

We also determined the storage conditions of the dried material. For this the material was removed from the drier by a vacuum loader and charged into a heated bunker (60-80°C) whose size was the same as that of the drier. To prevent agglutination of the material in the bunker, it was stirred by a stirrer having a speed on the order of 1 rpm. To simulate conditions of real operation, material was released (by periodically opening a door in the lower part of the bunker).

A number of experiments showed that some moistening of the material was observed (Fig. 4). Therefore, if the quality is to be guaranteed, the material has either to be dried more than necessary, which impairs the total productivity of the equipment, or else the bunker has to be evacuated.

NOTATION

W, moisture content; α , C, drying coefficients; τ , time; t, temperature of the layer; P, pressure in the chamber; n, rotary speed of the rotor. Indices: 0, initial.

LITERATURE CITED

1. N. V. Antonishin, V. S. Nikitin, O. G. Martynov, and G. F. Puchkov, "Investigation of heat and mass exchange in a vibrorotating layer," *Vestsi Akad. Nauk BSSR, Ser. Fiz.-Energ. Navuk*, No. 2, 63-67 (1977).
2. V. M. Kataev (ed.), in: *Plastics Handbook [in Russian]*, 2nd ed., Vol. 1, Khimiya, Moscow (1975), p. 106.
3. *Tekhnicheskie Usloviya (TU) 6-05-1587-74*.
4. *Tekhnicheskie Usloviya (TU) 6-05-1668-74*.
5. I. F. Pikus, L. S. Gimpeleva, and L. A. Koshepavo, "The basic regularities of the vacuum drying of electrically insulating cellulose materials," *Inzh.-Fiz. Zh.*, 22, No. 3, 420-427 (1972).

ELECTROMETRIC METHODS OF INVESTIGATING CRYOGENIC PHASE TRANSFORMATIONS OF LIQUID MOISTURE IN BUILDING MATERIALS

Yu. D. Yasin

UDC 536.425

The article explains the basic concepts of the electrometric methods of investigating cryogenic phase transformations of liquid moisture in building materials.

In the experimental investigation of cryogenic phase transformations of liquid moisture in materials with capillary pores and in disperse materials, several methods are currently in use: 1) the calorimetric method, which is based on measuring the heat effect due to the temperature of the phase transition [1]; 2) the dilatometric method, based on measuring the total volume of the liquid and solid phases of the pore moisture in its phase transformations [2]; 3) the method connected with the measurement of the thermal characteristics [3], etc.

Without examining in detail the advantages and shortcomings of the different methods, we want to point out only one common substantial shortcoming of all of them — experimental investigations are carried out with small specimens. This makes it impossible to investigate phase transformations of the moisture in local volumes of large fragments imitating building structures, or directly in actual building structures. In addition to that, and equally important, the investigation of the kinetics of phase transitions by the above methods encounters a number of methodological difficulties.

The electrometric investigation methods [4] are free of these shortcomings, and in addition to that they have a number of obvious advantages owing to the physical principles on which they are based. With these methods, the measured characteristic is any electrical pa-

Research Institute of Constructional Physics, Gosstroii SSSR, Moscow. Translated from *Inzhenerno-Fizicheskii Zhurnal*, Vol. 42, No. 3, pp. 437-442, March, 1982. Original article submitted December 18, 1980.

parameter, e.g., electrical resistance, that is functionally correlated with other parameters determining the state of the system (the building material).

The physical concept of the electrometric methods of investigating phase transformations (liquid moisture-ice) is based on the principle of continuity, which is formulated as follows: "with continuous change of the parameters determining the state of the system, the properties of separate phases change continuously, and the properties of the system, taken as a whole, also change continuously but on condition that no new phases originate and no existing phases disappear" [5].

Thus the electrometric method of investigating cryogenic phase transformations of liquid moisture reduces to finding a functional dependence of the measured electrical parameter on other parameters determining the state of the investigated system in the absence of phase transformations, bearing in mind that in case of phase transformations, they can be quantitatively interpreted.

