionation of hydrogen and oxygen isotopes in the course of the formation of crystallization and tightly bound water in clay minerals and their variations, according to Yu.A. Fedorov, is caused mainly by binding energy of water molecules and ions: H_3O^+ , H^+ , OH^- with the solid phase. However, according to V.G. Cheverev [2004], crystallization water and tightly bound water are not involved in the migration and ice formation during freezing, and therefore in these processes the attention should be focused on other categories of water, having lesser bonding with soil surface.

In terms of theory, the state of this portion of ground moisture in relation to the soil particles surface is determined by the van der Waals interactions between the dipoles of water with atoms and molecules on the surface of these particles, as well as the hydrogen or ionic bonds occurring between uncompensated charges on their surface and the ions H^+ , OH^- , H_3O^+ , D^+ , OD^- , H_2DO^+ , formed by the dissociation of water, which suggests it has both high and low content of heavy isotopes. The energy of the van der Waals interaction of molecules of heavy water with the atoms and molecules on the surface of mineral particles appears 10 % higher than in the case with isotopically light molecules. Given dipole-dipole interactions in water are at least an order of magnitude greater than their interaction with atoms and molecules on the surface of mineral particles, the heavier isotope molecules should concentrate in the volume of free or weakly-bound water.

The amount of bound water being relatively small, it is difficult to take this effect as an essential factor in redistribution of molecules with heavy and

light isotopes, analyzing ground water on the whole. The selectivity of interaction of uncompensated surface charges on the soil particles with ions H^+ , OH^- , H_3O^+ , D^+ , OD^- , H_2DO^+ driven by the water dissociation, which caused changes in the isotopic composition of bound and bulk water, can be broadly characterized by the difference in the degree of dissociation of isotopically heavy and light molecules, and mobility of the ions and complexes produced therewith.

Given the ionic product of heavy water is almost an order of magnitude smaller than ordinary water, the mobility of its ions and complexes is also several times lower, so the interaction of ions and light water complexes with uncompensated charges must occur much more frequently and, therefore, their content in the bound water is supposed to be higher. However, the redistribution of heavy and light water molecules, stemming from water–soil interactions, is very limited for the same reason of a small volume of bound water. These assumptions do not provide an unambiguous pattern of the isotopic composition formation for different types of soil moisture, which urged the decision to clarify the situation by means of the experiments.

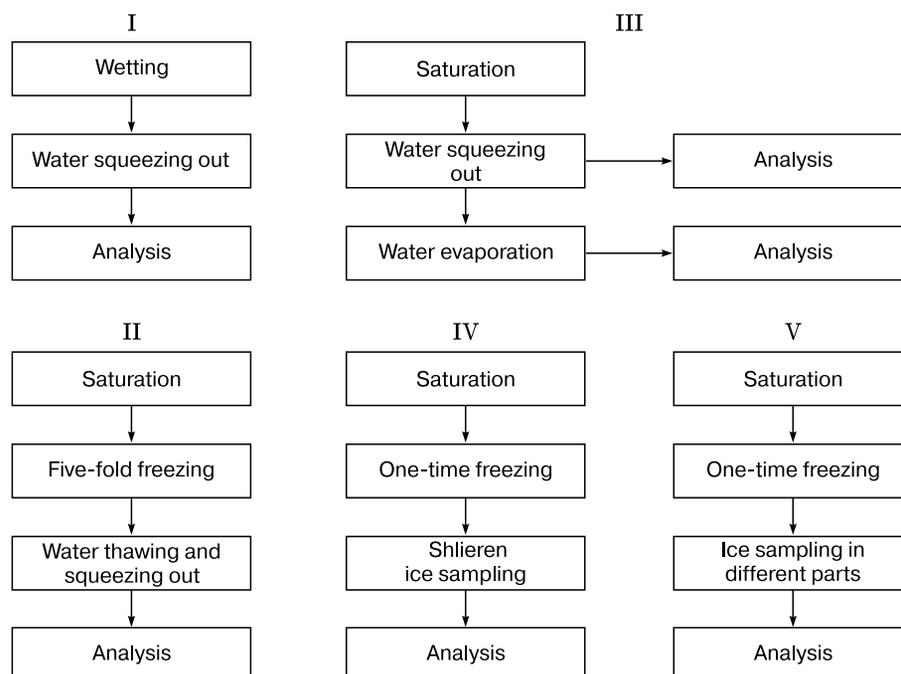
MATERIALS AND METHODS

The experiments were carried out with an aim to estimate changes in the isotopic composition of groundwater of different types using: bentonite from Khakasskoye field, kaolin from the Prosyankovskoye deposits, clay from the Igarka vault, samples of clay loam mantle of Vorkuta region, and silty (yedoma) clay loam, sampled from the Duvanniy Yar section on the Kolyma River. These soils properties have been well studied and are readily available in the literature.

