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DETERMINATION OF THE FREEZING POINT OF SOILS BASED ON MEASUREMENTS OF PORE WATER POTENTIAL

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The soils freezing point is usually determined by both the direct method of slow cooling with the temperature monitoring during ice crystallization in a supercooled system, and indirect methods using other measured parameters of the soil system with further calculation. The water-potentiometric method for soils freezing point determination based on a single measurement of a pore water potential with subsequent thermodynamic conversion into an equivalent temperature has been developed. This approach is one of the most promising indirect methods due to high productivity and its accuracy is comparable to direct measurements. The results have demonstrated good comparability of the obtained data by the water-potentiometric method with the direct measurements of the freezing point for the same soils. The values difference between the two methods is no more than 0.05 °C for all investigated permafrost soils with different particle size distribution, salinity and moisture content. The water-potentiometric method can be recommended for the freezing point determination applied to soils with natural moisture and salinity along with methods of direct experimental determination.

Soils, freezing point, water-potentiometric method, natural moisture content, pore water potential, pore water activity, salinity, Yamal Peninsula

INTRODUCTION

Water-saturated soils are multi-component systems, the transition of which from the thawed state to the frozen one occurs within the range of temperatures below zero Celsius. At the same time, the freezing point of pore water in porous media can be significantly different from 0 °C, which is due to the mineralization of the pore solution and its interaction with the soil skeleton. The freezing point corresponding to the temperature of the ice appearance in the pore space is an important soil characteristic and used in assessing the depth of soil freezing or thawing in permafrost engineering surveys.

The freezing temperature of non-saline sandy and coarse-grained soils in some cases (as an estimated one) can be taken equal to –0.1 °C, and for clay soils it can be assumed as –0.25 °C [*SP 25.13330.2012, 2012*]. However, the freezing point of saline and organic-reach (peat) soils should be determined empirically. In practice for geotechnical calculations the recommended values of the freezing point are often used, for example, those given in *SP 25.13330.2012* [*2012*]. They are determined taking into account soil particle size distribution (sandy, sandy loam, loamy and clayey ones) and the pore water mineralization (salinity), which is calculated based on the degree of salinity and the total moisture content of the soil. At the same time, the higher clay content and salinity manifest the lower freezing point.

It should be noted that the freezing and thawing temperatures of soils may be slightly different. For example, the thawing temperature of fine-grained soils is tenths of a degree higher than the freezing point, which can be explained by the influence of the capillary effect during the freezing [*Saveliev, 1989*]. However, with cyclic freezing-thawing of soils, those temperatures cease to differ practically, which is probably associated with structural and textural transformations in soils. Therefore, in practice, only the term 'freezing point' is usually used, implying that its value is practically equal to the thawing temperature of the soil.

ESTIMATION OF THE FREEZING (THAWING) TEMPERATURES OF SOILS

Methods for determining the freezing point of soils can be divided into the experimental and calculated ones. It is the experimental determination of the freezing–thawing temperature that has traditionally been given the primary role from the beginning of the permafrost study (Geocryology). At the same time, the experimental method for the freezing point determination is being improved in accordance with the development of the hardware base for fixing the temperature, being determined by the sensitivity of the measuring devices used as temperature sensors [*Andrianov, 1936; Bozhenova, 1954; Tsytovich, 1973; Ershov, 1985, 2004*]. It should be noted that the method of direct determination of the freezing point, developed by the Russian geocryologists in the middle of the 20th century, is used by specialists in other scientific fields to determine the freezing point of various liquids, including aqueous solutions of various substances [*Ershov, 2004; Gevorkyan, 2017*].

Initially, the thermometers with visual control of temperatures and discrete manual recording of results (often depending on the experimenter's efficiency) have been used to study the freezing temperatures of soils. Thermometers have been replaced by various types of temperature sensors (thermoelectric, thermoresistive, semiconductor, acoustic, piezoelectric ones), and it became possible to record temperatures in a continuous mode, owing to the introduction of electronic potentiometers, as well as to apply automatic processing of results. However, the sensitivity of the sensors and the automation of the measurement process have only increased the accuracy of fixation of temperatures in time, and the experimental technique for determining the desired value have remained practically unchanged.

