# Temperature Calibration of Rotational Rheometers with Electrically Heated Tools and Hood Oven

THOMAS SCHWEIZER

Institute of Polymers, ETH Hönggerberg, 8093 Zürich, Switzerland

Email: thomas.schweizer@mat.ethz.ch

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**Abstract:**
The calibration of the temperature control unit of a rotational rheometer with a hood oven is shown. The calibration technique shown for a Paar-Physica rheometer can be adapted to any rheometer with hood oven (indirect heating). The temperature of the bottom fixed plate and the air bearing suspended cone or plate are measured independently. By keeping the amount of venting gas constant, the set temperature of the hood oven is adjusted to reach a minimum gradient across the measuring gap. The calibration procedure is optimized to keep the oven as close as possible to the measuring position.

**Zusammenfassung:**
Es wird ausgeführt, wie die Temperiereinheit eines Rotationsrheometers mit Haubenofen kalibriert werden kann. Die Kalibriertechnik ist am Beispiel eines Paar-Physica Rheometers gezeigt, kann aber problemlos auf jedes Rheometer mit Haubenofen (indirekter Heizung) übertragen werden. Die Temperaturen der unteren festen Platte und des oberen im Luftlager gelagerten beweglichen Werkzeugs werden separat gemessen. Unter Konstanthaltung des Spülgasstroms wird die Temperaturvorgabe für den Haubenofen so eingestellt, dass ein minimaler Gradient über dem Messspalt resultiert. Die Kalibrierprozedur ist dahingehend optimiert, dass sich der Ofen so nahe wie möglich in Messsposition befindet.

**Résumé:**
Cette étude porte sur la calibration de l'unité d'échauffement d'un rhéomètre rotatif avec un four à capot. La technique est illustrée à l'aide d'un rhéomètre Paar-Physica, mais peut facilement être appliquée à d'autres rhéomètres avec four à capot (échauffement indirect). Les températures du plateau inférieur fixe et de l'outil suspendu au palier à air sont mesurées séparément. En maintenant constant le flux du gaz injecté, la température de consigne du four est ajustée jusqu'à ce que le gradient à travers l'entrefer soit minimal. Le procédé est optimisé de façon à positionner le four à capot le plus près possible de la base du rhéomètre.

**Key words:** plate-plate geometry, indirect heating, thermocouple, venting gas, temperature gradient across gap

## 1 INTRODUCTION

Shear rheometry with polymer melts in a rotational rheometer requires a pair of tools, usually plate/plate or cone/plate. One member is fixed, either to the instrument frame or a non-displacing transducer (ARES, TA Instruments). The rotating member is connected to a measuring motor or an actuator. For temperature control, both members can be surrounded by a gas convection oven. This is simple in design since the tools require no wiring and can be of low inertia. The convection oven can readily be used for different tool geometries, and also for solid samples. However, it is difficult to keep the temperature constant; unless very strong gas flow rates of up to 150 l/min are used (ARES, forced convection oven). Even if the gas temperature changes only by 0.1°C, the effect on the normal force data is serious since the instrument is usually very stiff. The alternative is direct electrical heating. Only the ARES or former RMS800 (Rheometric Scientific) design allows for a direct provision of electrical energy to the tools since the rotating member is driven by a strain controlling device insensitive to friction. Therefore collector rings can be used to feed heating power and read thermosensor signals [1]. The price to pay for the very accurate temperature control is wiring of the tools and an increase of the inertia. The third possibility is indirect electrical heating. In all rheometers with a measuring motor (stress controlled rheometers), the rotating member is sus-



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pended from a low friction air bearing and must not be connected to any device causing friction. In these rheometers, the rotating member is heated via radiation from a hood oven through a 0.5 - 1 mm air gap. Temperature control by indirect heating below the temperature of visible radiation is always susceptible to changes of the gas flow rate or the gap.