From the thermodynamic point of view, moist materials with capillary pores and disperse materials belong to the complex heterogeneous multiphase systems. Water-soluble components or the colloidal component of the skeleton of the material endow the pore moisture with the properties of ionic or colloidal solutions, and the developed surface of the material itself as well as of the product of phase transformations — ice — has a substantial effect on these properties in the adjacent layers of the solution. Even if we leave other aspects out of account, we can imagine the complexity and variety of the phenomena accompanying and influencing the phase transformations. Moreover, not all the parameters determining the state of the system can be directly measured or controlled. Therefore, the sought dependence correlating the electrical parameter with the parameter determining the state of the system is defined by a mathematical model in which it is taken that the influence of the uncontrolled parameters is randomized. Such a model representation makes it possible to describe the behavior of a complex system without detailing it.

For convenient statistical evaluation and subsequent interpretation, the mathematical model describing the sought dependence is represented in the form

$$y = b_0 + b_1x_1 + \dots + b_kx_k + e. \quad (1)$$

The model (1) is considered linear in the sense that the parameters b_0, b_1, \dots, b_k contained in it are linear with respect to y , whereas the response of the model y and each independent variable of the model x_i may be arbitrary functions of the measured as well as of a controlled parameter determining the state of the investigated system (moist building material).

The magnitude of the error e depends on the degree of adequateness of the adopted model, on the accuracy of measurement of the controlled parameters, and also on the interference due to the uncontrolled parameters. Determination of the point and interval values of the coefficients b_0, b_1, \dots, b_k , statistical evaluation, and verification of the adequateness of the model (1) are effected according to data containing $m > k + 1$ observed values of y, x_1, \dots, x_k by known statistical methods [6].

With the conductometric method of investigation, where the measured electrical parameter is the electrical resistance, the correlation between the variables of the model y, x_1, \dots, x_k and the measured and controlled parameters of the investigated system (moist building material) can be easily found from the a priori data [7].

In this case the sought dependence, in accordance with the adopted model, has the form

$$y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + e, \quad (2)$$

where

$$y = \lg R; \quad x_1 = t; \quad x_2 = \lg u; \quad x_3 = t \lg u.$$

The dependence (2) is intended to determine the total moisture content, and in cryogenic phase transformations the amount of nonfrozen water, in individual specimens of building materials, or in local zones of measurement of structures. In other electrometric investigation methods, especially in the dielectric method, the sequence in the construction and statistical evaluation of the model is analogous.

In the indirect method of measurement, when the parameter to be determined is described by a functional dependence of the form

$$y = f(x_1, \dots, x_k), \quad (3)$$

the total error of the function is determined from the expression [8]

$$E_y = \frac{\partial y}{\partial x_1} E_{x_1} + \dots + \frac{\partial y}{\partial x_k} E_{x_k}, \quad (4)$$

where

$$E_y = \Theta_y + \Delta_y, \quad E_{x_i} = \Theta_{x_i} + \Delta_{x_i} \quad (i = 1, \dots, k).$$

It may be assumed that the measured and the controlled parameters of the investigated system do not contain considerable systematic errors because it is always possible, by comparison with standards, to evaluate the existing systematic errors, be they constant or variable, and by introducing the corresponding corrections to eliminate their effect. In this sense expression (4) is transformed into

$$\Delta_y = \frac{\partial y}{\partial x_1} \Delta_{x_1} + \dots + \frac{\partial y}{\partial x_k} \Delta_{x_k}. \quad (5)$$

If we know the power of resolution of the measuring devices, we can, according to (5), evaluate the contribution of the measured parameters of the investigated system to the residual error of the model (2), and then the remaining part of this error characterizes the distortions of the uncontrolled parameters when the adopted model is adequate.