As is well known, the freezing point (i.e. temperature of pore water crystallization in the soil) is determined by the 'shelf' on the temperature curve, which is recorded immediately after the temperature jump resulting from the beginning of freezing of the supercooled soil [*Ershov, 2004*]. In practice, depending on the composition of the soil and the rate of cooling, the supercooled state of the system can be lengthy. Therefore, researchers sometimes determine the temperature of thawing of frozen ground, since in that case there is no effect of overheating of the system. However, certain problems arise here associated with the presence of a temperature field gradient [*Ershov, 2004; Teng et al., 2020*]. Note that the experimental and technical base for conducting experiments during freezing and thawing remains practically the same.

In the literature, interesting attempts are made to use new technical solutions for the direct measurement of the freezing point of the soil water, for example, an original method of 'beginning of crystallization' has been recently proposed [*Kolunin, Ishkova, 2019*], which, however, can only be applied to samples of a homogeneous structure, for which it has been developed.

The freezing point of the soils with known moisture content can also be determined by the curve of the dependence of the unfrozen water content on the negative Celsius temperature obtained for the given soil within a wide temperature range [*Sargsyan et al., 1973*]. Currently, many different methods, both experimental and calculated ones, are used to determine the unfrozen water content. At the same time, new, more accurate and faster methods continue to appear, and the previously proposed methods continue to develop and improve [*Chuvilin et al., 2020*]. However, it should be noted that the determination of the freezing point by the curve of unfrozen water is a longer and more laborious way than direct measurements.

To date, a lot of experimental work has been done to determine the influence of various factors on the freezing point of soils, such as particle size distribution, mineral composition, salinity, the organic matter content and various pollutants [*Ershov, 1996; Motenko, Grechishcheva, 2016; Aleksyutina, Motenko, 2017; Li et al., 2020; Teng et al., 2020*], as well as to define the effect of the freezing temperature on the strength and deformation characteristics of the frozen soils [*Roman et al., 1994*].

Along with experiments, many researchers proposed calculation formulas for assessing the freezing point of soils, using various correlations with the physical characteristics of soils. Thus, based on the statistical analysis and generalization of the experimental results obtained by the calorimetric method, an empirical formula for calculating the freezing temperature was proposed, taking into account the values of the plastic limit and the total moisture content of the studied soils [*Kozlowski, 2004, 2007, 2016*]. Also, the freezing point was estimated on the basis of the Clapeyron–Clausius equation by adding various additional parameters and empirical coefficients [*Koopmans, Miller, 1966; Kurylyk, Watanabe, 2013; Zhou et al., 2018*]. The freezing point at the same pore water content can depend on a number of factors, such as mineral composition, grain and pore size distribution, specific surface area and content of dissolved substances [*Kozlowski, 2004, 2007; Zhou et al., 2018; Wang et al., 2020*], and even the sample masses [*Kozlowski, 2009*], which requires further refinement of the calculation formulas and a clear definition of the limits of their applicability.

Returning to the methods of direct determination of the freezing (thawing) temperature of soils, it should be noted that they involve the use of various experimental installations, an obligatory element of which are freezing devices (thermostat or refrigerator), where soil samples are placed, as well as sensors of various modifications for recording temperature. However, methods of direct determination of the freezing (thawing) temperature require significant time expenditures (up to 6 hours or more), as well as the need for placement, connection and continuous operation of refrigeration equipment, which is not always possible to ensure.

