## INVESTIGATION OF THERMAL NOISE IN A DYNAMIC THERMOVACUUM METHOD FOR MEASURING MOISTURE CONTENT OF LOOSE MATERIALS

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The effect of thermal noise due to adiabatic expansion of a vapor-air mixture in the chamber of a primary transducer is considered and methods to decrease this destabilizing factor are proposed. Based on an analysis of thermal processes in the sample-vessel-medium system a mathematical heat and mass-transfer model for a dynamic thermovacuum method for measuring the moisture content is proposed.

The high metrological potential of the thermovacuum method (TV method) to measure the moisture content of loose materials [1-3] is counterbalanced to a great extent by a number of fundamental drawbacks, among which are the long duration of a measurement cycle and unsuitability to control materials with a high moisture content in which the moisture freezes during measurement.

The dynamic thermovacuum method that was proposed earlier [4, 5] permits determination of the moisture content of a material from the initial segment of a thermogram, which makes it possible to decrease the time of the measurement cycle and to considerably extend the measurement range.

This in turn imposes certain limits on the magnitude of thermal noise at the beginning of evacuation, principal of which is the thermal noise due to adiabatic expansion of the vapor-air mixture in the chamber, which induces parasitic cooling of the sample. This thermal action on the sample is a component of the additive error (curve 2 in Fig. 1).

In general form, the process is polytropic with a variable polytropic exponent *n*. In evacuation, at the initial instant, when the heat loss in the mixture can be disregarded, the process is adiabatic, i.e.,  $n = \kappa$ , where  $\kappa$  is the adiabatic exponent. Were the sample dry (were there no cooling of the sample through moisture evaporation), the temperatures of the element of the primary transducer and the vapor-air mixture would level off and the thermodynamic process would gradually approach an isothermal one (n = 1).

When analyzing the thermal effect of adiabatic expansion on the measurement result we can restrict ourselves to the "lower" bound for the mixture temperature, considering the process as being adiabatic over the entire time interval, as a result of which the thermal noise will be maximum. In this case, we obtain the most "unfavorable" regime as far as the accuracy of measurement is concerned.

The adiabatic equation can be written as:

$$VT^{\frac{1}{\kappa-1}} = \text{const}.$$
 (1)

On differentiation with respect to time we obtain

$$\frac{dV}{dt}T^{\frac{1}{\kappa-1}} + \frac{1}{\kappa-1}VT^{\frac{2-\kappa}{\kappa-1}}\frac{dT}{dt} = 0,$$

whence

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Fig. 1. Temperature of sample versus its evacuation time (1) legitimate signal, 2) temperature noise).

Fig. 2. Vessel in a heat-insulation cell.

$$\frac{dT}{dt} = -\frac{(\kappa - 1) ST(t)}{V(t)},$$
(2)

where S = dV/dt is the vacuum-pump capacity.

Let us calculate the strength of negative heat sources in the vapor-air mixture q. Since we consider the heat exchange between the vapor-air mixture and the chamber as being absent at the beginning of evacuation, the expression for q(t) can be represented as

$$q(t) = G_{\text{mix}}(t) \frac{dT}{dt},$$
(3)

where  $G_{\min}(t)$  is the total heat capacity of the vapor-air mixture in the chamber.

It is obvious that

$$G_{\rm mix}(t) = M_{\rm mix}(t) c_{\rm mix}, \qquad (4)$$

The mass of the mixture in the chamber  $M_{mix}$  will be calculated by the formula

$$M_{\rm mix}(t) = \frac{V_{\rm ch}V_0}{V_0 + St} \frac{P_0 \mu}{RT_0},$$
(5)

where  $V_0 = V_{ch} + V_{pipe}$  is the initial volume of the mixture in the entire evacuation circuit, which consists of the chamber volume  $V_{ch}$  and the pipeline volume  $V_{pipe}$ ;  $P_0$  and  $T_0$  are the initial pressure and temperature of the mixture;  $\mu$  is the molecular weight.