Let us examine the case when the effect of the interference due to the uncontrolled parameters can be substantially reduced. Because of the condition of randomization, the uncontrolled parameters in the electrometric investigation method upon construction of the sought mathematical model change randomly in the entire set of local zones of measurement, but for each actual zone these changes are of a systematic nature, and therefore the true error of determining the moisture content u in the j -th zone can be represented in the form

$$E_{u_j} = \Delta_u + (\bar{\Theta}_u + \check{\Theta}_u)_j \quad (j = 1, \dots, m).$$

The presence or absence of a systematic error can be judged from the discrepancies between the values of u predicted by the model (2) and the values of u' obtained by the comparative (standard) method:

$$|u - u'| > |\Delta_u| + |\Delta_{u'}|, \quad (6)$$

$$|u - u'| \leq |\Delta_u| + |\Delta_{u'}|, \quad (7)$$

where the inequality (6) presupposes the presence, and the equality-inequality (7) presupposes the absence of a systematic error. If there is a systematic error, it is expedient to represent inequality (6) in the form of an equality

$$(\check{\Theta}_u + \bar{\Theta}_u)_j = |u - u'| - |\Delta_u| - |\Delta_{u'}|. \quad (8)$$

After the systematic error has been determined from expression (8), it is introduced in the form of a correction into model (2), and consequently the results of determining u in the j -th local zone will be free of the influence of the uncontrolled parameters.

In the investigation of cryogenic phase transformations of the liquid phase in individual specimens of building materials, it is expedient to use as comparative method the gravimetric method; then the correction for each j -th specimen is determined from the discrepancies between the values of u predicted by model (2) according to the magnitude of the measured electrical parameter and of the controlled temperature in the region of above-zero temperatures, on the one hand, and the value of u' obtained by the gravimetric method, on the other hand. The results of calculating the nonfrozen water and ice at subzero temperatures, taking the found correction into account, will also be free of this kind of systematic error.

Another cause of the appearance of an undetermined systematic error may be the formation of a solid phase, viz., ice. To discover this kind of error, it is reasonable to use as comparative methods two independent electrometric methods — the conductometric and the dielectrometric methods — since in this situation the gravimetric method cannot be used because it provides information only on the total moisture content. Here it is assumed that although both compared electrometric methods may have systematic errors caused by the formation of a new phase, these errors will be of different origin, and it is therefore improbable that they will cancel out each other. Thus, if condition (7) is fulfilled, it may be assumed that the formation of a new phase does not introduce additional systematic errors. Previous investigations of this kind showed that there are no substantial systematic errors in a broad range of temperatures and moisture contents for clay and silicate bricks [9].

At the second stage of the investigation, the obtained experimental material on the determination of the amount of nonfrozen water u_2 is reduced to the equation of state

$$u_2 = f(u, t). \quad (9)$$

As before, the dependence (9) is expressed in the form of the mathematical model (1) in which we put by preliminary analysis of the experimental body:

$$y = u_2, \quad x_1 = u, \quad x_2 = 1/t, \quad x_3 = u/t.$$

The obtained dependence is represented in the following form, which is convenient for subsequent utilization and interpretation:

$$u_2 = b_0 + b_1 u + \frac{b_2 + b_3 u}{t} \quad \text{for } t \leq t_{i.f}. \quad (10)$$

The temperature of incipient freezing $t_{i.f}$ is determined from the expression

$$t_{i.f} = \frac{b_2 + b_3 u}{u(1 - b_1) - b_0}, \quad (11)$$

which was obtained from (10) by the transition

$$t = t_{i.f} \quad \text{for } u_2 = u.$$

Table 1 presents the coefficients of expressions (10) and (11) and the sampling root-mean-square deviations S_{u_2} for some building materials, where u and u_2 have the dimension kg/kg, and t is in °C. Dependence (10) makes it possible not only to reduce the obtained information on phase transformations to a compact form, but also to obtain additional information, especially in complex investigations of phase transformations directly in building structures. Let us examine the sequence of this kind of investigation. Small sensors for the electrical