Accordingly, there is a necessity to develop operational indirect methods for soil freezing point determination. As such a method, we propose the use of a water-potentiometric method for determining the freezing (thawing) temperature of soils based on a single measurement of the potential of pore water in a sample. To substantiate the practical use of the proposed method, a comparative analysis of the data on determining the freezing (thawing) temperature of soils, obtained by the traditional experiment during cooling and heating of soil samples, and a method based on measuring the moisture potential in the investigated samples with further thermodynamic conversion of the obtained parameter into the freezing point, has been carried out.

EXPERIMENTAL STUDY TECHNIQUE

The technique adopted in the work included the use of two methods for assessing the freezing-thawing temperatures of soils: a direct method for freezing point measurements based on using of the Kriolab Tbf experimental setup developed by KRIOLAB LLC, and a water-potentiometric method based on a one-time measurement of the pore-water potential of a soil sample with natural or specified moisture content.

Technique of experimental determination of the freezing (thawing) temperature of soils. Experimental determination of the freezing point of soils was carried out on the KrioLab Tbf equipment with special software. This setup consisted of a special mobile refrigerator-freezer, in which a container with the soil (diameter of 30 cm and a height of 40 cm) was placed (Fig. 1). A temperature sensor was inserted into the soil container through a hole in its lid, sealed into a needle sleeve with an outer diameter of 3 mm, connected to a thermo-chain, which was connected to a splitter. Through it, the thermo-chain produced by KRIOLAB LLC was connected to the ADC and then via a USB cable to the computer [*Manual..., 2019*]. The temperature sensors in the installation were calibrated with an accuracy of ±0.01 °C in a liquid thermostat using a special reference temperature sensor.

For determining the freezing point of the soil, a temperature in the mobile freezer was being maintained of –5...–10 °C, which was always lower than the temperature of possible supercooling of the soil. When determining the temperature of thawing of frozen soils, the experiments were being carried out at temperatures 1–5 °C higher than the expected temperature of complete thawing of the soil.

Determination of the freezing (thawing) temperature was carried out as follows. The containers (sample-bottles) were tightly filled with the test soil in order to maximally exclude the possibility of distortion when measuring the temperature with a sensor, which was installed in the geometric center of the bottle. The weighing bottles equipped with sensors were placed in a freezer with a preset temperature, after which the sensors were connected to a computer and the software was started in the mode of continuous recording of measurements. The completion of measurements in the freezing cycle was carried out with the readings of the temperature sensors in the samples close to the temperature set in the freezer, and maintaining that temperature of the samples for a long time. For the subsequent determination of the thawing temperature, the weighing bottles with samples were removed from the freezer, and the measurement of the temperature of the soil in the bottles was continued by means of the sensors installed in them until the end of the thawing cycle. At the end of the measurements, the moisture content of the samples was monitored.

The freezing point during the processing of the results in the freezing cycle was on the obtained diagram along the ‘shelf’, i.e. a temporary area with practically zero temperature gradient. The thawing temperature was determined as the point of intersection of two tangents to the sections of the initial stage of melting of pore ice and subsequent stage of intensive thawing of the sample.

The measurement time was more than 12 hours when determining the freezing point (when freezing samples from room temperature to –10 °C), and at least 8 hours when determining the thawing temperature (when defrosting from –10 °C to +10 °C) [*Manual..., 2019*].

Technique for determining the freezing (thawing) temperature of soils by the water-potentiometric method based on a single measurement. The technique for determining the freezing point of soils by the water-potentiometric method is based on measuring the potential of pore water in the studied soil sample with subsequent thermodynamic conversion of the pore-water potential of into an equivalent

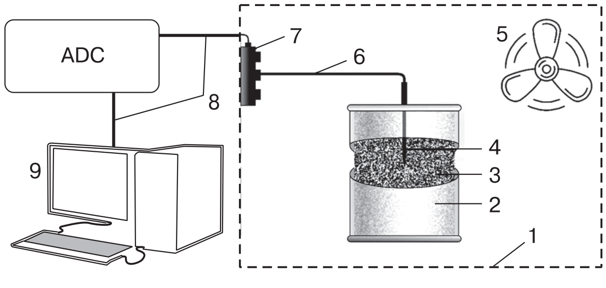

Fig. 1. Principal scheme of the Kriolab Tbf setup for measuring the freezing (thawing) temperature of soils:

