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TRANSPORTATION, PREPARATION AND STORAGE OF FROZEN SOIL SAMPLES FOR LABORATORY TESTING

by T. H. W. Baker

ANALYZED

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SOMMAIRE

L'augmentation des travaux de construction dans les régions froides exige une meilleure connaissance du comportement des sols gelés. Malgré la mise au point et l'amélioration des méthodes d'essai in situ, il est difficile de proposer des interprétations effectivement utiles puisque les conditions sur le terrain sont très variables et complexes. Les essais contrôlés en laboratoire sur des sols gelés aident donc à déterminer les comportements qui reflètent différentes conditions sur place et fournissent des données de calcul. Le présent article est un exposé de synthèse des méthodes employées et des difficultés que comportent le transport et le stockage des sols gelés de façon naturelle ou artificielle et les essais de laboratoire. L'auteur examine également le problème des tassements des sols causés par des variations de température et par la présence de glace. Il souligne le besoin de poursuivre l'étude des effets de ces phénomènes et des propriétés mécaniques des sols gelés.

T. H. W. Baker¹

Transportation, Preparation, and Storage of Frozen Soil Samples for Laboratory Testing

ANALYZED

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ABSTRACT: Increased construction activities in cold regions require a better understanding of the behavior of frozen soil. Although *in situ* testing procedures are being improved or developed, the highly variable and complicated conditions usually encountered in the field make it difficult to formulate interpretations that are useful in practice. Controlled laboratory tests on frozen soils are helpful, therefore, in determining behavior indicative of diverse field conditions and will assist in establishing design information. This paper is a review of the procedures used and the difficulties encountered in the transportation, preparation, and storage of naturally and artificially frozen soil samples required for testing in the laboratory. The effective control of sample disturbance by temperature variation and sublimation and the need for further investigation as to their effect on the mechanical properties of frozen soil are discussed.

KEY WORDS: soils, frozen soils, transportation, preparation, preserving, sublimation, thermal degradation, mechanical properties, laboratory tests

Procedures for transporting, preparing, and storing the naturally and artificially frozen soil samples that are required for laboratory testing are still in the formative stages. This paper reviews the current practices used for naturally frozen field samples from the time of sampling, and for artificially prepared laboratory samples from the start of the molding operation. A review of the literature is included, as well as the experiences of industry, university groups, and government departments and agencies.

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Factors Affecting Laboratory Tests on Frozen Soils

Laboratory tests are performed on naturally and artificially prepared frozen soil samples to investigate (a) strength and creep deformation behavior and (b) volume change.

Tests conducted under (a) include: stress and strain controlled unconfined compression tests, stress and strain controlled triaxial shear tests, ring shear tests, Brazilian tests, tension tests, load-controlled indentation tests, flexural strength tests, dynamic loading tests, and torsion tests.

Tests conducted under (b) include: uniaxial consolidation upon thawing under constant stress, triaxial consolidation upon thawing under constant stress, and uniaxial heave during freezing.

The strength and deformation properties of frozen soil samples are known to be affected by sublimation, evaporation, and thermal disturbance. Their effect is in the redistribution and ultimate loss of moisture from the specimen as the result of a temperature gradient or low humidity environment or both. Loss of moisture reduces the cohesion between soil particles and may reduce the strength (which is dependent on temperature). The effects of moisture redistribution in frozen soil are not known, but are thought to change the strength and creep behavior.

Thermal disturbance of a frozen sample refers not only to thawing, but also to temperature fluctuations. Soil structure may be changed completely if the sample is thawed and then refrozen. Temperature fluctuations can set up thermal gradients, causing moisture redistribution and possible change in the unfrozen moisture content. Care must be taken, therefore, to ensure that frozen soil specimens remain in their natural or prepared states and that they are protected against the detrimental effects of sublimation and thermal disturbance until testing is completed. The type of laboratory test to be conducted will determine the degree of care required in providing this protection.

