THE INFLUENCE OF THE SAMPLE GEOMETRY ON THE ACCURACY OF THE HEAT CAPACITY EVALUATION

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A new simple modification of the heat capacity evaluation of small samples with low thermal diffusivity is considered. The temperature-time response of the specimen to a step-wise Joule heating power $\phi=I_0^2 R_e$ is measured. The specimen was connected to a sink by a finite thermal resistance r_0 of the heater and thermocouple leads in the thermal short circuit at the sample. Such an essential part of a calorimetric system may be presented by a circuit given in Fig.1a. All parasitic heat capacities can be included in c_0, while a line of $n \gg 1$ distributed $r_i c_i$ elements may be to a good approximation equivalent to the sample itself with the total heat capacity c_s and the thermal resistance r_s. A final temperature rise of the specimen corresponding to its thermal equilibrium with sink is $\Delta T=\phi r_0$ [1], while the complete response depends on chosen "thermal geometry" of the sample.

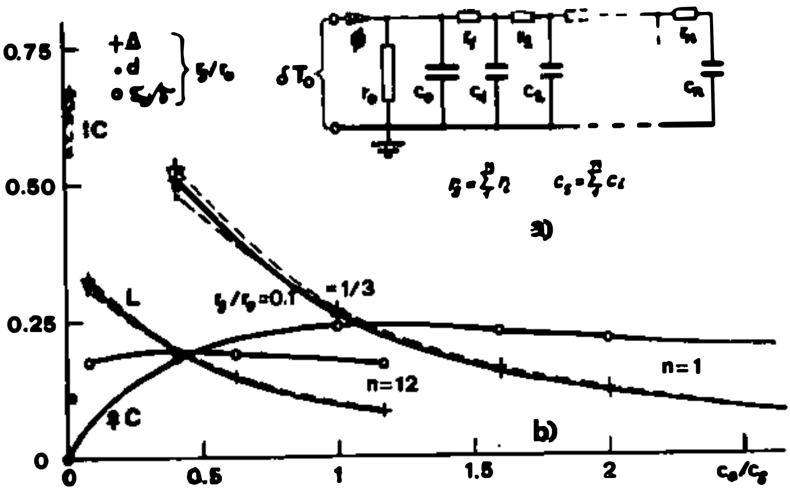

Fig.1 a) An equivalent circuit with distributed $r_i c_i$ elements; b) Calculated parameters of the equivalent response: the correction Δ of the main time constant τ_{s0}, the relative amplitude d and small time constant τ_{s1}/τ in dependence of c_0/c_s.

Namely, different mutual relations of $r_i c_i$ elements correspond to the same, for example, cylindric-like sample, depending on whether the heat pulse is supplied to the base, to the outer cylinder, or to an inner coaxial cylinder. It gives: linear system L with equal $r_i c_i$ elements of the equivalent circuit, a cylindrical system C with heat capacites falling down from the maximal input capacity c_0 to the minimal c_n at the end of rc line and with resistances correspondingly increasing from r_1 to r_n, or inversely, for an inverse cylindrical system IC. To find associated equivalent temperature-time responses, calculations were performed by a computer. A general dimensionless form of the response was obtained, including a set of exponential terms:

$$\delta T_0/\Delta T = 1-(1-d)\,e^{-t/\tau_{s0}} - \sum_1^n d_s\, e^{-t/\tau_{s\ell}}$$

The first term has a large time constant $\tau_{s0} \doteq \tau(1+\Delta) = r_0(c_0+c_s)(1+\Delta)$, due to the specimen-to-sink relaxation, and the others have small relative total amplitude d and small equivalent time constant τ_{si}, due to the internal relaxation of the specimen itself, with $d=\sum_1^n d_\ell$ and $\tau_{si}=\sum_1^n \tau_{s\ell}$.

Calculated parameters Δ, d and τ_{si}/τ expressed in values of r_s/r_0 in dependence of c_0/c_s are given in Fig.1b. They are presented for n=1, for the most simplified circuit, and for n=12, when above parameters slowly reach their saturated values. It can be seen that per cent amount of the main time constant correction Δ is approximately equal to the relative amplitude d for r_s/r_0 up to 1/3. Because d can be determined from the response, by extrapolating the single exponential part of the curve (for $t > \tau_{si}$) to its origin, the c_s may be evaluated from $\tau \doteq \tau_{s0}/(1+d)$ inside the promille accuracy. One also can conclude, due to the smallest values of Δ=d and τ_{si}/τ, that the cylindrical C system will be the most suitable one. Analogously, large parasitic capacities c_0 (of a metal container, for example) if accurately determined, should be preferred in the L system. On the contrary, the IC system with markedly larger value of Δ, which at the same time rather more differs from d, is the most unfavourable one. However, these conclusions will valid only if the transfer resistance from the heater to the sample could be neglected in respect to the thermal resistance of the sample itself. Otherwise, the advantages, even of the C system, will vanish. More detail discussion about the influence of the complete specimen arrangement on the accuracy of the heat capacity evaluation, based on computer calculations, will be published in J.Phys.E:Sci.Instrum.

We wish to point out here, that the relative measurements on different types of samples, from metals to semiconductors and liquid crystals, follow high theoretically possible precision of a few promilles, while the absolute measurements, including also inaccuracies of c_0 and r_0, remain inside a few per cent.

References

1) D.Đurek and J.Baturić-Rubčić J.Phys.E:Sci.Instrum. 5 (1972) 424