Employing (2)-(5), with allowance for the fact that

$$T(t) = T_0 \left(\frac{V_0}{V(t)}\right)^{1-\kappa},$$
(6)

we write the final expression for q(t)

$$q(t) = -\frac{(\kappa - 1) SV_{ch}V_0^{2-\kappa} P_0 \mu c_{mix}}{R (V_0 + St)^{3-\kappa}}.$$
(7)

From (7), it can be seen that the strength of the negative heat sources in the chamber decreases quite rapidly with time, the magnitude of q(t) being the smaller, the smaller the chamber volume  $V_{ch}$ . This governs one of the methods

for reduction of the thermal action of the vapor-air mixture on the sample in its adiabatic expansion by decreasing the size of the primary transducer.

Since moisture from the sample begins to evaporate intensely as time  $t^*$  passes from the beginning of evacuation (Fig. 1), i.e., when the pressure of the mixture in the chamber is equal to the pressure of saturated vapors  $P_{\text{vap}}$ , the adiabatic equation can be written as

$$P_0 V_0^{\kappa} = P_{\text{vap}} \left( V_0 + S t^* \right)^{\kappa}, \tag{8}$$

from which we obtain

$$t^* = \frac{V_0}{S} \left( \left( \frac{P_0}{P_{\text{vap}}} \right)^{1/\kappa} - 1 \right).$$
(9)

Thus, we have derived a formula for the time of the beginning of intense evaporation which permits better selection of the delay time for the beginning of measurements  $t_{del}$ .

The decrease in the chamber size, in addition to the reduction in q(t), also leads to a decrease in  $t^*$ , which impoves the speed of the method.

Since the investigated thermal noise is complicated in character, measurement of its maximum  $\Delta T_{ext}$  (Fig. 1) and its subsequent subtraction from the result of measuring the sample temperature make it impossible to substantially decrease the effect of the noise on the accuracy of measurement. Therefore it is most rational to structurally change the parameters of the vessel and the entire primary transducer with the aim of reducing the thermal noise to a permissible level.

Let us consider the decrease in the thermal noise due to the introduction of thermal protection of the vessel with the sample. The sample 1 (Fig. 2) is placed under a cover 2 of heat-insulating material. Holes are drilled in the cover for the escape of moisture from the sample. The vessel's bottom is also covered by cell of heat insulation 3. The problem is in selecting the material and thermal-protection thickness so that the variation in sample temperature due to a thermal disturbance from the undisturbed value of the temperature does not exceed  $\Delta$ .

Let us formulate a thermal statement of the problem. In connection with the fact that consideration is given to the initial time interval, during which the thermal disturbance penetrates only to distance l from the boundary, it will be reasonable to employ a half-space model rather than a plate one, which makes solution of the problem substantially simpler. A mathematical formulation of the problem for a boundary condition of the third kind has the form:

$$\frac{\partial T(x, t)}{\partial t} = \alpha \frac{\partial^2 T(x, t)}{\partial x^2}, T(x, 0) = T_0,$$

$$\left(\lambda \frac{\partial T}{\partial x} + \alpha_T (T_{\text{med}} - T)\right) \Big|_{x=0} = 0; \quad \frac{\partial T}{\partial x} \Big|_{x \to \infty} \to 0.$$
(10)

Let us write the solution in criterial form. Let  $Fo_x = at/x^2$  denote the Fourier number for the point with the coordinate x and  $Bi_x = hx$ , where  $h = \alpha_T/\lambda$  is the relative heat-transfer coefficient, denote the Biot number.

Thus, the solution of the problem can be written [6] as

$$\Theta(x, t) = \frac{T(x, t) - T_0}{T_{\text{med}} - T_0} = \operatorname{erfc}\left(\frac{1}{2\sqrt{Fo_x}}\right) - \exp(\operatorname{Bi}_x + (\operatorname{Bi}_x)^2 \operatorname{Fo}_x)\operatorname{erfc}\left(\frac{1}{2\sqrt{Fo_x}} + \operatorname{Bi}_x\sqrt{Fo_x}\right).$$
(11)



Fig. 3. Quantity  $\Delta/(T_{med} - T_0)$  versus heat-insulation thickness l, m.