Frozen Soil Samples

Naturally Frozen

Undisturbed block and core samples of frozen soil must be protected from thawing and loss of moisture, from the time they are taken from the ground, throughout the period of transportation, storage, machining, and testing. In most cases, the sample must be maintained under the same thermal and moisture conditions existing at the time of sampling. The implications and practicality of this will be discussed later.

Methods of handling frozen soil samples in the field have not changed greatly in recent years. When the samples have been examined visually and logged at the field site, most investigators wrap the frozen samples in cellophane and place them in polyethylene bags (6 mil) (Fig. 1). All air

FIG. 1—*Large block sample of frozen soil delivered from the field in a polyethylene bag (scale in tenths of a foot).*

must be evacuated from the bag, and it must be sealed to prevent sublimation. This is accomplished best by using a vacuum pump, but can be done by forcing most of the air out by hand before sealing. Humidity can be maintained by placing some snow or crushed ice in the bag. Heat sealing the plastic bag after de-airing gives the most permanent seal, but a locking nylon tie (of the type used for bundling electric cables) is adequate if it is fastened properly. The seal should be checked regularly; any air that has entered the bag should be removed, and the bag should be resealed. This packaging procedure keeps moisture loss from the sample within acceptable limits and has been used successfully by the author to

store frozen samples for periods of eight months or more. To minimize still further the moisture loss due to transfer of vapor through the plastic, Roggensack [1]² suggested that the wrapped samples should be packed in snow or ice chips. This will reduce vapor pressure gradients. Even with these precautions, some water loss occurs as vapor leaves the sample and crystallizes on the inside of the plastic wrapper. This will be discussed in the section on sublimation.

After the sample has been packaged to keep sublimation to a minimum, some facility is required at the site to prevent thermal disturbance. For example, if unfrozen samples have been obtained for frost heaving experiments, they must not be allowed to freeze so that they can be tested in truly undisturbed conditions. If ambient temperatures are below 0°C, these samples should be placed in well-insulated boxes and kept in heated tents or some other warm enclosure until they are shipped. On the other hand, when frozen soil samples are obtained for thaw consolidation, strength, and creep tests, the *in situ* temperature must be maintained. As ground temperatures vary with depth during the year, the temperature should be determined at the time of sampling. In addition, depending on the tests to be conducted, it may be necessary to determine the annual amplitude of the temperature fluctuations at the sample depth.

Maintaining the *in situ* temperature of the frozen samples has been attempted, using several methods. Portable refrigerators powered by 110-V generators have been used successfully at field sites to store frozen soil samples. These can control adequately the thermal environment and can be set to maintain the temperature measured at the field site when the sample was obtained. Insulated boxes packed with ice, dry ice, or freezer "jelly" packs have been used also to prevent thawing of frozen samples in the field. This is considerably less expensive than the use of portable refrigerators, but, of course, temperature cannot be controlled accurately. A typical insulated box used for field storage and shipping by the University of Alberta [1] is shown in Fig. 2. When using solid slabs of dry ice, thin sheets (25 mm) of insulation must be placed between the dry ice and the soil samples to minimize "burning" effects caused by contact with carbon dioxide. The dry ice is placed between the insulation and the top, bottom, and sides of the box. If freezer packs are used, they can be arranged in the same manner. A double-walled insulated box has been used, where dry ice is placed in the space between the two walls. The temperatures of samples, packed in this manner, are not constant; they will undergo a fairly rapid drop in temperature, the rate depending on how well protected they are by insulation. The dry ice evaporates slowly, and the temperature of the box (and consequently of the samples) will

²The italic numbers in brackets refer to the list of references appended to this paper.

FIG. 2—Typical insulated box used for field storage and shipping.

increase. When ice or freezer packs are used, there is no drastic cooling of the samples, but, in time, the temperature of the insulated box and samples will increase unless the ice is replaced periodically.

If the boxes are to be stored for any length of time (for example, a few weeks), it is advisable to pack snow or crushed ice around the sample bags to prevent sublimation and to fill any air spaces with crushed newspaper to provide added insulation and reduce movement of air in the box. The cover of the box should be fastened securely and made airtight by applying a thin bead of room temperature vulcanizing silicone rubber (RTV) caulking compound around the edge of the lid before the box is closed.