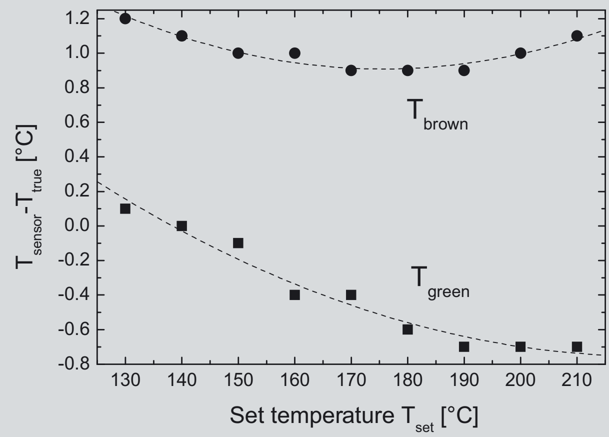
This paper is to show how an accurate temperature measurement can be made in both members of the Paar-Physica rheometers UDS200 and MCR300 and how these results can be used to properly set the heater control parameter "0". Although this report is specific to the Paar-Physica instruments, it can likewise be adapted to similar rheometers of other manufactures.

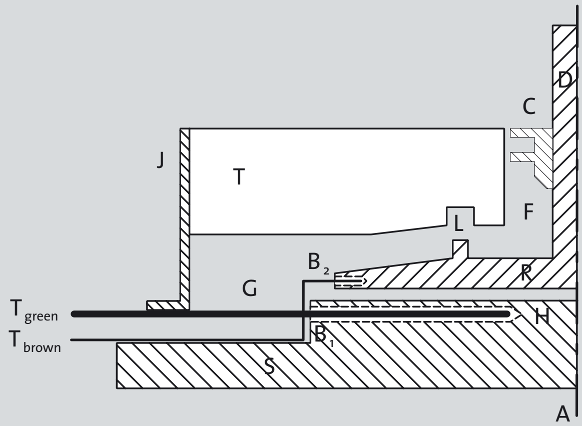
## 2 EXPERIMENT

For all temperature measurements reported, two thermocouples were used. Thermocouple GREEN is jacketed in a stainless steel tube with outer diameter 1.5 mm. Thermocouple BROWN is tissue insulated with an open welding bead of outer diameter 0.5 mm. Both thermocouples were calibrated in a JOFRA ATC-320B temperature calibration station from METEK Calibration Instruments, Largo, Florida. To read the thermocouple's temperatures, a DTM-506RS display from Monacor was used. The calibration results for both thermocouples are shown in Fig. 1.
In order not to depend on surface temperature measurements for the rotating member, a hole $B_2$ 0.6 mm in diameter and 3 mm deep was drilled in the rim of the tool to receive thermocouple BROWN.

The temperature is measured independently in both members. A gap of 1 mm is kept between both plates. A temperature calibration with a thermocouple inserted in a sample between the plates has been reported by Zhang & Martins [2]. However, this calibration procedure does not yield information about thermal gradients in the sample.

Fig. 2 shows the calibration set-up A, as formerly used. Thermocouple BROWN is inserted into bore $B_2$ with some $Al_2O_3$-thermoconductive paste and bent to accommodate the step in the stationary member. Thermocouple GREEN is inserted into bore $B_1$ (∅ 2.9 mm) which protrudes to the centre of the heating plate. Then the oven is lowered until the jacket J touches thermocouple



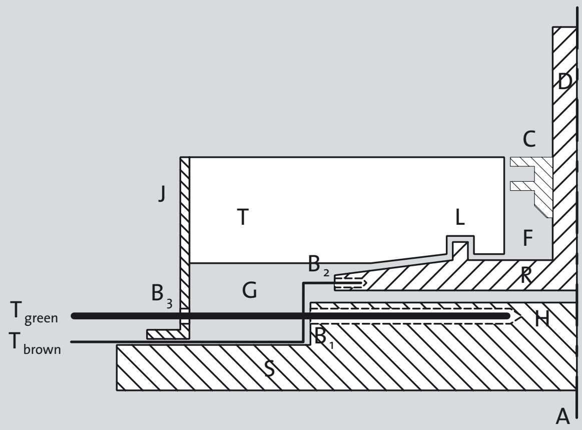




*Fig. 1 (above): Calibration curves for the two thermocouples used. Details on the instruments used for temperature display and calibration are mentioned in the text.*