Most of the analytical procedures for determining the isotopic composition of water and ice were carried out in the laboratory for stable isotope geochemistry of the MSU Faculty of Geography, using DELTA V mass spectrometer with standard gas-bench option. The analyses were done by N.A. Budantseva and Y.N. Chizhova under the guidance of Yu.K. Vasilchuk. Another part of the samples was investigated by I.V. Tokarev with the use of Picarro L2120 laser analyzer for isotopic composition of water, at the “Geomodel” Resource Center with St. Petersburg State University.

EXPERIMENTAL SETUP AND RESULTS

Since there were no data available on the isotopic composition of the residual soil moisture in the air-dried samples of natural soils used at the outset of studies, the authors employed the method of consecutive complication of the experimental procedures. The experimental setup is shown in Fig. 1.


Fig. 1. Experimental schemes:

I – first, II – second, III – third, IV – fourth, V – fifth and sixth series of the experiments.

The isotopic composition of free wafer

The purpose of the *first series of experiments* was to determine any possible changes in the isotopic composition of water in volume, when wetting the soil. Equal amounts of air-dry samples of bentonite, kaolin and clay loam were given prolific wetting to (ratio: 3 to1) with distilled water with the known isotopic composition and subjected to continuous stirring for three days. Then, this water was analyzed for oxygen and deuterium isotopes. The results of the analysis of the isotopic composition of the bulk water are shown in Table 1 exhibiting relatively small changes in the isotopic composition of water versus the initial values. $\delta^{18}\text{O}$ values in free water in all the samples increased by 0.3–0.4 ‰, and δD values, up to 3 ‰. The heavy molecules accumulation in free water was taking place in parallel with the deuterium excess (d) reduction.

Table 1. $\delta^{18}\text{O}$ and δD variations in water after wetting the surrounding soil

Sample	$\delta^{18}\text{O}$, ‰	δD , ‰	d
Source water	-14.6	-95.5	21.3
Water after wetting the samples of:			
kaolin	-14.2	-95.5	18.1
bentonite	-14.3	-95.1	19.3
clay loam	-14.2	-93.5	20.1

Soil freezing effect on the isotopic composition of free water

In the light of the current understanding, the water that wets the soil, is hardly involved in the cryogenic migration. The question arises as to what extent the fractionation of isotopes of hydrogen and oxygen confirmed in the first series of experiments, affects the isotopic composition of loosely bound and capillary water taking part in the cryogenic migration in the course of the wet dispersed sediments freezing through [Yershov, 2002].

To deal with this issue, the *second series of experiments* was run with the same soil samples, as in the first one. In the first experiment, the samples were saturated with distilled water at room temperature and cured for one day. In the second, the samples wetted in same manner, were subjected to single freezing-and-thawing, and to thawing-freezing five times (cycle: from -20 to $+20$ °C) in the third. Then, the water was squeezed out from each sample using lever press, to analyze the isotopic composition of hydrogen and oxygen. The isotopic composition of the source water was also known. The experiments results reaffirmed that oxygen and hydrogen isotope fractionation occurs during water– dispersed soil interaction (Table 2).