Although not entirely satisfactory, temporary measures to protect samples in the field can be used in some instances. During the winter, frozen soil samples, packaged in polyethylene bags, can be buried in snow drifts, or, if the ambient air temperature is below 0°C , they can be left in the open air but must be protected (shaded) from the sunlight. During the summer, frozen samples have been stored successfully for up to three days in shallow pits dug in the frozen ground or put into plastic sleeves and stored in the boreholes from which they were taken. The pit should be lined and covered with some insulating material. Moss serves quite well for this purpose. The top of boreholes, used to store samples, should be covered or plugged with insulation to prevent thawing and closing of the hole.

If adequate storage facilities are not available at the field site and the samples are to remain frozen, they must be transported immediately to a

temperature- and humidity-controlled storage area. The frozen soil samples must be shipped in refrigerated containers or insulated boxes. Power requirements for shipping a typical refrigerated unit by aircraft are outlined in Fig. 3 [2]. A competent person should accompany frozen samples

FIELD SITE

FIG. 3—Power for refrigerated units.

transported by commercial airlines from the field to the laboratory. (Refrigerated units not attended could be left unplugged on the parking apron or in a storage area, and thawing or undesirable temperature changes of the samples might occur.) Arrangements can be made to have the insulated boxes placed in airport freezers between flights. Cold storage areas are not always available, but restaurants and cafeterias at airports are usually most helpful in allowing their facilities to be used for this purpose if the containers are not too large or too many; arrangements for storage en route should be made prior to shipment. The author has found that insulated boxes packed properly can be left without auxiliary freezing up to 8 or 9 h without apparent deterioration of the samples. The samples can be left in the insulated containers and placed in cold storage upon arrival at the laboratory until required for testing.

Preparing and transporting naturally frozen undisturbed soil samples for laboratory testing is a very difficult and costly operation. In most cases, and for most purposes, insulated boxes have been used successfully to transport frozen samples to the laboratory, provided care has been taken in packaging, storing, and handling the containers.

Artificially Frozen

The physical properties that affect the mechanical behavior of soil, for example, homogeneity, grain size, moisture content, compaction, and, to

some extent, ice lens structure, should be closely controlled. This can be achieved in the laboratory by using special molds and controlled methods of freezing.

The steps followed in the preparation of laboratory frozen soil specimens are:

1. Assembly of the mold.
2. Compaction of the soil sample.
3. Saturation of the sample.
4. Freezing.
5. Disassembly of the mold and removal of the sample.

The geometry of the mold is determined by the desired shape of the test specimen. Rectangular, cylindrical, and dumbbell specimen shapes commonly are used, depending on the test to be performed. As unidirectional freezing of the sample is usually desirable, the mold is fabricated from or covered with materials having good insulating properties. Use of split molds with all joints fastened by easily removed connectors greatly facilitates specimen removal after freezing. All interfacing surfaces of the mold and all threads on metal connectors should be coated with a lubricant to ensure a tight fit for evacuating the air from the samples and to aid in extracting the sample from the mold in the cold room. Care must be taken to prevent the lubricant (especially the silicone types) from coming in contact with the sample.

Air-dried soil usually is placed into a mold in 30-mm-thick layers and compacted by a drop hammer or vibration action, following standard soil mechanics procedures (for example, Modified Proctor, American Association of State Highway Officials' Standard). Each layer should be scarified to a depth of approximately 5 mm before the next layer is placed. If the soil has a wide range of grain sizes, vibration may cause the particles to separate into layers of uniform size. For sands, the vibratory technique is the better compaction method.

The following method of evacuation and saturation has been found by Haynes [3] to give a fully saturated compact sample.

After the sample has been compacted, caps can be placed on each end of the mold. Both end caps should be fitted with hose connections, which include porous stones, to prevent the loss of soil during the air evacuation and water saturation phase. Plastic tubes are connected to both of the end caps. The tube from the top cap is connected to a vacuum pump; the tube from the bottom cap leads to an on/off valve and then to a reservoir of distilled water. A vacuum pump is also connected to the water reservoir. The on/off valve allows for separate deaeration of the mold and the reservoir. A photograph of the apparatus is shown in Fig. 4.