*Fig. 2 (middle): Schematic cross section through rheometer with plate/plate geometry. Calibration set-up A. C collar to reduce draft, L labyrinth sealing, $B_1$ bore one, diameter 2.9 mm, $B_2$ bore two, diameter 0.6 mm, $B_3$ bore three through jacket, diameter 2 mm, J jacket of hood oven, T heating block of oven, S stationary member, R rotating member, G venting gas volume of oven, D driving shaft of rotating member, A axis of rotation, F annular funnel.*

*Fig. 3 (below): Schematic cross section through rheometer with plate/plate geometry. Calibration set-up B.*

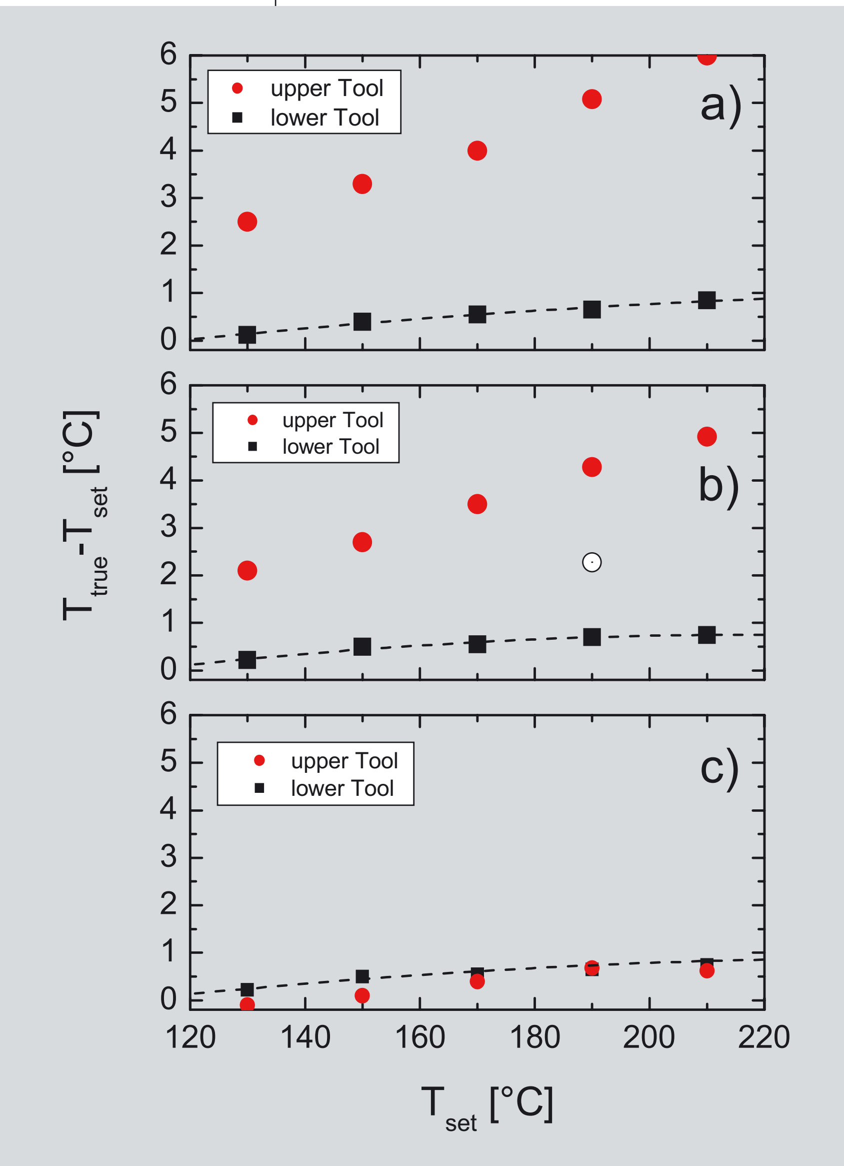



*Fig. 4a: True temperature in both plates of MCR300 rheometer. Parameter o = 127, gap = 1 mm, calibration set-up A (Fig. 2). 5 l/min of air flowing to the hood. No shaft cooling.*