In this case, the experiments have shown a correlation between the clay mineral properties and the degree of fractionation. The removed water appeared

Table 2. $\delta^{18}\text{O}$ and δD variations in squeezed out moisture in various types of soils

Soil	W_{init} , %	W_{squ} , %	$\delta^{18}\text{O}$, ‰	δD , ‰
<i>Wetted sample</i>				
Kaolin	77.2	50.2	-11.2	-79.1
Bentonite	92.0	54.8	-10.8	-77.4
Clay loam	32.4	24.2	-11.2	-79.7
<i>Wetted and frozen sample</i>				
Kaolin	62.8	47.7	-11.5	-73.1
Bentonite	97.9	55.4	-8.8	-73.1
Clay loam	33.9	25.8	-10.5	-73.8
<i>Wetted and fivefold frozen sample</i>				
Kaolin	81.2	54.2	-10.2	-71.8
Bentonite	94.5	59.6	-9.2	-72.3
Clay loam	33.8	23.7	-10.6	-75.5

Note. Isotope content in source water $\delta^{18}\text{O}$ -12.6 ‰; δD -89.0 ‰. W_{init} - initial moisture content in samples, ‰; W_{squ} - moisture in samples after squeezing, ‰.

to have heavier composition both by oxygen and hydrogen in all the soils used in the experiments. The removed water is very likely to be composed of mainly porous (immobilized) water, i.e. the water least affected by the surface forces of mineral skeleton.

It would be reasonable to assume that water remaining in the samples is a mixture of different categories of water, however, the soil-bonded water tends to account for a much greater portion. According to the experimental data, this water (termed "bound") consists mostly of a lighter isotopic composition fraction. The fractionation degree varies in different soils: it is slightly larger with bentonite, than with kaolin and clay loam, which may have been caused by its greater adsorption capacity. This general suggestion is reaffirmed, although not unambiguously, by single and especially fivefold freezing of wet soil.

Thus, the results of experiments with the regimes of samples freezing indicate that the isotope fraction-

ation of hydrogen and oxygen water may increase, depending on the soil properties and conditions of freezing, with its degrees varying for the different isotopes of water. In this case, the bound water (capillary and osmotic) in soils tends to be easier in their isotopic composition, as compared to the original.

Isotopic composition of water

To further verify the assumptions made, the *third series of experiments* was run. In order to determine the isotopic composition of water remained after the mechanical removal of moisture, the samples were evaporated. To do so, they were placed in a hermetically sealed system, with one container with the sample placed in the drying cabinet, and the other linked to the first vessel through the spiral tube cooler, was used to collect the condensed water vapor. The analysis was performed for two samples differing, firstly, in the predetermined humidity. The results showed that water remaining in the soil after squeezing and therefore having a greater ability to binding to soil particles, is always easier than that squeezed out, and it appeared to be easier even than source water for some soils (Table 3).

Alterations in ^{18}O and D contents at phase change in soil

When assessing the isotopic composition of the texture-forming ice it is necessary to consider not only the composition of the source water, but the factor causing alterations in the isotopic composition of water during its phase change. This problem is addressed to by *Yu.K. Vasilchuk* in his paper [2011]. He showed that during freezing of the ice forming bulk water the ice tends to be isotopically heavier by at least 1-3 ‰ (for oxygen), than the source water. Deuterium behavior is assumed to be more complicated. According to *K.L. Danilov et al.* [2009], deuterium fractionation degree is higher vs. oxygen, and the magnitude of deuterium enrichment may increase at higher freezing rates, and decrease at lower.

Table 3. $\delta^{18}\text{O}$ and δD in water from different categories of various groups

No.	Sample	Water recovery method	W, %	$\delta^{18}\text{O}$, ‰	δD , ‰	d
1	Initial water			-12.43/-12.3	-82.7/-89	16.7/9.4
2	Yedoma clay loam	Squeezed	30/25	-11.99/-	-90.3/-	5.62/-
3	Yedoma clay loam	Evaporated		-12.41/-	-91.6/-	7.68/-
4	Clay loam mantle	Squeezed	30/24	-11.61/-12.1	-90.1/-89	2.78/7.8
5	Clay loam mantle	Evaporated		-16.99/-13.0	-119.5/-94	16.42/10.0
6	Kaolin	Squeezed	250/200	-12.24/-8.5	-92.15/-76	5.77/-8.0
7	Kaolin	Evaporated		-14.42/-12.9	-108.5/-102	6.86/1.2
8	Bentonite	Squeezed	600/400	-2.95/-10.0	-75.1/-82	-51.5/-2.0
9	Bentonite	Evaporated		-12.75/-10.4	-89.6/-81	12.4/2.2

Note. Values for predetermined sample moisture (W) and isotope content were defined using DELTA (the numerator) and Picarro (the denominator) analyzers.

