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TEMPERATURE GRADIENTS IN HORIZONTAL TUBE FURNACES *

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ABSTRACT

Radial temperature differences in a 1 in. diameter horizontal tube furnace similar to those used in thermogravimetry were measured as a function of purge gas flow rate and pressure at temperatures from 25 to 525°C. The temperature difference between a thermocouple at the tube axis and one 0.8 cm off center was 45°C at 400°C, 1 atm pressure of nitrogen and 25 ml min^{-1} flow rate. This large temperature difference was attributed to laminar flow conditions.

Effects of these large radial variations on temperature measurement and calibration are discussed. Several methods for minimizing these temperature differences by insertion of in-line preheaters and mixers are suggested.

INTRODUCTION

Tube furnaces with horizontal balance beams have been used for thermogravimetric measurement for many years and are presently used in several commercial instruments. They are often of more rugged construction than vertical of "hang down" type furnaces and permit much faster flow rates of purge gas without causing excessive vibration of the specimen container. This latter advantage is in part due to the setting up of a laminar rather than turbulent flow under many frequently applied conditions for temperature, pressure and purge gas rate. However, this advantage is a double-edged sword as it can result in large errors in temperature calibration and measurement. Since temperature is by far the least accurately measured variable, precise temperature measurement is critical to thermogravimetry experiments.

This paper defines some experimental conditions at which large temperature gradients occur. It also emphasizes the importance of temperature sensor placement and suggests both some precautions to be taken in the calibration of temperature and techniques for reducing temperature gradients in flowing gases in horizontal tubes.

* Based on a paper, "Temperature Measurement and Control of TGA", presented at the 5th NATAS Conference, Peterborough, Ontario, Canada, 1975.

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EXPERIMENTAL

Two chromel–alumel (type K) thermocouples were inserted into the furnace tube as shown in Fig. 1. One thermocouple was situated at the axis of the 1 in. diameter tube and the other about 0.5 cm from the side (0.8 cm off center). The temperature of the axially placed thermocouple and the temperature difference between the two thermocouples were measured. The tube was heated by a typical exterior furnace 4 in. in length and the thermocouples were placed 5.3 cm into the furnace from the downstream end of the tube which was attached to a manometer and vacuum system.

RESULTS

The temperature difference vs. the axial temperature are plotted in Fig. 2 for nitrogen at a pressure of 760 Torr and purge gas flow rates of 0.0, 0.4,

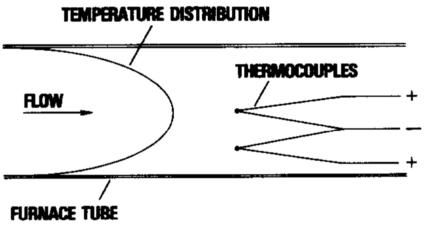

Fig. 1. Thermocouple placement in the furnace tube.

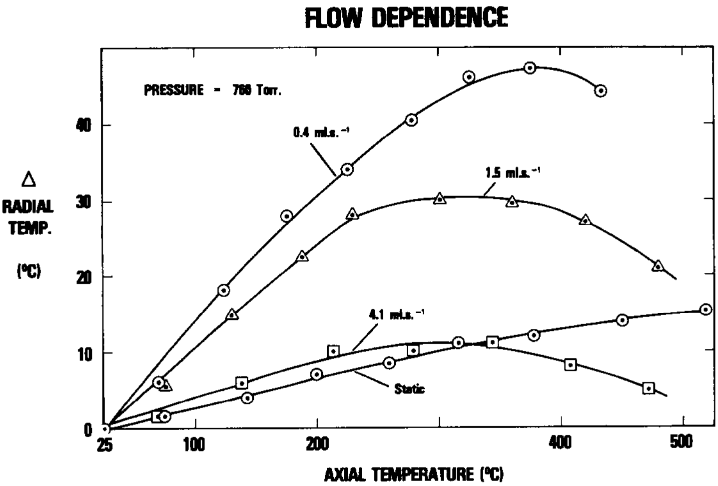

Fig. 2. Purge gas flow dependence of radial temperature difference at 760 Torr for temperatures from 25 to 525°C (radial temperature difference vs. axial temperature).