*Fig. 4b: True temperature in both plates of MCR300 rheometer. Parameter o = 127, gap = 1 mm, calibration set-up B (Fig. 3). 5 l/min of air flowing to the hood. No shaft cooling. The dotted circle shows the effect, when additional 5 l/min of air are used for shaft cooling.*

*Fig. 4c: True temperature in both plates of MCR300 rheometer. Parameter o = 118, gap = 1 mm, calibration set-up B (Fig. 3). 5 l/min of air flowing to the hood. No shaft cooling.*

GREEN. This leaves about 2.5 mm (MCR300) or 4.3 mm (UDS200) between the lower rim of the jacket and the bottom plate of the stationary member. Now, with 5 l/min of nitrogen or air flowing into the hood (measuring conditions), no shaft cooling, and the collar C installed to reduce draft, the temperature is increased stepwise and both temperatures read after equilibration. Using the thermocouple calibration (Fig. 1), the true temperature can be determined as shown in Fig. 4.

Fig. 3 shows the calibration with set-up B, as used now. For this, a hole $B_3$ (∅ 2 mm) was drilled in the jacket, at the same height as the hole $B_1$ in the bottom plate. The peripheral position of $B_1$ is best chosen at a fin of the plastic cage. Thus it is easy to file a small slot into the latter without splitting it. Through this hole, after insertion of thermocouple BROWN in hole $B_2$ and lowering of the oven to the lowermost position, thermocouple GREEN can be pushed into hole $B_1$. This set-up has the advantage that the oven is almost at measuring position, because the wires of thermocouple BROWN are only 0.5 mm thick.

## 3 RESULTS AND DISCUSSION

In the case of the UDS200 and MCR300 from Paar-Physica, the set temperature of the hood oven is given by the set temperature of the bottom plate $T_{set}$ plus some extra $\Delta T_{set}$ to compensate for heat losses due to convection. $\Delta T_{set}$ is controlled by the parameter "o" to be specified in the settings of the temperature controller TC20. The function $\Delta T_{set}(o, T_{set})$ has been determined by the manufacturer and is not accessible to the user. For "o" = 100, the same set temperature as for the bottom plate is used.

Fig. 4a shows that the upper temperature for "o" = 127 (setting at shipment of rheometer) is much too high. With a sample in, the upper temperature will be lowered a bit. However, it seems reasonable to look for conditions, where no temperature gradient exists without sample. Using calibration set-up B in Fig. 4b, the situation is already improved. As shown in Fig. 3, the labyrinth sealing L is only effective in this position of the hood oven and less venting gas can escape through the funnel F. With calibration set-up B, the parameter "o" was successively lowered, until no gradient existed any more, as shown in Fig. 4c.

The dashed lines in Figs. 4a to c are quadratic fits to the true bottom plate temperature. As can be seen, it does not depend strongly on calibration conditions. Therefore, for a test, this line $T_{true} = f(T_{set})$ is used to determine the proper set temperature to be entered in the temperature controller TC20 to get the desired measuring temperature. Once the parameter "o" has been determined, the true temperature in the instrument can be checked at any time, since the bottom plate temperature is independent of the calibration set-up and gas flow rate, and there is a bore $B_3$ in the jacket.

## 4 CONCLUSION

The following conlusion can be drawn:

- A direct measurement of the temperature in both members is necessary to set the upper heater control parameter "o" properly and to determine the function $T_{true} = f(T_{set})$.
- The temperature measured in the rotating tool is quite sensitive to the separation of the oven from the bottom plate.
- The temperature in the stationary member is not affected by the parameter "o". Therefore, the measurement of only the lower temperature can give you a wrong feeling of accuracy of the whole heating set-up.
- A temperature calibration performed under not measurement-like conditions can lead to errors.
- Once the proper parameter "o" is determined, the true temperature in the sample can be determined without lifting the hood oven and even during a test, thanks to the small hole in the jacket.
- Gas flow rate, hood oven position, and the placement of the collar should always be the same, if reproducible temperatures are expected in indirect heating devices.

## REFERENCES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