A vacuum is applied to the top of the mold with the on/off valve closed. At the same time the distilled water reservoir is placed under a

FIG. 4—*Apparatus for molding and saturating artificial samples.*

vacuum to remove as much air as possible. After about 2 h, the vacuum pressure in the reservoir is decreased, and the valve is opened. A differential pressure between the mold vacuum and the water tank vacuum is maintained so that the water travels up through the sample and saturates it at a slow steady rate. Fast saturation must be avoided to prevent: (a) piping of water through channels along the outside of the sample; (b) a "quick" condition disturbing the dense packing obtained in compaction; or (c) incomplete evacuation of air. When the samples appear to be saturated (that is, water can be seen in the top tube), the vacuum pumps are turned off, and the sample is allowed to saturate under a 0.5-m head of water for about 1 h.

Goughnour and Andersland [4] and Hooke et al [5] prepared sand-ice specimens by mixing natural snow uniformly with a selected proportion of precooled sand. This mixture was compacted in the mold, and water, precooled to 1°C, was admitted under pressure. Little snow melted during this process, and the sand particles remained dispersed during freezing. Using this procedure, frozen sand specimens were obtained with large ice contents.

Vyalov [6] reported that frozen soil without ice segregation can be obtained by freezing samples rapidly at temperature below -30°C . To obtain

specimens having a layered or reticulate structure (that is, horizontal or randomly oriented in layers, respectively), the soil samples should be frozen at temperatures of about -6°C or above. A layered, ice-lensed structure is obtained by cooling one end of the sample and providing free access to a constant supply of water at the other end.

Tsytoich and Sumgin [7] investigated the influence of the freezing method on the unconfined compressive strength. The results indicated that a freezing temperature between -12 and -17°C gave a relatively lower strength if the freezing time was less than three days for sand and four days for clay. They also found that the strength properties were not affected significantly if the freezing occurred from all sides or only from the top down.

To achieve a uniform moisture content throughout a sample of Fairbanks silt, Haynes [3] froze specimens unidirectionally in a cold chamber at a temperature of -37°C . The bottom mold connection was attached to a water supply; the top of the mold was exposed to freezing temperatures. Loose insulation (granulated cork) was placed under and around the mold to reduce radial heat loss and to allow the sample to freeze from the top down. Vermiculite can be used for this purpose instead of granulated cork (Fig. 5). A constant supply of water was maintained at the bottom of the mold to replenish the water that migrated upward from the unfrozen zone

FIG. 5—Vermiculite placed around the mold to allow the sample to freeze unidirectionally.

to the freezing front. Thermistors, placed at locations along the center line of the specimen, recorded the freezing rate.

Cold rooms, refrigerated cabinets, and constant temperature baths have been used by many investigators to control the freezing rate of remolded soils. Many cooling systems have slow response times, so that rapid adjustment of the rate of heat extraction from the sample is usually very difficult.

More accurate control of the freezing process can be obtained, using a thermoelectric cooling plate which operates on the Peltier effect [8]. The design of a cooling plate (Peltier module) is described in detail elsewhere [9,10]. The temperature of the plate can be maintained with an accuracy of at least $\pm 0.02^\circ\text{C}$. As heat removal from the sample is uniaxial, this method is well suited for unidirectional freezing of a soil specimen. Samples of various sizes, with variations in moisture content of ± 1.5 percent by dry weight, have been frozen successfully under many different test conditions, using a thermoelectric plate at the Division of Building Research (DBR), National Research Council of Canada (NRC) [9].

