

EXPERIMENTAL STUDIES AND MODEL CALCULATIONS ON HEAT ACCUMULATION TESTS USING DEWAR VESSELS

H. Fierz and R. Gyax  
Central Research Laboratories, CIBA-GEIGY Ltd., Basle

ABSTRACT:

Measurements of the thermal response of systems, where heat is transported primarily by conduction, and comparison of the results with theoretical models.

Estimates of the contributions of heat loss from Dewar vessels of different sizes due to conduction, radiation and convection, assuming a sample of uniform temperature, and comparisons to values obtained from cooling curves.

For safety problems related to the self heating of chemicals during storage, the Dewar heat accumulation test is still widely used in industry [1], mainly because of its simplicity.

For highly viscous and solid samples, however, an exact picture of the heat loss of such a system, i.e. Dewar vessel and sample is still lacking. The predominant mechanism of heat transfer within such samples is conduction and the temperature within the sample is found to be a function of both time and the position of the thermocouple.

If we assume a standard Dewar vessel with 0.04 m internal diameter and 0.170 m height with a ratio of internal surface area to volume of 106/m, and heat conduction only along the vessel axis, the critical heat production rate would be very low: calculation according to Frank-Kamenetskii's formula [2] for  $\delta_{crit}$  gives ca. 30 mW/kg. The actual measured value is much higher: ca. 200-600 mW/kg.

The aim of this paper is to give some, although rather qualitative, insight into the mechanism of heat transfer from a Dewar vessel. For lack of space, only the results and short comments on how they were obtained can be presented.

### MEASUREMENTS OF THERMAL RESPONSE

The thermal response of a Dewar vessel filled with an inert substance was measured. A smaller vessel of 100 ml volume was filled with finely ground aluminum oxide, placed in an oven, and the temperature response to a sudden jump in the oven temperature measured with thermocouples placed along the axis on the top, in the middle and at the bottom of the vessel.

The resulting response curves were compared to

- a) response curves calculated for an infinite slab [3],
- b) the results obtained by a computer simulation model, which allows the calculation of the thermal response for cylindrical systems with the additional inclusion of heat transfer across the wall [4]. The values for the radial heat transfer coefficients for the top, the side and the bottom can be different.

Neither model a) nor model b) could explain the observed temperature response. Clearly, there must be an additional mechanism of radial heat transfer different from that across the wall.

### ESTIMATION OF THE DIFFERENT CONTRIBUTIONS TO HEAT LOSS

The results are summarized in Table 1.

The contributions are calculated according to [3] for Dewar vessels of different capacity, i.e. 100 ml, 200 ml, 500 ml, with an internal length of 0.095 m, 0.17 m, 0.21 m and an internal diameter of 0.04 m, 0.04 m and 0.057 m respectively. We assume the sample to be a low viscosity liquid with an uniform temperature. This makes comparisons with experimental cooling curves possible.

#### a) Conduction across the wall

Values must be assumed for the gap and vacuum in the Dewar's double wall. At a pressure of 3 Pascal (ca. 0.02 mm Hg) the mean free path of the molecules in the gap equals the estimated size of the air gap of 0.002 m. If we assume a pressure of about 0.015 Pascal ( $10^{-4}$  mm Hg), a thermal conductivity of ca.  $10^{-4}$  W/m °C is obtained.

b) Radiation across the wall

Values must be assumed for the emittance of silver (ca. 0.02) [4]. At 80 degrees the heat lost by radiation is about 10 mW/kg for one degree temperature difference. Estimates for a temperature difference of 60 degrees are also included, and a mean value for both differences listed in Table 1.

c) Axial conduction along the walls

Heat is lost by axial conduction along the walls. The thermal conductivity of Pyrex is about ten times higher than that of an average organic substance. Heat is, therefore, carried away along the circumference of the vessel. The rate of heat loss along the walls depends on their thickness (assumed: 0.002 m) and on the temperature gradient along them. We assume about 20 °C/m.

d) Heat loss through the opening

Here we assume either natural convection, the heat transfer coefficient being on the order of 5 W/m<sup>2</sup> °C, which corresponds to an open vessel, or we assume the vessel to be closed by a lid of 0.003 m thickness and a conductivity of 0.024 W/m °C, which corresponds to an isolating material such as styrofoam.

For the 100 ml Dewar, the estimated total rate of heat loss rate compares reasonably well to the one measured from cooling curves (see Table 2).

For the 100 ml vessel our conclusions are as follows:

- Radial conduction and radiation losses are comparatively small.
- For the open vessel, natural convection on the surface of the sample and axial conduction along the wall contribute about equally.

The main source of heat loss of a Dewar vessel seems, therefore, to be the glass wall, whose conductivity is an order of magnitude higher than that of an average organic sample.

TABLE 1

Estimations of individual contribution to heat loss of Dewar-vessels:

| CONTRIBUTION  | Heat loss in mW/kg °C |              |              |
|---|-----------------------|--------------|--------------|
|   | 100 ml Dewar          | 200 ml Dewar | 500 ml Dewar |
| a) Conduction across wall                             | 5.5                   | 5            | 4            |
| b) Radiation across wall<br>inner temp.   outer temp. | 11   11               | 8   8        | 7.5   7.5    |
| 81   80   | 8.5   8               | 6   6        | 6   6        |
| 80   20   | 10   10               | 10   10      | 7   7        |
| Mean  | 10                    | 10           | 7            |
| c) Conduction along wall                              | 60                    | 30           | 17           |
| a) Heat loss through surface                          |                       |              |              |
| d1) with lid  | 5                     | 2.5          | 4            |
| d2) without lid                                       | 63                    | 32           | 25           |
| a) + b) + c) + d1)                                    | 81                    | 48           | 32           |
| a) + b) + c) + d2)                                    | 139                   | 77           | 53           |

TABLE 2

Comparison of estimated and measured heat loss rates

|                            | Heat loss in mW/kg °C |        |        |
|----------------------------|-----------------------|--------|--------|
|                            | 100 ml                | 200 ml | 500 ml |
| Estimate from Table 1      |                       |        |        |
| a) + b) + c) + d1)         | 81                    | 48     | 32     |
| a) + b) + c) + d2)         | 139                   | 77     | 53     |
| Measurements according [1] | 160                   | 80     | 40     |
| this work with lid         | 120                   | --     | --     |
| without lid                | 200                   | --     | --     |

## REFERENCES

- [1] Th. Grever, in Runaway Reactions, I. Chem. E. Symposium Series No. 68, (1981).
- [2] P. Gray and P.R. Lee, Oxydation and Combustion Review 2 (1967) 1.
- [3] J.H. Lienhard, A Heat Transfer Textbook, Prentice Hall, New Jersey (1981).
- [4] F. Brogli, M. Burga and K. Dixon-Jackson, unpublished results