Table 4. $\delta^{18}\text{O}$ and δD variations in shlieren of segregated ice formed in the course of freezing

Sample	$\delta^{18}\text{O}$, ‰	δD , ‰	d
Source water	-14.6	-95.6	21.2
Ice in kaolin	-14.0	-93.9	18.1
Ice in bentonite	-12.3	-88.3	10.1
Ice in clay loam	-13.5	-96.7	11.3

The isotopic analysis of segregated ice inclusions (the fourth series of experiments) was carried out, to verify these provisions, and the results allowed for an inference, that the oxygen isotopic composition of such inclusions of ice is heavier than that of source water (Table 4). The data on deuterium showed that kaolin and bentonite tend to “grow heavier”, while clay loam “grow lighter”. Provided that coarsely dispersed soils (in our instance, clay loam) freeze faster than finely dispersed (clays – kaolin and bentonite), it agrees well with the inferences made by *K.L. Danilov et al. [2009]* on how the water freezing rates and volume affect differentiation of its isotopes. The results of this series of the experiments have shown that the differentiation takes place in a manner very much similar to the bulk water freezing.

Freezing conditions in the cryogenic migration influence on the segregated ice composition

It is obvious that for better understanding of the varying isotopic composition of bound water and segregated ice, the processes of moisture migration and ice formation should be studied integrally. To this end, a one-time freezing was applied to the soil samples in the next series of experiments, which allowed the samples to freeze at the predetermined rate of 2.5 cm/day (the fifth series of experiments). Ice and water were sampled in different portions: schlieren, frozen massive and incompletely frozen. The experiments results show that when entering into the soil, water is subjected to a series of alterations, which is reflected in its isotopic composition (Table 5). Lesser bound (interstitial) water appears to be prevailing in the incompletely frozen portion of the samples, with its isotopic composition being heavier, as shown above.

The middle portion of the sample, which according to the experimental conditions was freezing later than at the top, had a massive texture. Relying on the data from Table 5, moisture migrated to the uppermost part of the sample, where the freezing commenced and schlieren formed. Its isotopic composition proved lighter than even that of source water, which agrees with the hypothesis on a lighter isotopic composition of bound water (Table 5). The schlieren part of the sample (the largest ice formation area) has a heavier composition. It’s easier than the source water composition, though. Probably, it’s the law of

Table 5. $\delta^{18}\text{O}$ and δD variations in different parts of the studied samples

No.	Sample	$\delta^{18}\text{O}$, ‰	δD , ‰	d
1	Initial water	-13.56	-100.3	8.1
<i>Kaolin freezing at W = 100 %</i>				
2	Shlieren texture	-13.30	-95.4	11.0
3	Massive texture	-14.35	-106.4	8.4
4	Non-frozen part	-12.93	-98.6	4.8
<i>Kaolin freezing at W = 80 %</i>				
5	Shlieren texture	-11.11	-88.0	0.8
6	Massive texture	-14.09	-98.0	14.7
7	Non-frozen part	-13.46	-98.6	9.1
<i>Yedoma clay loam freezing at W = 30 %</i>				
8	Shlieren texture	-12.93	-93.4	10.1
9	Massive texture	-12.66	-92.56	8.7
10	Non-frozen part	-12.44	-85.66	13.8

isotopic composition weighting in the course of water-to-ice transition that works there. These patterns are well traced by the example of kaolin, while in the yedoma clay loam they are very weak, which can account for inconspicuous migration, and very low ice content in the clay loam (the schlieren that formed has a thickness of a fraction of a millimeter).

A more differentiated sampling was done for the isotopic analysis in the experiments to follow in the fifth series. The samples were taken directly from the ice schlieren and from interschlieren gaps, as far as it was technically possible (Table 6). In this case, the picture appears to be more complicated, but the basic patterns can be discerned here as well: the isotopic composition of the ice schlieren proves heavier than in the ice-cement from the interschlieren intervals (Fig. 2).