Fig. 4. Relative error of measuring moisture content as a function of initial temperature head (1)  $U_0 = 0.005$ , 2) 0.5).  $\vartheta_1^0$ , K.

To calculate the heat-insulation thickness, we took the following values of the parameters: t = 30 sec (the maximum time in the dynamic regime of measurements);  $\alpha_T \approx 60 \text{ W/m}^2 \cdot \text{K}$  [1, 7, 8].

The experiments showed that the use of porous materials with low thermal conductivity, for example, foam plastic, as heat insulation is undesirable, because of adiabatic expansion of the air in the pores of the material itself. Therefore, we took nonmetallic structural materials with a sufficiently low thermal conductivity (textolite, Plexiglas, etc.). Figure 3 shows  $\Delta/(T_{med} - T_0)$  as functions of the heat-insulation thickness for Plexiglas (1), paper-based laminate (2), and textolite (3). From the plots, it is evident that, for the thickness l = 5 mm, the effect of the temperature disturbance in adiabatic expansion of the mixture does not exceed 2% of the undisturbed state. In a real situation, the effect will be even smaller, since the measurement time in the dynamic regime for different moist materials lies within 10 to 30 sec.

Furthermore, to reduce still further the effect of the considered thermal noise, we developed a new design of the vacuum chamber, in which, in addition to the introduction of thermal protection, the volume of the vapor-air mixture is significantly decreased and the chamber itself is made of material of high thermal conductivity, such as aluminum.

The experiments showed that the maximum value of the noise  $\Delta \tilde{T}_{ext}$  does not exceed the measurement error for the temperature and is approximately 0.02-0.05 K.

Based on analysis of thermal processes in the sample-vessel-medium system, we proposed a mathematical heat- and mass-transfer model for a TV method of measuring moisture content. It is a system of ordinary differential equations for the superheatings of the sample  $\vartheta_1$  and the vessel  $\vartheta_2$  with respect to the medium temperature:

$$C_{1} \frac{d\vartheta_{1}}{dt} + \sigma \left(\vartheta_{1} - \vartheta_{2}\right) = -Q_{0} \exp\left(-\alpha_{u}t\right),$$

$$C_{2} \frac{d\vartheta_{2}}{dt} + \sigma \left(\vartheta_{2} - \vartheta_{1}\right) + \sigma_{\text{med}}\vartheta_{2} = 0, \quad \vartheta_{1}\left(0\right) = \vartheta_{1}^{0}, \quad \vartheta_{2}\left(0\right) = \vartheta_{2}^{0},$$
(12)

where  $Q_0$  is the strength of the internal heat sources (sinks) in the sample  $(Q_0 \sim U_0)$ ;  $C_1$  and  $C_2$  are the heat capacities of the sample and vessel;  $\sigma$  and  $\sigma_{med}$  are thermal conductances between the sample and vessel and the vessel and medium.

Analysis of the solution for  $\vartheta_1$  shows that the effect of the initial superheating of the sample  $\vartheta_1^0$  turns out to be insignificant in the dynamic regime of measuring the moisture content. In particular, for  $\vartheta_1^0 = 15$  K, the relative error is 5% when  $U_0 = 0.005$  and 0.2% when  $U_0 = 0.5$  (Fig. 4). The investigations which had been performed earlier showed that the initial temperature difference between the sample and the walls of the primary transducer's chamber is the main source of the error in measuring by the traditional extremum thermovacuum method [2, 3]. Application of continuous temperature compensation in the secondary transducer [4] permits a decrease in this type of error to a level comparable with the error of signal recording. When estimating the dynamic measurement error for the temperature  $\Delta_{dyn}$ , which is calculated by the formula [9]