1 – freezer; 2 – container for soil; 3 – soil; 4 – temperature sensor; 5 – fan; 6 – thermo-chain; 7 – hub for connecting additional temperature sensors; 8 – USB cable; 9 – computer with the Kriolab Tbf software.

temperature at which the pore water is in equilibrium with ice in the free volume [*Istomin et al., 2009*]. Moreover, all measurements are carried out at the positive Celsius temperature and do not require special refrigeration equipment for the sample preparation.

The proposed method for determining the freezing point is actually an express method. That is achieved by quickly measuring the moisture potential values of the test sample, and by using a scheme for converting the pore water potential values through its activity into the freezing point.

The experimental measurement of the pore water potential was carried out using the WP4-T or WP4-C instruments [*Istomin et al., 2008, 2009, 2017a*] developed by the Decagon Devices (USA) [*Campbell et al., 2007*] (Fig. 2). The measuring system of those devices is based on the determination of the water vapor pressure over wet soil using the dew point method [*Campbell et al., 2007*]. The used devices allow obtaining the values of the pore water potential within the temperature range of from 15 to 43 °C. The error in measuring the potential of pore water within the range of 0 to –5 MPa is ±0.05 MPa, and within the range of from –5 to –50 MPa it can increase to ±1 % [*Chuvilin et al., 2020*].

Recalculation of the measured potential value of pore water (φ) into the activity of pore water (α) is carried out according to the ratio:

$$\varphi = \frac{RT\rho}{M} \ln \alpha. \quad (1)$$

Where R is the universal gas constant (8.314 J/(mol·K)); T is the temperature of the investigated sample of the porous medium, K; M is the molecular weight of water (18.015 g/mol); ρ is the density of water (1.0 g/cm^3).

Taking into account all types of errors, the measurement technique makes it possible to obtain activity values with an accuracy of 0.0005 at $0.9 \leqslant \alpha \leqslant 1.0$, and with that of about 0.0025 at $0.7 \leqslant \alpha \leqslant 0.9$ [*Chuvilin et al., 2020*].

To measure the potential of pore water in devices of the WP4 type, soil samples with natural or specified moisture were placed in the measuring cup of the device, with the internal dimensions of 3.8 cm in diameter and 1.0 cm in height. Wet soil of disturbed structure was evenly distributed over the cup's bottom so that a soil pellet about 0.5 cm high was obtained (that size is due to the specifics of the operation of the measuring system of the device). Undisturbed samples should also have a cylindrical shape with a diameter of about 3.8 cm and a height of about 0.5 cm. The initial moisture content of a soil sample was determined by weighing it on an electronic balance with an accuracy of ±0.003 g, and the moisture control was carried out before and after measuring the potential of pore water. For the known value of the moisture content of the sample, the value of the potential of the pore water was initially measured on devices of the WP4 type, then the values of the pore water activity were calculated using the ratio given above. Subsequently, the activity values were recalculated into the equilibrium temperature, which actually represented the freezing temperature at a given value of the soil sample moisture. The recalculation of the obtained values of pore water activity (α) within the range of from 1.0 to 0.6–0.7 into the equivalent temperature (t_{eq}, °C) was carried out according to the formula [*Istomin et al., 2017a,b; Patent RU 2654832 C1, 2018*]:

$$t_{\text{eq}} = 103.25 \ln \alpha + 5.57\,(1 - \alpha)^2. \quad (2)$$

The obtained temperature value is the freezing (thawing) temperature of the studied soil sample with given initial moisture content.

