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Design and performance of the S6 calorimeter

Lars Wadsö

Design and performance of the S6 calorimeter

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1 Summary

The S6 calorimeter is a simple isothermal heat conduction calorimeter for measurements on six samples of up to a few 100 ml in size. Each sample cup is placed on three thermocouple plates that are in contact with a large heat sink. The calorimeter is not actively thermostated, but only protected from the ambient environment by a 50 mm polystyrene insulation.

2 Nomenclature

C	heat capacity	J/K
E	Seebeck coefficient	V/K
k	thermal conductivity	W/K
P	thermal power	W
q	heat flow rate	W
T	temperature	K
ε	calibration coefficient	W/V
λ	heat flow loss factor	1
τ	time constant	s

indices:

0	ambient
1	heat sink
2	measurement cup
3	reference cup

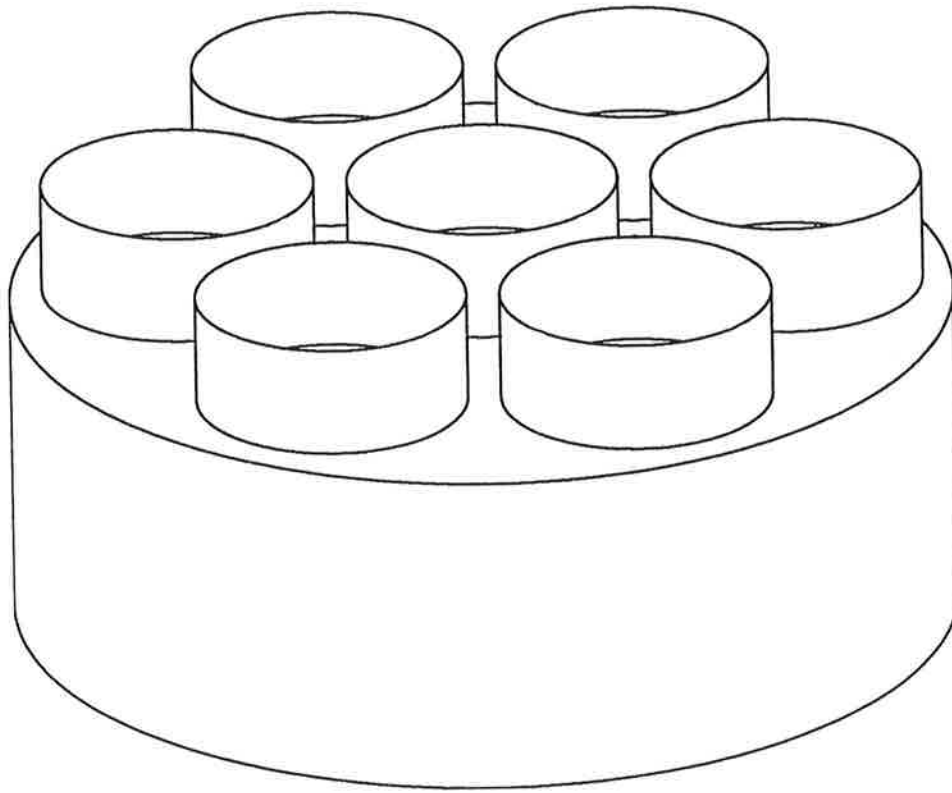


Figure 1: A schematic drawing of the S6 calorimeter which shows the seven cups and the heat sink. Three heat flow sensors (not seen in the figure) are placed between each cup and the heat sink.

3 Design

Figure 1 shows the design of the calorimeter. Six measurement cups are placed on the heat sink around one reference cup. Between each of the cups and the heat sink three serially connected thermocouple plates (Melcor CP1.4-71-045L) are placed as heat flow sensors. The thermocouple plates are only mounted with Melcor's thermal interface pads (no bolting etc.). The voltage measured in the reference cup is subtracted from each of the voltages from the measurement cups (differential measurement).

The heat sink is an aluminum can filled with water (and aluminum shavings to prevent temperature layering of the water). It has a diameter of 400 mm and an height of 120 mm. The top and the bottom of the can are made of

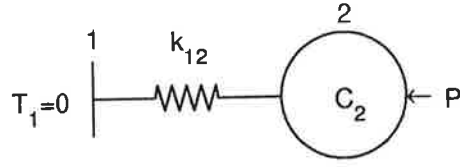


Figure 2: The simple thermal system used to derive the Tian equation.

12 mm aluminum and the mantle is made from 3 mm aluminum. Under each cup on the bottom side of the top part are placed finned cooling elements of aluminum to get a good thermal contact between the top of the calorimeter and the heat capacity of the water. Five millimeter from the edge of the top and bottom parts groves are made into which a large o-rings and the mantle fits. The whole can is held together by six long screws going from the top to the bottom part (through the water). There is also a hole for filling and emptying the water. A large screw closes this hole. Each sample cup is made from the bottom part of a small aluminum saucepan with 1.2 mm material in the mantle and 4 mm in the bottom.

4 Theory

The simplest view of a calorimeter of the S6 type as a thermal system is as shown in Fig. 2. With this model the produced thermal power P (W) may be calculated from the measured voltage U (V). The sample and the sample cup has a heat capacity C_2 that is connected to the constant temperature heat sink through the effective thermal conductivity k_{12} of the three thermocouple plates and other heat flow paths. To simplify the calculations we set the temperature of the heat sink to 0.

