Application Note
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Improved Chemical Reaction Temperature Control with Thermo Scientific HAAKE DynaMax System

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Key Words:

- Reaction Temperature Control
- Dynamic Heat Load Suppression
- Methanol
- Tri-Ethyl-Amine
- Chloro-Tri-Methyl-Silane
- Thermo Scientific HAAKE DynaMax 1700

Abstract

This application note highlights the temperature control performance of a Thermo Scientific HAAKE DynaMax 1700 connected to a reactor vessel during an exothermic reaction and compares its performance to a competitor's unit. The HAAKE DynaMax™ 1700 unit delivers significantly tighter temperature control than the competitor unit.

Introduction

This application note compares the performance of two "sealed" architecture circulator units, the HAAKE DynaMax 1700 unit and a well known competitor unit, when used to control exothermic reactions in a reactor vessel. The competitor unit was already in use at the testing scientist's laboratory at a large, well known pharmaceutical company on the US East Coast. The HAAKE DynaMax 1700 unit was shipped in for this comparison. Based on the results presented here, the scientist has since purchased a HAAKE DynaMax 1700 unit.

Although this application note is based on controlled experiments, these experiments were nevertheless created to reflect real world conditions testing scientists encounter during day-to-day process work in the laboratory. As such, they comprise a fast mixing experiment and a slow mixing experiment.

Experiments

In both experiments, a three-liter jacketed glass reactor vessel was connected to a HAAKE DynaMax 1700 unit and separately also to the competitor unit. Both units were controlled off of the batch temperature. The coolant used was Silicone Oil. 500 ml of Methanol (MeOH) and 500 ml of Tri-Ethyl-Amine (TEA) were mixed in the reactor vessel. This batch was agitated and its temperature controlled to 20°C. Upon reaching steady state temperature, 100 ml of Chloro-Tri-Methyl-Silane (TMSCl) was added to the batch, resulting in an exothermic reaction. The resulting rapid rise in batch temperature was controlled by the circulator units.

In the fast mixing experiment, TMSCl was added at about 20 ml/second, so the exothermic reaction lasted roughly 5 seconds. In the slow mixing experiment, TMSCl was added at about 16.5 ml/minute, so the exothermic reaction lasted roughly 6 minutes or 360 seconds.

The DynaMax Advantage

The temperature difference ($\Delta T = T_{Batch} - T_{Coolant}$) between batch temperature (T_{Batch}) and coolant supply temperature ($T_{Coolant}$) enables heat to flow from/into the batch into/from the coolant. The faster a sufficiently large temperature difference can be established, the more quickly heat can be removed from or added to the batch, the more rapidly an exothermic or endothermic reaction be counteracted upon.

During cooling, this temperature difference increases due to a temporary increase in batch temperature above set point, but it is also enhanced by a temporary decrease in coolant supply temperature. During heating, this temperature increases due to temporary decrease in batch temperature below the set point, but is also enhanced by a temporary increase in coolant supply temperature.

Thus, the coolant supply temperature difference ($\Delta T_{Coolant}$) between the HAAKE DynaMax 1700 unit and the competitor unit, and the speed of its establishment, will manifest itself in a batch temperature difference (ΔT_{Batch}) between a reactor vessel cooled by the HAAKE DynaMax 1700 unit and a reactor vessel cooled by the competitor unit.

Results

Coolant Supply Temperature Advantage

The coolant supply temperature of the HAAKE DynaMax 1700 unit and the competitor unit are shown as open green circles and open red triangles, respectively, in Figure 1 for the fast mixing experiment, and in Figure 2 for the slow mixing experiment. Note that the batch temperature of the HAAKE DynaMax 1700 unit and the competitor unit are shown as closed green circles and closed red triangles, respectively, for reference.

During the initial phase of the experiment, where the exothermic reaction is triggered at around 50 seconds for both fast mixing and slow mixing, fast cooling with a reduced coolant supply temperature is needed to maintain the batch temperature as well as possible.

As can be seen, the HAAKE DynaMax 1700 unit can drop its coolant supply temperature to a lower value in less time than the competitor unit. The result is a larger temperature difference ΔT in less time for the HAAKE DynaMax 1700 unit than the competitor unit. As a result, the HAAKE DynaMax 1700 unit can remove more heat more quickly than the competitor unit. In fact, the HAAKE DynaMax 1700 unit supplies coolant at least 5°C cooler than the competitor unit at most times.

Furthermore, in the final phase of the experiment, where the exothermic reaction has subsided at around 200 seconds for fast mixing and 400 seconds for slow mixing, fast heating with a warm coolant supply temperature is needed to reverse the earlier cooling, again so as to maintain the batch temperature as well as possible.