The time of obtaining one value of the pore water potential in the WP4 device (the time for equilibration in the measuring chamber between the water in the sample and the air containing water vapor) is usually about 20–30 minutes. Only for clayey soils with low humidity, it can increase up to 1 hour. During measurements, the WP4 device was connected to a computer via the standard Hyper Terminal program

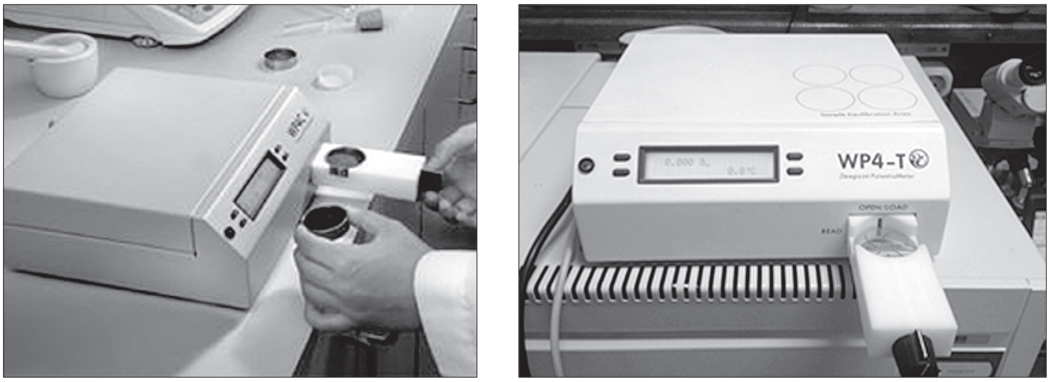

Fig. 2. The WP4-T device of the Decagon (USA).

Table 1. **Brief description of the investigated soils**

Sample number	Sampling depth, m	Name of soil*	Water content, %	Plasticity index	Salinity, %	Soil freezing point**, °C
1	1.7–2.0	Light clay with organic matter	74.8	24.3	1.10	−1.0
2	4.0–4.3	Silty sand	23.3	–	0.29	−0.8
3	4.8–5.0	Fine sand	28.5	–	0.22	−0.5
4	7.5–7.7	Light loam with peat	33.9	9.7	2.71	−4.1
5	11.1–11.3	Silty sand	23.7	–	0.43	−1.1
6	14.0–14.5	Fine sand	20.2	–	0.29	−0.9
7	24.5–24.7	Light loam	14.9	8.8	0.50	−2.0
8	33.6–33.9	Light loam	18.6	8.5	0.54	−1.8

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* [*GOST 25100-2011, 2013*].

** Calculated by [*SP 25.13330.2012, 2012*].

to automate the process of receiving, accumulating and processing data.

Thus, the presented method for determining the freezing point makes it possible to carry out a large number of measurements with high accuracy, to obtain data on freezing (thawing) temperatures for several tens of individual soil samples within a wide range of chemical and mineral composition and particle size distribution within one working day.

Characteristics of research objects. Methodological studies using two methods for determining the freezing point were carried out on natural soils typical of the upper horizons of permafrost soils of the Yamal Peninsula. Cores of frozen soils were taken from the boreholes up to 34 meters deep (Table 1), drilled during engineering surveys on the territory of the South Tambey gas field.

The soils used for the comparative determination of the freezing temperatures by different methods are represented within a wide range of clay particles content (sand, loam, clay) and salinity, which in the studied samples differs by more than an order of magnitude and varies from 0.22 to 2.71 % (Table 1).

To determine the freezing temperatures, soil samples with natural moisture content were collected. Clay samples obtained from the upper part of the section from a depth of about 2 m were characterized by the maximum moisture content (about 75 %). Down the section, a decrease in natural moisture was noted, while the minimum value (about 15–19 %) was characteristic of loam from the depths below 24 m (Table 1). At the same time, the density of soils varied from 1.25 g/cm^3 (in clay samples out of the upper part of the section) to 1.90–1.95 g/cm^3 in silty sands, and the density of dry soil varied in the samples by more than 1 g/cm^3 from 0.56 to 1.59 g/cm^3, and it was the more, the lower the moisture content of the samples. The porosity coefficient of clay also had maximum values 3.64, decreasing to 0.9 in loam, and to 0.7–0.8 in sandy samples. Soil particle density was 2.61–2.66 g/cm^3.