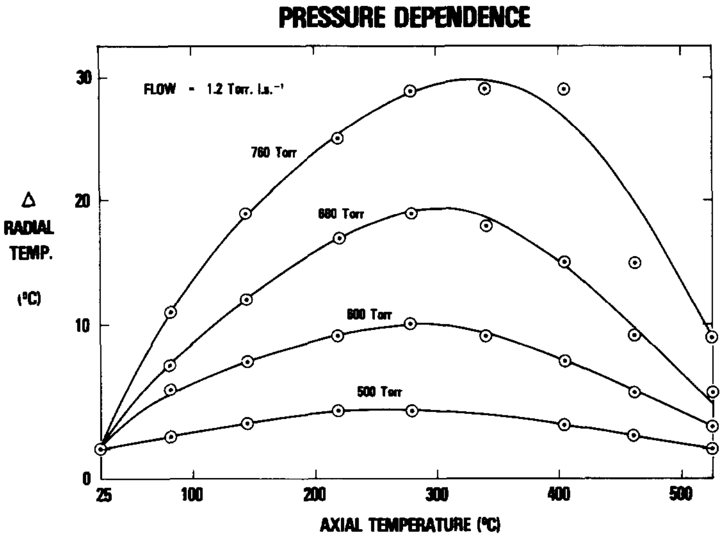

Fig. 3. Pressure dependence of radial temperature difference at 1.2 Torr l s^{-1} flow rate for temperatures from 25 to 525°C (radial temperature difference vs. axial temperature).

1.5 and 4.1 ml s^{-1}. In the extreme case, 400°C and 0.4 ml s^{-1} (24 ml min^{-1}) flow, temperature differences between the two thermocouples of greater than 40°C are found. These results are not surprising as the calculated Reynolds number for nitrogen flow rates of 30–300 ml min^{-1} and temperatures of 25–400°C ranges from about unity to 16. This is near the center of the laminar flow range, and it would have to be thirtyfold larger to be within the turbulent flow region.

The temperature difference for the same system is plotted as a function of the axial thermocouple temperature at a purge gas mass flow rate of 1.2 Torr l s^{-1} for pressures of 760, 680, 600 and 500 Torr in Fig. 3. The difference in temperature between the two thermocouples decreases with decreasing flow gas pressure and is no longer a significant factor at pressures of less than 0.5 atm.

The temperature difference between the thermocouples also changed as a function of distance into the furnace tube. However this effect is relatively unimportant in temperature calibration.

DISCUSSION

The conditions described above are for the worst possible case in which steady state laminar flow has been established and no obstructions are present in the furnace tube to cause turbulence. The entry of the purge gas through a nonsymmetrical orifice of smaller diameter than the furnace tube may disrupt the establishment of steady laminar flow. If the positions of the specimen and temperature sensor with respect to the furnace tube do not change, and the purge gas composition, flow rate and pressure have the same values as during temperature calibration conditions, then errors in temperature measurement may be minimal. On the other hand, if any of the above positional or flow factors are changed, errors in temperature measurement can be expected, and recalibration under the new conditions is necessary. Fortunately nitrogen and oxygen have similar thermal and flow properties so that their interchange in the purge gas is not a serious perturbation. However, this is not the case for helium, and there is also the unsolvable problem of the effect of autogenerated gases on the temperature profile.

An obvious technique to lessen the radial temperature gradient in these systems is to preheat and premix the entering purge gas. This has been accomplished by first passing the purge gas through an extension of the furnace tube containing metallic mixing element fins and heated by a second furnace which raises the gas temperature to a value slightly below the experimental temperature. However, due to the high thermal inertia of this system, the apparatus is best adapted to isothermal measurement. A rapid-response furnace was developed by Dickens and Flynn [1] and has been used successfully with the temperature jump method. As with the method above, the direction of gas flow was reversed, and several in-stream nichrome wire preheating grids inserted across the flow. The external furnace supplied by the manufacturer was replaced by a rapid-response furnace inside the tube. This furnace consists of many strands of bare nichrome wire strung between pyrophillite disks and maintains the temperature of the specimen. This system eliminates radial temperature gradients and combines rapid response with good temperature control.

CONCLUSIONS

Very serious radial temperature gradients due to laminar flow may occur in horizontal tube furnaces when they are used in a thermogravimetric apparatus at typical temperatures, pressures and purge gas flow rates. These gradients can cause considerable error in temperature calibration and measurement if factors such as the positions of specimen and temperature sensor and flow rate, composition and pressure of purge gases are not carefully matched during calibration and experiment. In-stream nichrome wire preheating grids have been found to be effective in reducing the temperature gradients.

REFERENCE

1 B. Dickens and J.H. Flynn, Thermogravimetry Applied to Polymer Degradation Kinetics, in C.D. Craver (Ed.), ACS Advances in Chemistry Series No. 203, Polymer Characterization: Spectroscopic, Chromatographic, and Physical Instrumental Methods, Chap. 12, American Chemical Society, Washington, DC, 1983, pp. 209–232.