Rapid sample freezing minimizes redistribution of soil moisture. Freezing too rapidly, however, can cause entrapment of air bubbles in the specimen and tensile failures due to thermal shock. Typical freezing rates used by Sayles and Haines [11,12] on small specimens (69 mm diameter by 140 mm high) were:

Ottawa sand (20 to 30 mesh)	38 mm/day
Manchester fine sand	40 mm/day
Hanover silt	80 mm/day
Suffield clay	170 mm/day

Methods of freezing large remolded soil specimens have been reported by Warder and Andersland [13] (125 mm diameter by 300 mm high) and Laba [14] (100 mm diameter by 241 mm high), but no reference was made to their homogeneity. It is not known whether it is possible to produce large uniform samples using existing freezing techniques. Samples frozen in the laboratory are molded normally in the test apparatus and frozen just prior to testing, thus eliminating the difficulties associated with the storage of frozen specimens.

Machining and Preparation of Specimens for Testing

The machining and preparation procedures used for frozen soils depend upon the size and shape of the specimen required, the type of soil, and the particular test being performed. Similar procedures are followed for cutting and machining both naturally frozen and artificially frozen samples.

Frozen soil samples should be handled with gloves and all tools and equipment kept in the cold room to avoid sample damage by localized thawing. A temperature of $-5 \pm 1^\circ\text{C}$ is the most suitable ambient temperature for machining with respect to material workability and personal comfort. At warmer temperatures, surface thawing is a problem, and cutting tools must be cleaned frequently, for they become coated and clogged with frozen soil, reducing their cutting efficiency. Working at colder temperatures is uncomfortable and slow. The soil is also difficult to work with because of increased hardness; cracks can be formed easily in it, due to increased brittleness.

Any cracking induced in the specimen during preparation will reduce the load-carrying capability and influence the test results. Thermal disturbance, due to frictional melting by a cutting tool, may be insignificant when preparing specimens for thaw consolidation tests, but it may be very significant for strength tests. Extreme care must be taken when machining frozen soil samples for testing to keep disturbance to an absolute minimum.

Various cutting and grinding techniques have been used to machine specimens to the shape required for the test to be performed.

Rough Cutting Methods

A number of methods for cutting frozen soil, including using a hand, band, or chain saw, forcing a cutting edge into the frozen soil, and using a hot wire have been investigated at NRC. Others have found rotary diamond saws satisfactory. The advantages and disadvantages of these methods are summarized in Table 1.

Finishing Methods

After being cut roughly to the required dimension, rectangular specimens are finished usually by one, or a combination, of the following methods, listed in increasing order of precision:

1. Hand shaving with a sharp, straight cutting edge (for example, a draw knife).
2. A coarse wood rasp or file.
3. Grinding with several grades of emery paper or grinding stone.
4. Milling machine equipped with heavy-duty cutters.
5. Drill press (heavy-duty) equipped with an end milling tool.

These methods have been used on a wide range of frozen soils, and the particular application of each is dependent upon the required specimen tolerances. The allowable specimen tolerance is dependent on the type of test to be performed and is discussed next.

Cylindrical specimens are machined either on a metal working lathe or cut carefully with a coring tube in the laboratory.

A lathe is being used extensively at the Division of Building Research to obtain well finished cylindrical specimens of most types of frozen soils up to 150 mm in diameter. Soils containing pebbles are extremely difficult to machine on a lathe. If pebbles are not plucked out by the lathe, the specimen becomes jammed on the cutting tool and breaks between the tool post and the chuck. The various steps in the preparation of specimens on a lathe are outlined pictorially in Figs. 8-10.

Samples are cut initially to eight-sided cylinders on the bandsaw. A V-block is used to hold the specimen so that the ends can be squared on the bandsaw. The dimensions of the eight-sided cylinder are chosen so that the specimen can be turned to a right cylinder with both ends faced. A finished specimen 75 mm in diameter by 150 mm long is turned from an eight-sided cylinder 85 mm across the end and 200 mm long.

The eight-sided cylinder is placed on the lathe, with one end supported in a four-jaw chuck and the other end supported by the tailstock. The tailstock is separated from the specimen by the round wooden block which has sandpaper glued to one side and a cone seat (for the tailstock) on the other. Pressure applied to the specimen by the tailstock is distributed evenly over the face of the specimen by the wooden block.