According to the data of X-ray diffraction analysis performed on a DRON 3 diffractometer using monochromatic CuK$_\alpha$-radiation, in all studied soils in the sandy fraction, the predominant mineral was quartz (up to 71 % in fine sand), plagioclase (up to 19 %) and potassium feldspars (up to 14 %). The clay fraction of the studied soils was represented by chlorite, illite and kaolin. In addition, the presence of amphibole, pyroxene, as well as traces of cristobalite, goethite and pyrite (no more than 1 %) was noted in the soils. The data on the mineral composition of the studied samples, obtained by the method of profile processing of X-ray patterns from non-oriented preparations and the calculation of the number of identified phases by the method of reference intensity ratio (Chung's method, the internal standard method), demonstrated a certain similarity, which is expressed in close values of the content of the main rock-forming minerals, which may indicate similar initial conditions of sedimentation.

RESULTS AND DISCUSSION

Based on two methods described above, the freezing (thawing) temperatures of soil samples with natural moisture have been determined and a comparison has been made, which has revealed good convergence of the results (Table 2).

As can be seen from Table 2 above, the experimental freezing point of the soils, measured using high-precision temperature sensors, practically coincided with the freezing temperature calculated from the pore water potential measured in the same samples. In the considered soils, the difference between the experimental and calculated values did not exceed 0.05 °C and is comparable with the accuracy of each method. It should be noted that for most experimental installations, including for determining the freezing point of soils, the accuracy of temperature measurements should be no worse than ±0.1 °C, which is sufficient for solving standard geocryological and geotechnical problems [*Ershov, 2004*].

Table 2. **Comparison of freezing temperatures obtained by experimental and calculation methods for the studied soils**

Sample number	Name of soil	Water content, %	Pore water potential, MPa	Pore water activity, fraction of units	Calculated freezing point, °C	Measured freezing point, °C	Difference in freezing point values, °C
1	Light clay with organic matter	74.8	−0.90	0.9935	−0.67	−0.68	+0.01
2	Silty sand	23.3	−0.73	0.9947	−0.55	−0.52	−0.03
3	Fine sand	28.5	−0.66	0.9952	−0.49	−0.49	+0.00
4	Light loam with peat	33.9	−3.79	0.9728	−2.84	−2.81	−0.03
5	Silty sand	23.7	−1.78	0.9872	−1.33	−1.31	−0.02
6	Fine sand	20.2	−1.48	0.9893	−1.11	−1.16	+0.05
7	Light loam	14.9	−1.63	0.9882	−1.22	−1.17	−0.05
8	Light loam	18.6	−0.99	0.9928	−0.74	−0.70	−0.04

If the freezing point values obtained by the two methods are rounded to tenths of a centigrade, then obtained calculated values coincide with the results of direct measurements, with a possible difference in some cases by no more than 0.1 °C. That indicates a fairly high reproducibility of the results obtained by the considered methods (Fig. 3).

The presented graph demonstrates that all experimental and calculated points have a minimum deviation from the diagonal. The correlation coefficient (R^2) was 0.9981. It should be noted that a high correlation between the two methods is observed within a wide range of freezing temperatures due to different particle size distribution, water content and salinity (Table 1). In fact, the proposed water-potentiometric method can be widely used to determine the freezing point of almost any natural soils and can be a reliable alternative to the direct experimental methods. In that case, one should bear in mind the compactness of the potentiometric device, the absence of consumables, as well as the speed of measurements and calculations and their high accuracy.