As shown, the HAAKE DynaMax 1700 unit can raise its coolant supply temperature to a higher value in less time than the competitor unit. The result is a smaller temperature difference ΔT in less time for the HAAKE DynaMax 1700 unit than the competitor unit. As a result, the HAAKE DynaMax unit can stop the removal of heat more quickly than the competitor unit, or even introduce heat to stabilize the batch temperature set point.

Batch Temperature Advantage

The batch temperature of the HAAKE DynaMax 1700 unit and the competitor unit are shown as closed green circles and closed red triangles, respectively, in Figure 3 for the fast mixing experiment and in Figure 4 for the slow mixing experiment, see next page. Note that the coolant supply temperature of the HAAKE DynaMax 1700 unit and the competitor unit are shown as open green circles and open red triangles, respectively, for reference.

From the figures it can be seen that the coolant supply temperature advantage is turned directly into a batch temperature advantage in that after the exothermic reaction, the HAAKE DynaMax 1700 unit returns to the set point batch temperature about 100 seconds sooner than the competitor unit for both fast and slow mixing. Furthermore, it is capable of stabilizing the set point batch temperature, while the competitor unit never really achieves this.

The HAAKE DynaMax 1700 unit does this while providing less overshoot (2°C vs. 4°C) above 20°C during the exothermic reaction and less undershoot (2°C . 4°C) below 20°C after the exothermic reaction, when compared to the competitor unit, for both fast and slow mixing.