TABLE 1. Empirical Coefficients Contained in Expressions (10) and (11) and Sampling Root-Mean-Square Deviations

Building material	γ , kg/ m ³	Coefficients, formula (10)				$S_{u_2} \cdot 10^2$
		$b_0 \cdot 10^2$	b_1	$b_2 \cdot 10^2$	b_3	
Cement block	2300	4,222	0,474	1,939	-0,645	0,10
Cement-sand mortar 1:1	2120	3,239	0,411	9,052	-1,637	0,08
Cement-sand mortar 1:2	1935	0,549	0,786	0,573	-0,605	0,07
Cement-sand mortar 1:4	1725	2,197	0,260	1,915	-1,193	0,08
Foam concrete	850	2,353	0,242	-7,670	-1,135	0,18
Keramzit concrete	1430	3,833	0,219	-4,269	-0,304	0,08
	1000	4,448	0,129	0,934	-1,758	0,08
Vermicular concrete	715	6,783	0,016	4,241	-0,767	0,39
Porous mortar	1320	3,210	0,138	-2,732	-0,930	0,23
Brick:						
silicate	1750	0,161	0,258	-1,751	-1,197	0,12
clay	1550	0,151	0,023	-3,717	-0,321	0,08
Foamglass	170	0,095	0,014	-24,208	-0,146	0,30

parameter and the temperature are placed into the investigated building structure, measurements are carried out, and for each local zone of action of the sensors the amount of non-frozen water is determined according to the previously specified dependence (2). The total amount of moisture in the local zone of the sensor is determined from expression (10) after the found amount of nonfrozen water and the corresponding measured temperature are substituted. After the total moisture content has been determined, we also determined the amount of the solid phase, viz., ice.

NOTATION

y , x_1 , ..., x_k , dependent and independent variables of model (1); b_0 , b_1 , ..., b_k , empirical coefficients of model (1); e , residual error of model (1); R , electrical resistance; t , temperature; u , total moisture content; u_2 , amount of nonfrozen water; γ , volumetric weight; E , θ , Δ , total, systematic, and random error, respectively; $\bar{\theta}$, $\tilde{\theta}$, constant and variable components, respectively, of the systematic error; S , rms deviation.

LITERATURE CITED

1. Z. A. Nersesova, "Calorimetric method of determining the icing of soils," in: Materials on Laboratory Investigations of Frozen Soils [in Russian], No. 1, Izd. Akad. Nauk SSSR, Moscow (1953), pp. 77-85.
2. A. S. Berkman and I. G. Mel'nikova, Structure and Frost Resistance of Walling Materials [in Russian], Gosstroizdat, Leningrad-Moscow (1962).
3. A. M. Rozenfel'd, "Determination of the ice content of soil with the aid of thermal characteristics," Dokl. VASKhNIL, No. 3, 29-34 (1951).
4. M. I. Frimshtein and Yu. D. Yasin, Inventor's Certificate No. 197233, "Method of determining the amount of nonfrozen water at below-zero temperatures of structures," Byull. Izobret., No. 12 (1967).
5. Ya. I. Gerasimov et al., Course of Physical Chemistry [in Russian], Vol. 1, Khimiya, Moscow (1964).
6. D. M. Himmelblau, Process Analysis by Statistical Methods, Wiley (1970).
7. M. A. Berliner, Measurements of Moisture [in Russian], Énergiya, Moscow (1973).
8. Yu. V. Kemnits, Theory of Errors of Measurement [in Russian], Nedra, Moscow (1967).
9. M. I. Frimshtein, Yu. D. Yasin, and A. K. Khurtsilava, "Investigation of the physico-chemical properties of moist building materials," in: Investigations of Building Materials by Methods of Engineering Physics, UralNIISTromproekt, Chelyabinsk (1972), pp. 25-30.