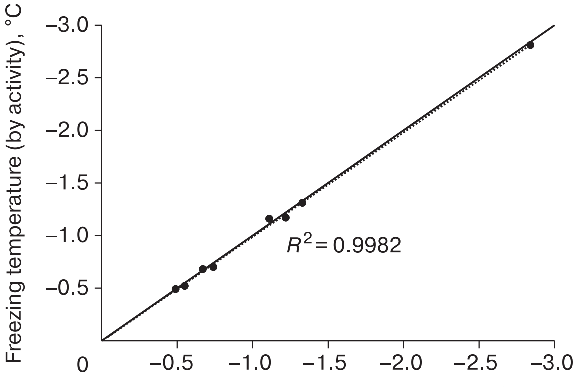

Fig. 3. Comparison of results on freezing temperatures obtained by experimental data and calculated through the activity of pore water.

At the same time, if we compare the freezing point results obtained by the experimental and water-potentiometric methods with those calculated according to [*SP 25.13330.2012, 2012*], it can be seen that the differences in the latter case reach 0.5–1.0 °C or even more, especially when comparing saline samples. Thus, the calculation method according to [*SP 25.13330.2012, 2012*] ought to be considered as a preliminary result, which should be further confirmed by experimental determinations (Fig. 4).

It should be noted that in some cases, for example, for soils contaminated with easily evaporating

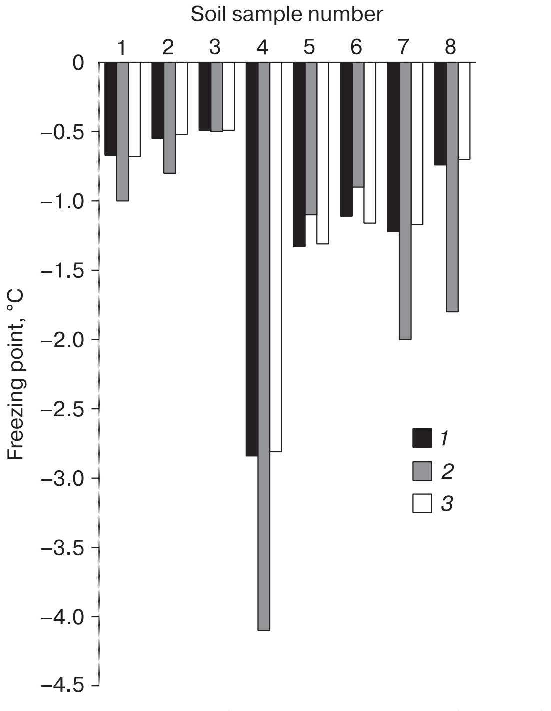

Fig. 4. Comparison of the freezing point obtained by experimental (*1*) and water-potentiometric (*2*) methods with calculations (*3*) according to [*SP 25.13330.2012, 2012*].

organic compounds, the use of the water-potentiometric method may have certain limitations. But the authors did not carry out special studies to determine the boundaries of the use of the water-potentiometric method for determining the freezing point of soils polluted with easily evaporating organic compounds.

CONCLUSIONS

The freezing point is an important characteristic of soils, which makes it necessary to experimentally determine it during various geocryological studies and engineering surveys. However, the standard technique for the experimental measurement of the freezing point of soils is distinguished by significant labor intensity, energy consumption and duration, therefore, at present, the development of various indirect (experimental-calculation) methods for its assessment is of particular relevance.

The water-potentiometric method for determining the freezing point of soils proposed in the paper is based on measuring the pore water potential with its subsequent conversion to an equivalent temperature, which is actually the freezing (or thawing) temperature. That method has a number of advantages, primarily associated with the use of a simple WP4 serial device, which carries out all the necessary measurements at room temperature in a fairly short time interval (up to 30 minutes per sample). At the same time, to obtain the values of the freezing point of the studied soils, there is no need to use any refrigeration equipment.

A comparison of the freezing point results for the same soils determinate by the direct measurement and by the water-potentiometric method has demonstrated good agreement with the maximum deviation no more than 0.05 °C, which is within the accuracy of experimental freezing point installations.

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