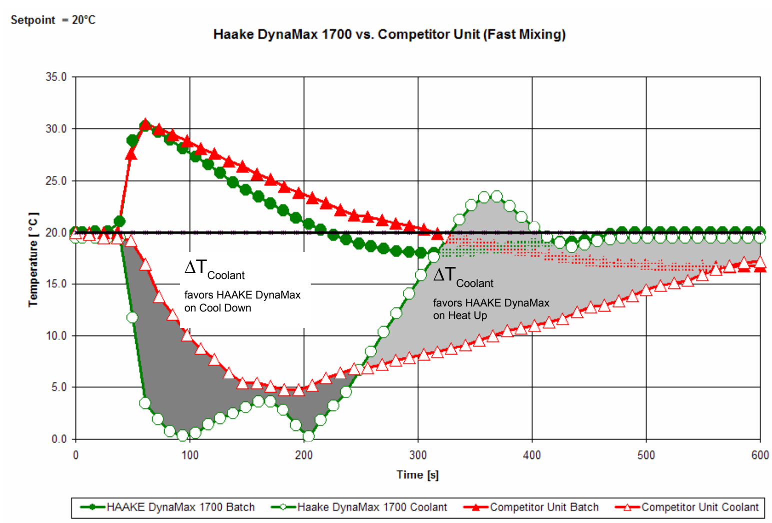

Figure 1: Coolant Supply Temperature Advantage – Fast Mixing

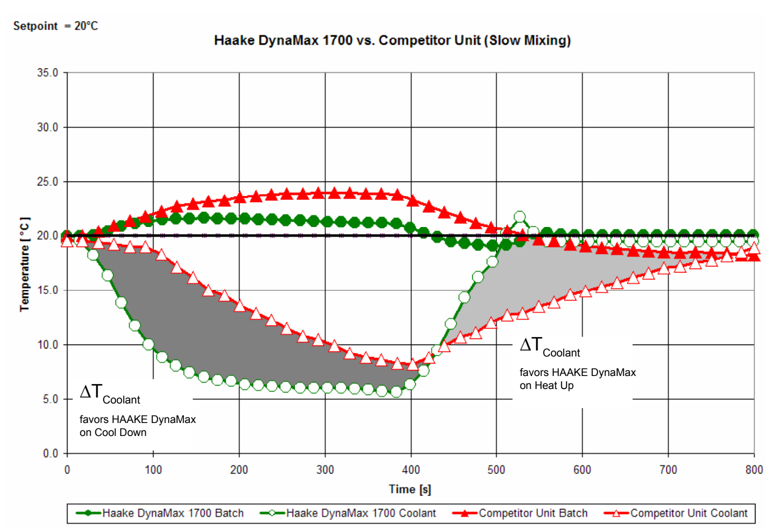

Figure 2: Coolant Supply Temperature Advantage – Slow Mixing

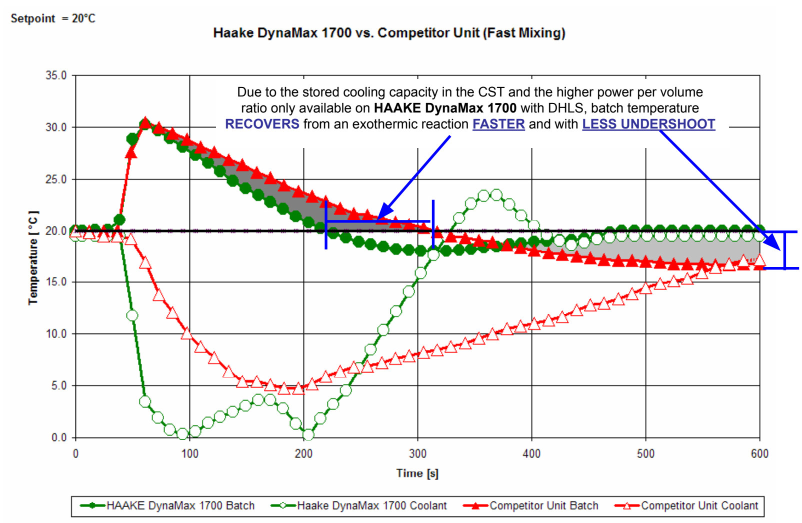

Figure 3: Batch Temperature Advantage – Fast Mixing

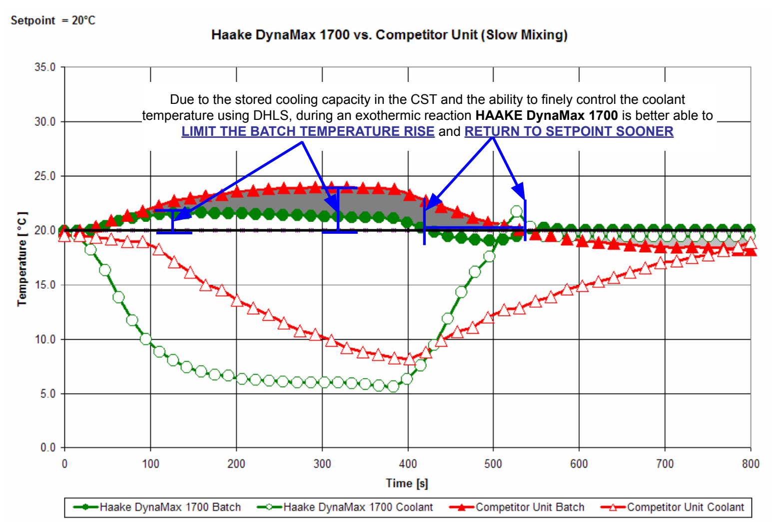

Figure 4: Batch Temperature Advantage – Slow Mixing

Technology

The innovative technology that makes the performance of the HAAKE DynaMax 1700 possible is called Dynamic Heat Load Suppression (DHLS). The major components are the Cold Storage Tank (CST) mixing valve, heating element and pump.

To achieve the Coolant Supply Temperature Advantage as seen above, the HAAKE DynaMax 1700 unit has stored cooling capacity in its CST, where the coolant in these experiments was maintained at -20°C. When an exothermic reaction is sensed, the content of the CST is introduced into the recirculating flow via the mixing valve, thus providing a “cold shot” to the coolant. This effect, which depends on the application, can be the equivalent of 8, 12, 16 or more kW of effective cooling capacity during the first 30 seconds of mixing.

When the exothermic reaction subsides, the HAAKE DynaMax 1700 unit continues to have a coolant supply temperature advantage in returning (heating) the supply temperature back to that required to hold set point. This is because of a very small fluid volume in general and a very small volume flow-through resistive heater in particular, and because the mixing valve is now closed and thus the refrigeration system is bypassed completely. This allows for a very quick increase in the coolant supply temperature.

DHLS, CST, mixing valve and flow-through heater technologies are only found in the HAAKE DynaMax system. Overall, they permit minimization of both amount and duration of set point overshoot and undershoot, resulting in a faster return to set point. Simply put, the CST, mixing valve and heater work together to mix cold and warm fluid together much like the hot and cold faucets on a sink allow rapid adjustment of the output temperature — warmer or colder as needed.

Conclusion

From the figures on the left, it is evident that rapidly acting DHLS technology, which stores cooling capacity using the CST and uses it in combination with a small volume flow-through heater via the mixing valve, minimizes the batch temperature rise during energy release from an exothermic reaction, as well as reduces the time required to regain set point temperature after the exothermic reaction has subsided.

Whether it is during cooling or heating, with comparable capacities or possibly even with less capacity, the HAAKE DynaMax 1700 unit is able to out-perform and out-control a comparable competitor unit. Generally, reaching reaction temperature more quickly, as well as maintaining it more stably, increases the yield of the desired product and minimizes the generation of undesired by-products.

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