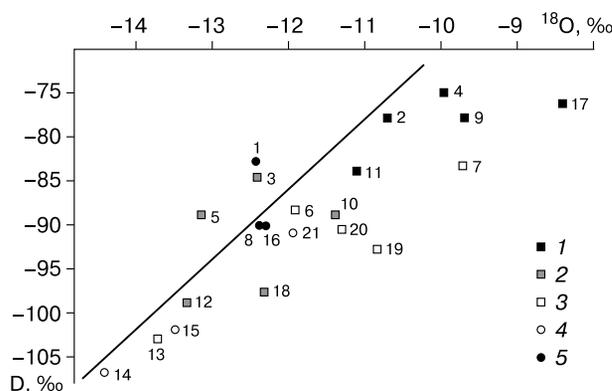


Fig. 2. Distribution of water isotopic composition values in the samples from the fifth series of the experiments (Table 6).

1 – slieren ice; 2 – interschlieren ice-cement; 3 – ice-cement in the massive part; 4 – water in the thawed portion; 5 – source water.

Table 6. $\delta^{18}\text{O}$ variations δD in different parts of frozen soil samples

No.	Sample	$\delta^{18}\text{O}$, ‰	δD , ‰	d
<i>Kaolin freezing at W = 100 %; DELTA V analyzer data</i>				
1	Source water	-12.43	-82.7	16.7
2	Shlieren ice from upper part	-10.71	-78.2	7.44
3	Intershlieren ice-cement from upper part	-12.40	-84.8	14.36
4	Shlieren ice from lower part	-9.96	-75.1	4.58
5	Intershlieren ice-cement from lower part	-13.13	-88.9	16.14
6	Ice-cement from lower massive part (top)	-11.93	-88.5	6.94
7	Ice-cement from lower massive part (base)	-9.71	-83.4	-5.76
<i>Kaoline freezing at W = 80 %; Picarro L-2120 analyzer data</i>				
8	Source water	-12.4	-90	9.2
9	Shlieren ice from upper part	-9.7	-78	-0.4
10	Intershlieren ice-cement from upper part	-11.4	-89	2.2
11	Shlieren ice from lower part	-11.1	-84	4.8
12	Intershlieren ice-cement from lower part	-13.3	-99	7.4
13	Ice-cement from lower massive part	-13.7	-103	6.6
14	Water from non-frozen part (top)	-14.4	-107	8.2
15	Water from non-frozen part (base)	-13.5	-102	6.0
<i>The Igarka clay loam freezing at W = 50 %; DELTA V analyzer data</i>				
16	Source water	-12.4	-90.0	9.2
17	Shlieren ice	-8.41	-76.2	-8.92
18	Intershlieren ice-cement	-12.32	-97.8	0.76
19	Ice-cement from lower massive part (top)	-10.84	-93.0	-6.28
20	Ice-cement from lower massive part (base)	-11.34	-90.3	0.38
21	Water in the underlying sand	-11.86	-92.0	2.84

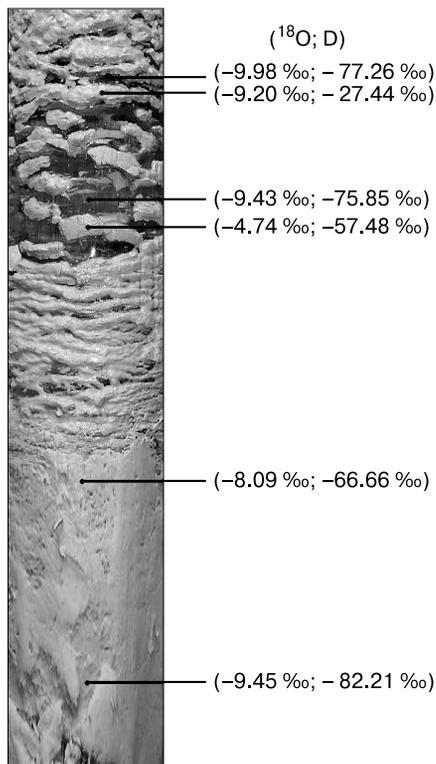


Fig. 3. Distribution of samplings and the isotopic composition values in the sixth series of the experiments (^{18}O ; D).

