

Buoyancy Phenomenon in TGA Systems

INTRODUCTION

It is a scientific fact that a specimen heated or cooled in a thermobalance will exhibit an increase or decrease in weight, which is not related to chemical or physical changes in the specimen. The effect is observed with all conventional thermobalances to some degree. A number of earlier explanations have been given by other researchers. 1,2 Modern thermobalances have design features that reduce these effects to a minimum and under normal conditions no corrections are required. However, it is not possible to completely eliminate the buoyancy effects.

What is Buoyancy?

The term buoyancy, which is generally used, is actually a combination of factors:

1. True buoyancy effects
2. Convection currents
3. Gas flow drag effects
4. Gas velocity effects
5. Thermomolecular forces
6. Thermal effects on the balance mechanism

True buoyancy

During an experiment, the gas in the system is heated, primarily by conduction at low temperatures and by radiation at higher temperatures. In the initial stage the gas temperature is in advance of the sample/crucible temperature. The buoyancy of the gas itself decreases and therefore the sample/crucible appears to gain weight. This is, in part, due to the differences in thermal conductivity, density, and heat capacity for the gas and the sample/crucible. This effect becomes less at higher temperatures as radiation becomes the primary route for heat transfer. Thus the largest effect is seen at lower temperatures, usually at the start of the experiments, with only smaller increases being seen at higher temperatures.

Convection Currents

On initial heating in a furnace, the wall of the furnace is hotter than the sample area. The gas at the furnace wall heats up first and starts to rise. Cold gas is forced down the centre of the furnace and this impinges on the crucible/sample giving an increase in weight. This convection effect is minimized in modern systems by careful design and by having a small volume within the furnace.

Gas Flow Drag

If the design of the instrument allows the gas to flow over the hang-down system, then there will be a viscous dragging effect in the direction of the gas flow. The gas flow rate and viscosity of the gas at a specific temperature will affect the level of the viscous drag as will the type of atmosphere used.

Thus, as the temperature increases, the viscosity will decrease and the effect will lessen. The gas flow rate will affect the level of the drag as will the type of atmosphere used. The length of the hangdown used also is critical to the amount of drag observed.

Gas velocity

The velocity of a gas flow increases with temperature. This will affect the apparent weight due to the increased force of the gas impinging on the sample/crucible. This effect will change with the direction of the gas path and also the flow rate.

Thermomolecular forces

The thermomolecular forces among the gas molecules and sample holders can cause small weight change. However, these forces are not that obvious for most of applications, and only evident under extremely high vacuum conditions.

Thermal effects on the balance mechanism

Since the balance is very sensitive to heat, any thermal impact on the balance mechanism will cause a weight change on the measured weight. In Thermo Cahn's TG systems, great care has been taken to reduce the buoyancy effects to a minimum. These include the purge of the balance mechanism by using an inert gas and specially designed flowing pattern to reduce the thermal impact onto the balance mechanism, and the patented upper and lower baffles to reduce the effect of changing gas velocity. However, in special cases, e.g., very small samples or fast gas flows, blank correction experiments may need to be carried out. With the Thermo Cahn analysis software, data for buoyancy run can be stored and subtracted from the actual experiment runs to give true weight changes.