$$\Delta_{\rm dyn}\left(t\right) = -\varepsilon \, \frac{dT\left(t\right)}{dt}\,,\tag{13}$$

we should note that  $\Delta_{dyn}$  is mainly governed by the magnitude of the thermal lag of the transducer  $\varepsilon$ , which depends on the heat capacity of the transducer and the thermal conductances of the transducer with the medium, sample, and various elements of the structure. As a result of the earlier investigations with semiempirical methods we derived the expression for the dynamic error of temperature recording [10]

$$\Delta_{\rm dyn}(t) = \vartheta_1^0 \left[ 0.54 \exp\left(-\frac{t}{0.98}\right) + 0.46 \exp\left(-\frac{t}{0.35}\right) \right].$$
(14)

The magnitude of  $\Delta_{dyn}$ , within 4-5 sec after the beginning of measurement, decreases to a level of 0.05 K, and when it is considered that the time to measure the moisture content for the dynamic regime of measurement is 10-30 sec [11] in the thermovacuum method  $\Delta_{dyn}$  can be disregarded.

Variations in the thermophysical properties of the sample (heat capacity, heat conduction, density, dispersivity, heat of vaporization) in the middle range of moisture content (to 25%) lead to a relative error of  $\sim 4\%$ .

In connection with the fact that in the dynamic regime of measurement, an informative parameter is the coefficient of moisture desorption  $\alpha_{\mu}$  [5]

$$U_0 \sim \frac{1}{\alpha_u},\tag{15}$$

one of the main factors that affect the accuracy of measurement is the rate of vapor-air mixture pumping from the chamber, whose variation can attain 10%. Application of flow-rate stabilizers eliminates this undesirable effect.

The complex analysis of methodological and instrumental errors performed enabled us to reveal and to eliminate the principal errors, which are additive and multiplicative in character, as a result of which the principal error of the moisture meter for a moisture content of under 3% is no more than 0.1-0.2% of abs. moisture content, while for a moisture content of 50% it is 7% of relative humidity.

## NOTATION

 $U_0$ , U, initial and current moisture contents of material; n, polytropic exponent;  $\kappa$ , adiabatic exponent;  $T_0$ , T, initial and current temperatures, K;  $T_{med}$ , medium temperature, K; S, vacuum-pump capacity,  $m^3/sec$ ; q, heat power of cooling in vapor-air mixture, W;  $C_{mix}$ , total heat capacity of vapor-air mixture in chamber, J/K;  $c_{mix}$ , specific heat of vapor-air mixture in chamber, J/(kg·K);  $M_{mix}$ , mass of vapor-air mixture in chamber, kg;  $V_0$ , mixture volume in the entire evacuation circuit,  $m^3$ ;  $V_{ch}$ ,  $V_{pipe}$ , chamber and pipeline volumes,  $m^3$ ;  $\mu$ , molecular weight of moisture, kg/mole; R, universal gas constant, J/(mole·K);  $P_{vap}$ , pressure of saturated vapors, Pa;  $t^*$ , time of the beginning of desorption once the vacuum pump is switched on, sec;  $\Delta T_{ext}$ , maximum change in the sample temperature, K; l, sample thickness, m;  $\lambda$ , thermal conductivity of material, W/(m·K);  $\alpha_T$ , heat-transfer coefficient, W/(m^2·K); Bi, Fo, Biot and Fourier numbers; h, relative heat-transfer coefficient,  $m^{-1}$ ;  $C_1$ ,  $C_2$ , total heat capacities of sample and vessel, J/K;  $\sigma$ ,  $\sigma_{med}$ , thermal conductances between sample and vessel, vessel and medium, W/K;  $Q_0$ , strength of internal heat sources in sample, W;  $\vartheta_1$ ,  $\vartheta_2$ , superheatings of sample and vessel, K;  $\Delta_{dyn}$ , dynamic measurement error for temperature, K;  $\varepsilon$ , thermal inertia of transducer, sec.

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