Best results are obtained when the frozen soil specimen is turned at 690 rpm and the carriage feed set at 30 mm/36 revolutions. The maximum depth of cut should be limited to 0.38 mm. A tungsten carbide cutting tool, with a minimum back clearance of 45 deg, gives the best results. For clean cuts, the tool has to be sharpened often, as the abrasive action of the soil dulls the edge quickly.

A tool post grinder can be used for dressing coarse-grained soils. Grinding efficiency for fine-grained soils is reduced by buildup of material on the grindstone. A special holder that screws onto the spindle of the lathe and allows the ends of the specimen to be squared and faced with the cutting tool is shown in Fig. 10.

Cylindrical samples can be cored from block samples in the laboratory, using a diamond set core barrel and a large industrial drill press. The block is held to the drill press frame by clamps. This procedure has been used in Alaska to produce sand and gravel specimens with less than 0.5 percent variation in diameter and for silt and clay samples with less than 0.4 percent variation [15]. The drill press was fitted with a gas swivel through which pressurized liquid nitrogen vapor was passed to blow the cuttings away. The configuration of the diamond matrix and vents on the core bit was varied in order to core each type of soil most effectively. To obtain specimens of consistent diameter, the internal diameter of the core bit must be checked often and the diamond matrix replaced if the allowable tolerances are exceeded.

TABLE 1—*Rough cutting methods.*

Method	Advantages	Disadvantages
Sharp edge Axe (for example)		poor, as the ice in front of the cutting edge chipped, leaving voids in the sample
Hot wire (piano) 23 kg weights 2 to 4 V	wire passed quickly through 10-cm specimens of frozen clay	a cheese cutter frame was rigged with a hot wire, but not enough pressure could be applied by hand to obtain efficient cutting; it is suggested that this method not be used to cut laboratory specimens of frozen soil due to its thawing action
Hand saws Miter saw Hack saw Swede saw	a swede saw was used for rough cutting large blocks of frozen clay (Fig. 6); use of a miter box improves geometric control	inadequate due to slow progress and poor geometric control; for effective cutting the blade had to be continually cleaned to remove partially thawed clay and to prevent binding
Chain saw Electric powered 254 mm arm Gas powered 381 mm arm	excellent for rough cutting large blocks of frozen peat and silt which have high ice contents	ineffective in cutting frozen clay with the teeth and chain becoming clogged with clay and the blade binding in the cut
Band saw 1/2 hp, 1725 rpm 5 mm blade, 2 teeth/cm 10 mm blade, 1 tooth/cm	very satisfactory for cutting fine-grain specimens; quite adequate for coarse sands (Fig. 7) fast cut straight cut	largest sample that could be cut was 159 mm thick, but with height attachment up to 311 mm blade wanders when cutting specimens 60 to 200 mm thick cuts slowly and dulls very quickly due to the abrasive action of the frozen soil; tooth life very short with no significant

Diamond saw	in conjunction with table and guide, it is the most desirable method for cutting frozen soils; very clean straight cut even in coarse-grain pebbly soils	difference between ferrous, non-ferrous and wood cutting blades depth of cut restricted by the radius of the blade; clayey soils greatly reduce cutting efficiency of the saw blade as the blade must be cleaned regularly
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FIG. 6—Use of a swede saw for rough cutting large block samples.

At the University of Alberta, a core barrel attached to a milling machine was used to core cylindrical specimens from block samples [1]. An 80 mm length of steel tubing (67 mm inside diameter) was fabricated with a cutting edge on one end. The cutting edge was formed by rolling the lip of the tube inward and sharpening it to an inside diameter of 64 mm. The core barrel, clamped in the drive chuck of a milling machine, was rotated at 300 rpm. Only short specimens were prepared for testing. Long specimens break during coring, particularly if the frozen soil has a high ice content.

Dumbbell-shaped specimens for tensile testing are prepared in molds or by machining samples on the lathe, using these procedures.

Greater care is necessary in machining naturally frozen samples, as they

FIG. 7—Use of band saw to cut smaller samples.

may contain large ice inclusions (lenses, etc.) that are very fragile and easily chipped. Unexpected occurrences of pebbles can ruin many hours of machining if care is not taken when they are detected. Many samples, obtained at high cost in the field, have not been tested because they were too difficult to machine.