The isotopic composition of segregated ice affected by freezing rates

Moisture migration, ice segregation and nature of ice textures are known to be dictated not only by the soil properties (particle-size composition (texture), moisture content), but in a substantial way by the freezing rates. The isotopes differentiation in the course of freezing is likely to be affected by this factor as well. To verify the fifth series of experiments, they were repeated at a lower freezing rate – up to 1 cm/day (*the sixth series of experiments*). In this case, the ice schlieren formed in kaolin were somewhat larger, than in the previous case, and a 12 mm thick schlier was formed on the boundary of the schlieren layer and massive cryostructure (Fig. 3). This allowed for more accurate sampling directly from the schlieren and from a thin layer of frozen soil exactly beneath the schlier. The results of these experiments turned out to be somewhat different (Table 7): ice of massive texture underlying the ice schlier was heavier than the schlier ice, and the difference proved to be fairly essential under the second schlier. Apparently, at slower freezing rates, the possibility of light bound water migration in the freezing soil proves to be so great, that all of its reserves are used up in the formation of schlieren, and they become depleted in the schlier-feeding layer, underlying the growing schlier.

Table 7. $\delta^{18}\text{O}$ and δD variations in different parts of kaolin sample at predetermined freezing rate 1 cm/day and moisture content 100 %

No.	Sample	$\delta^{18}\text{O}$, ‰	δD , ‰	d
1	Source water	-10.50	-78.68	5.32
2	Shlieren ice from upper part	-9.98	-77.26	2.58
3	Intershlieren ice-cement from upper part	-9.20	-27.44	46.16
4	Shlieren ice from lower part	-9.43	-75.85	0.41
5	Intershlieren ice-cement from lower part	-4.74	-57.48	19.56
6	Ice-cement from lower massive part	-8.09	-66.66	1.94
7	Water from thawed part	-9.45	-82.21	5.01

DISCUSSION OF THE RESULTS

A hypothesis on the presence of tightly bound water with heavy isotopic composition in the “dry” soil [Fedorov, 1999] could be the simplest explanation for the observed alterations in the isotopic composition of water after its wetting the soil and blending with the makeup water. In such a case, the water squeezed out from the soil after wetting it may turn out heavier (in terms of the isotopic composition), than water used for the soil wetting. In the course of evaporating, the isotopic composition of the residual water can be expected to be easier than that of the water used for wetting, provided some of the heavy tightly bound water preserved in the soil after evaporation.

Providing elucidations on some of the results obtained, the scenario proposed above implies a fairly free exchange between the tightly bound and free water, which does not agree with the well-known difference in their mobility. Moreover, it does not explicate the isotopic composition of ground ice profiles obtained in the experiments on the controlled freezing of soils and oriented towards the freezing that produced alternating lenses and textures. The isotopic composition of all lenses were always lighter than that of the water used for wetting the samples, but heavier than bound water migrating towards the freezing front. The profiles have shown considerable variability in the isotopic composition of ground ice inclusions of various types and proved the dependency of the isotopic composition on the activity of moisture migration to the freezing front. This variability can hardly be explained only by variations in the isotopic composition as a result of phase change.

A more universal explanation consists in the fact that “light” water molecules have an advantage in the formation of bound water, which is more mobile than water with heavy isotope composition. Given that light water migrates to the freezing front, it fractionates again in the course of the crystallization (the formation of schlieren), depending on the soil composition and freezing conditions.

Thus, the obtained data indicate that the water – dispersed soil interactions may result in the variability

of heavy isotopes of oxygen and hydrogen both in free (with the isotopic composition growing heavier) and bound (with lighter isotopic composition) water. Such a change is not currently taken into account when considering the processes of formation of the isotopic composition of ground ice, while this process is quite complicated and is dictated by the properties of soil, freezing conditions and the intensity of moisture migration.

CONCLUSIONS

1. The isotopic composition of bound water is easier (according to the experiments, oxygen: from 0.5 to 9.8 ‰ and deuterium: from 1.3 to 29.4 ‰), than free water filling in the pore space (immobilized water).

2. The formation of the isotopic composition of the ice-cement and schlieren ice in segregated ice is dictated by several factors: source water composition; water–fine-grained soil interactions; freezing conditions and soil moisture migration; nature of ice formation. Defining the role and degree of each of the factors involved in the formation of the isotopic composition of the texture-forming ice requires further experimental and field studies.

3. The interpretation of isotopic data on the texture-forming ice with an aim to determine its origin and build paleogeographic reconstruction should include intrasoil metamorphism of the isotopic composition of water.

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