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METHOD OF CRITICAL-TEMPERATURE DETERMINATION FOR INDICATOR MATERIALS

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UDC 536.522.3

A method is described for measuring the switching temperature of an indicator coating in the range 50-1200°C.

Temperatures can be monitored by means of temperature-sensitive coatings, which provide rapid, fairly simple, and exact monitoring of thermal processes [1, 2]. Sometimes such coatings are used in conjunction with traditional temperature transducers to provide additional information on the thermal state, but sometimes they are the only devices that can be employed.

Previously, these indicators were used only when high accuracy was not necessary or when other methods could not be used, but nowadays new types make it possible to measure temperature very precisely, and the error may be comparable with that of many thermometers. For example, the TI indicators [3] clearly allow one to measure a temperature to 0.01 degree. However, the practical accuracy is dependent on the error involved in measuring the critical temperature of the indicator itself.

There is therefore a need for precision methods of measuring switching temperatures, since this is the only way the devices can be fully utilized; the phase transition in such an indicator always occurs at the same temperature, no matter what the conditions of use. The problem is therefore to determine the melting point with the highest precision.

Current methods of switching-point measurement employ visual definition of the melting boundary or color transition in conjunction with temperature measurement at the boundary [1, 4]. There is a subjective error in visual measurements, and this results in a substantial spread in the results, even though it is usual to perform a series of measurements in order to obtain a reliable result.

We have developed an objective method of switching-point definition that provides very precise melting-point measurement over a wide temperature range.

The indicator is heated along with a thermally indifferent substance, and the melting is detected from the temperature difference as recorded with a differential thermocouple. The temperature difference is plotted as a function of the temperature itself on a chart recorder.

We used standard instruments made in this country; for example, the TPP thermoelectric thermometer was used with a PDS-021 XY recorder, an F-116 photoelectronic amplifier, and an SUOL oven.

Figure 1 shows the block diagram; the signal from the differential couple passes to the amplifier 5 and then to the Y input, while the X input receives the heating temperature, which is provided by one of the junctions in the differential couple.

The indicator (temperature-sensitive substance and bonding agent) is placed in the quartz crucible and compacted; the crucible is then placed in the oven along with one junction of the differential couple, which is

Translated from *Inzhenerno-Fizicheskii Zhurnal*, Vol. 36, No. 4, pp. 685-688, April 1979. Original article submitted June 15, 1978.

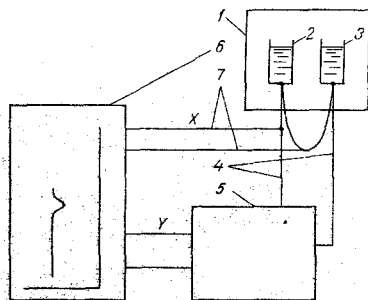


Fig. 1

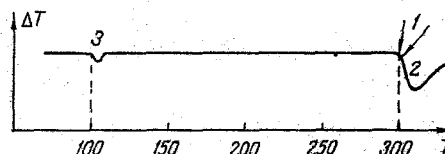


Fig. 2

Fig. 1. Block diagram of the system for determining indicator-switching point: 1) oven; 2) quartz crucible containing indicator; 3) quartz crucible containing standard substance; 4) differential thermoelectric thermometer; 5) photoelectronic amplifier; 6) self-balancing potentiometer; 7) thermoelectric thermometer.

Fig. 2. Recording for determining indicator-switching point; ΔT in relative units and T in $^{\circ}\text{C}$.

TABLE 1. Melting-Point Results for TI-110 Indicator

Method			Method		
1	2	3	1	2	3
111	110	109	108	108	109
107	109	109	111	108	110
109	110	110	109	109	110
112	110	109	108	110	109
107	111	110	108	109	110

connected to the X input of the PDS-021. The other junction is placed in a quartz crucible containing the indifferent substance, which was extra-purity alumina powder.

The two substances are heated together and the differential signal is constant outside the melting range of the indicator (the recording is parallel to the abscissa). The indicator absorbs heat as it melts, which results in a change in the differential emf and a corresponding stepout on the recording. The temperature corresponding to the onset is the switching point of the indicator. Figure 2 shows the characteristic form of a recording.

The onset of melting is indicated by a sharp stepout if the substance is of high thermal conductivity or if the entire volume melts almost simultaneously. The powder used in our technique does not give such a sharp stepout; instead, the onset is more or less smooth (Fig. 2, region 1). This is due to temperature differences between the central and other parts of the specimen. The deflection curve becomes rectilinear in the steady state (part 2). The true melting point is deduced from the start of this linear part [5].

During the calibration of certain indicators, the differential trace shows a slight kink around 100°C (part 3). This is due to evaporation of water in the powder.

The equipment was calibrated throughout the working temperature range before measurements were made on a batch of indicators; the standards in the calibration were extra-purity substances with exactly known melting points.

The differential couple indicates the melting point with high precision (to 0.01°), and this implies much more accurate measurement (by comparison with visual definition of the change in state of aggregation). Also, the chart recording eliminates subjective error. The apparatus can provide virtually any heating rate (up to hundreds of degrees per second).

Table 1 gives results for TI-110 fusible indicators examined by the following methods: 1) with the indicator on a metal plate bearing a single thermoelectric thermometer [1]; 2) with the specimen on a shaped plate bearing several thermoelectric thermometers [4]; and 3) with a differential thermoelectric thermometer.

The instrumental error in each case was not more than 1%, but the table does show that the spread in the results is over 1%, which is due to the random errors arising from visual determination.

The standard deviation may be employed [6] to determine the purely random error of measurement in each method: one assumes that the data from a series of measurements fit Student's distribution, and a confidence limit σ of 0.99 is adopted (σ is the probability that the true result falls in the confidence range), in which the results for the TI-110 indicator are as follows: first method $(109 \pm 1.8^\circ\text{C})$, second method $109.5 \pm 1^\circ\text{C}$, and third method $109.5 \pm 0.5^\circ\text{C}$.

The theory of errors [6] shows that not less than 25 measurements are required in the first method to obtain the melting point with an error of not more than 1%, as against not less than ten measurements in the second method, whereas the third method does not give a spread over 1% in any case.

This method was used to measure the switching points of indicators developed at the All-Union Lumino-phor Research Institute, as well as for extra-purity substances with melting points in the range $50\text{--}1200^\circ\text{C}$ [7]. These indicators have been used to advantage in research on the thermal processes in the VV-12 high-through-put glass-blowing machine.

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THEORY OF A CAPILLARY VISCOMETER

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UDC 532.517.2

Problems in the theory of a capillary viscometer are considered where the test fluid flows through the capillary under a varying pressure drop. The effect of transients on the flow is evaluated.

Calculation of the viscosity according to the capillary method is based on the dependence of the volume V of incompressible fluid which during time τ flows through a capillary of radius R and length L on the constant pressure difference ΔP between both ends, namely

$$\eta = \frac{\pi R^4 \Delta P \tau}{8VL} \quad (1)$$

Relation (1) was first established experimentally [1] and then confirmed theoretically on the basis of the steady-state solution to the Navier-Stokes equation [2].

In practical viscometry one often uses instruments where the test fluid flows through the capillary under a varying pressure drop $\Delta P(\tau)$. Most common among such instruments is the Golubev-Petrov viscometer [3]. With it has been measured the viscosity of water, ammonia, carbonic acid, noble gases, alcohols, and other fluids over wide ranges of states. According to the conventional method by which test data obtained with a

Translated from *Inzhenerno-Fizicheskii Zhurnal*, Vol. 36, No. 4, pp. 689-694, April, 1979. Original article submitted April 6, 1978.