These combined cutting and coring processes are used to prepare frozen soil specimens for all forms of load testing. Dimensional tolerances for uniaxial thaw-consolidation test specimens are particularly critical, as the sample must fit tightly into the consolidometer ring. The nature of the other tests is such that the dimensions of specimens are less critical.

It is important that the ends of the specimens are parallel and plane, so that intimate contact occurs with the loading platens. Seating errors have been observed on many loading curves [4]. Eccentric loading can give lower strength values [3], but these are often ignored.

Although the effects of thermal disturbance induced by machining procedures have not been investigated, every effort should be made to eliminate or reduce them, especially on clay samples. Surface thawing has been noticed on several occasions when machining specimens of frozen clay.

FIG. 8—*Eight-sided cylinder cut on band saw.*

Storage and Protection During Laboratory Testing

Careful control of temperature and humidity in storage areas is necessary to protect the frozen sample from sublimation and thermal disturbance, from the time it has been molded or machined or both until testing has been completed. Protection methods are similar to those used when transporting frozen samples from the field. Samples should be wrapped in an impermeable material and stored in a refrigerated room or freezer. Khomichevskaya, one of the earlier Russian investigators, wrapped naturally frozen samples in insulation and stored them in underground rooms for up to seven days with good results [16].

Sublimation of samples can be minimized by controlling the humidity and reducing air flow around the specimen. Most small capacity freezers do not have air circulation fans in the freezing compartments, and air

FIG. 9—Specimen turned on lathe to required diameter.

flow is limited to that caused by convection currents. Large walk-in freezers rely on fans to circulate the air and control the temperature effectively throughout the storage chamber. Frozen samples can be protected from these air currents by wrapping them in cellophane and placing them in plastic bags. Snow or ice shavings can be placed in the bags to raise the humidity around the sample. The bags should then be evacuated to remove as much air as possible and heat sealed. These procedures are shown in Figs. 11 and 12.

In studies of ways to store and preserve frozen samples, Livingston [17] found that samples lost considerable weight, due to surface evaporation, when the storage temperature ranged from 0 to -6°C . To study the effect of the storage temperature on surface evaporation and sublimation, small specimens of frozen Keweenaw silt (approximately 25 mm cubes with polished surfaces) were prepared and stored at various temperatures. The results indicated that storage at temperatures between -8 and -12°C reduced the rate of surface evaporation to an acceptable value. Various methods were investigated to reduce sublimation further, as failure to control this moisture loss affects test results. Synthetic resins, silicone oil, varnishes, natural oils, and other commercially available products were sprayed or painted on the surface of the specimens. Vegetable and min-

FIG. 10—Facing specimen ends on the lathe using special holder.

eral waxes could not be used as they must be heated. Although many of the surface coatings reduced the rate of surface evaporation, none of the materials investigated provided satisfactory protection for more than one week.

Frozen soil specimens at the University of Alberta have been coated with a thin layer of ice by applying a fine spray of supercooled distilled water [7]. This film of ice prevents sublimation of the sample and is removed easily prior to testing.

Yershov et al [18] published information on the sublimation of frozen soils. As shown in Fig. 13, different soil types have different sublimation rates. Finer grained clay soils have a higher evaporation rate than do coarser sands. The rate of sublimation is affected greatly by the ambient temperature (Fig. 14). The parameters studied were temperature, relative humidity, and air velocity, but no details were given on how the experiments were undertaken nor on the apparatus used.

A similar study was undertaken by the author to determine the rate of sublimation on three soil types: Ottawa sand (20 to 30 mesh), Niagara silt, and Leda clay. Four specimens of each soil type were prepared by adding 50 g of water to equal volumes of each soil and placing the mixture in

FIG. 11—*Evacuating plastic bag to prevent sublimation.*

plastic boxes 1.9×10^{-2} m deep, having a surface area of 3.31×10^{-3} m². Two samples of each soil type had their surfaces covered with cellophane; the others were left uncovered. All the samples were weighed and placed in the cold room to freeze at a temperature of -4.4°C and a relative humidity of 37 percent. The specimens were weighed after 8 h and then once a day for 23 days. Results are presented in Fig. 15.