If k_{TCP} and E_{TCP} are the properties of a single TCP, the following values are the effective values for the three TCP:s for each cup in the S6 calorimeter:

$$k_{12} = 3\lambda k_{TCP} \quad (1)$$

$$E = 3E_{TCP} \quad (2)$$

Here, λ is a factor that corrects for that not all heat that leaves the cup passes through the TCP:s. Even for a very good microcalorimeter λ is usually

thought to be 1.1–1.2. For simplicity the indices for the following parameters are left out in the following: $E = E_{12}$; $U = U_{12}$; $P = P_2$.

The following equations will apply (cf. Fig. 2):

$$q_{12} = k_{12}T_2 \quad (3)$$

$$C_2 \frac{dT_2}{dt} = P - q_{12} \quad (4)$$

$$U = ET_2 \quad (5)$$

If T_2 is replaced by U/E and q_{12} from Eq. 1 put into Eq. 2 the result will be the Tian equation:

$$P = \varepsilon(U + \tau \frac{dU}{dt}) \quad (6)$$

Here, the calibration coefficient ε (W/V) is:

$$\varepsilon = \frac{k_{12}}{E} \quad (7)$$

The time constant τ (s) is:

$$\tau = \frac{C_2}{k_{12}} \quad (8)$$

The temperature of the heat sink may be influenced by the heat flows from the sample cups and from the ambient room through the insulation. This situation is given in Fig. 3. The following equations apply when the same thermal power P is produced in each cup and only slow temperature changes are taken into account (the temperature increase in the cups is not taken into account as C_2 and C_3 are much smaller than C_1):

$$C_1 \frac{dT_1}{dt} = k_{01}(T_1 - T_0) + 6P \quad (9)$$

Usually only one (if any) of the right hand side terms have to be taken into account:

- If the ambient temperature (T_0) is close to the temperature of the calorimeter (T_1) and the heat production in the calorimeter is small, then T_1 will not change significantly.

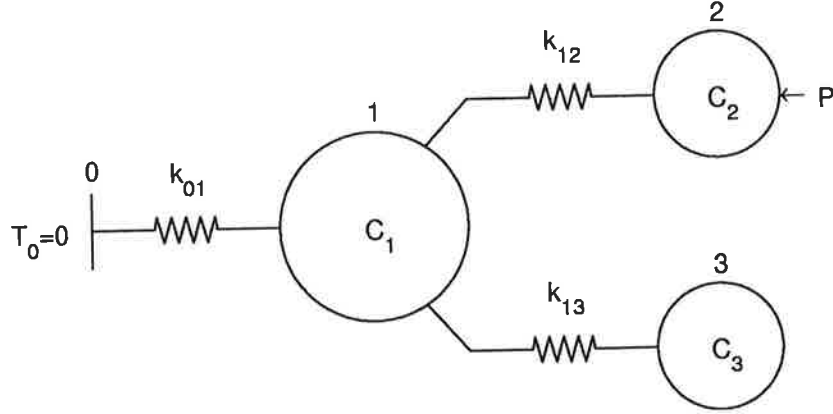


Figure 3: A more complete thermal description of an unthermostated calorimeter.

- If the ambient temperature (T_0) is close to the temperature of the calorimeter (T_1) *but* the heat production in the calorimeter is high, then T_1 will change according to:

$$C_1 \frac{dT_1}{dt} = 6P \quad (10)$$

- If the heat production in the calorimeter is small, *but* the ambient temperature (T_0) differs significantly from the temperature of the calorimeter (T_1), then T_1 will change according to:

$$C_1 \frac{dT_1}{dt} = k_{01}(T_1 - T_0) \quad (11)$$

If the temperature of the heat sink changes with time and the measurement cups do not have the same heat capacity as the reference cup one may have to add a correction for this to the Tian equation:

$$P = \varepsilon(U + \frac{dT_1}{dt}(C_2 - C_3) + \tau \frac{dU}{dt}) \quad (12)$$

This equation is difficult to use in practice as C_2 and C_3 are not well known. However, by assuming a certain difference, e.g. $C_2 - C_3 = 0.3C_2$, one may get an idea of how large the error is if one does not take this correction into account.

5 Performance

The Melcor CP1.4-71-045L thermocouple plates have the following properties (approximate):

$$k = 0.37 \text{ W/K}$$

$$E = 0.025 \text{ V/K}$$

The calorimeter has the following properties (approximate):

$$\text{heat capacity of heat sink: } C_1 = 60000 \text{ J/K}$$

$$\text{thermal conductivity of 50 mm insulation: } k_1 = 0.3 \text{ W/K (calculated)}$$

$$\text{heat capacity of empty sample cup: } C_2 = 140 \text{ J/K (calculated)}$$

$$\text{heat capacity of sample cup with 200 g cement paste: } C_2 = 400 \text{ J/K (calculated)}$$

$$\text{calibration coefficient } \varepsilon = 16 \text{ W/V}$$

$$\text{sensitivity } S = 0.062 \text{ V/W}$$

$$\text{heat flow loss factor } \lambda = 1.1$$

$$\text{time constant (empty cup) } \tau = 120 \text{ s}$$

$$\text{time constant (empty cup) } \tau = 330 \text{ s}$$