The sublimation rates of the frozen soils investigated by the author are within the same range as those reported by Yershov et al [18]. Similarly, it was found that the finer clay soils have higher sublimation rates than the coarser sands. This is because the larger particle surface area in fine-grained soils allows for film flow, which brings the water to the surface of the sample. Samples that were covered with cellophane had significantly lower sublimation rates than those that were left uncovered.

Many investigators [5,7,17,19-21] have found that the compressive strength of frozen soil is greatly dependent on its overall moisture content. Compressive strength increased with increasing moisture content. It

FIG. 12—Heat sealing the plastic bag.

reached a maximum when the soil was saturated completely and then decreased to the strength of ice. Any loss of moisture from a specimen, therefore, will cause a change in its compressive strength.

The compressive behavior of frozen soil is also affected greatly by temperature and by temperature fluctuations. It is imperative, therefore, that specimens be stored and tested in a freezing chamber that has only a small temperature fluctuation to minimize thermal disturbance. The effect of fluctuations in temperature can be reduced by enclosing the specimen in an insulating jacket during storage and testing. Vyalov [6] suggested the following permissible temperature variations when storing and testing frozen soils within different ranges:

Temperature, °C	up to -2	-2 to -5	-5 to -10	below -10
Permissible deviation	±0.1	±0.2	±0.5	±1.0

Guarded hot plate experiments carried out by Hoekstra [22] using Fairbanks silt (see Fig. 16) show that any thermal gradient in the sample, even at temperatures below freezing, will cause moisture redistribution in fro-

FIG. 14—Sublimation and temperature (Yershov et al [18])

FIG. 13—Sublimation rates of frozen soils (Yershov et al [18])

FIG. 15—Sublimation in NRC/DBR cold room.

FIG. 16—Effect of temperature gradient on moisture redistribution in frozen Fairbanks silt (Hoekstra [22]).

zen soil. This moisture movement will increase the sublimation/evaporation rate, as well as change the fabric of the specimen.

Many investigators [19,23] have found that the unfrozen moisture content will influence greatly the compressive strength of frozen soils. Williams [24], using calorimetric methods, found that there was a hysteresis effect in the temperature dependence of the unfrozen water content. The unfrozen water content of a frozen soil, at a given temperature, is dependent on whether the temperature is reached by cooling or warming. If temperature fluctuations occur, even though the specimen remains below 0°C, the unfrozen moisture content will change. This is of great concern when testing frozen soils at temperatures above -5°C.

The cold rooms at DBR/NRC provide good examples of well controlled environments. The temperature of the cold rooms can be controlled accurately by a reheat system to within $\pm 0.1^\circ\text{C}$ at any location in the room. Each refrigeration unit is operated by a two-stage compressor with a capacity of 3.5 tons of refrigeration circulating Freon 22. The cooling capacity is adjusted automatically to be in excess of that required, and the temperature is controlled by electric reheating. The reheat unit has a capacity of 12 kW and is operated by a solid state controller. Two evaporation units are located in each of the cold rooms so that one unit can be defrosted without shutting the cold rooms down. A separate auxiliary compressor and control system is available in case one of the main systems becomes inoperative. In addition, a freezer of 1 m³ capacity is available also in the laboratory as a backup system. Samples can be transferred to the freezer in case of emergency shutdown. The need for an alarm system to provide warning of equipment failure and for several backup systems is appreciated only after samples are lost due to thawing because of unexpected equipment breakdowns.

Conclusions

The important factors to be considered in transporting, preparing, and storing frozen soils for laboratory testing are the control of the effects of sublimation/evaporation and minimization of thermal disturbance. Their effect on the ice content, fabric, and unfrozen moisture content of the specimen, and, therefore, on the strength and deformation behavior of the material, is of great significance. Further evaluation of existing procedures and development of new or improved techniques are required before standard methods can be proposed for the laboratory testing of frozen soils.

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