Julabo

CASE STUDY

30L Jacketed-Reactor controlled by Julabo *Presto LH 50* System

Prepared by staff engineers at Julabo USA, Inc.

The Objective:
Constant temperature control of a 30 Liter Jacketed Glass Reactor utilized in Chemical, Petrochemical, and Pharmaceutical applications.

The Motivation:
Despite its advantages in chemical resistance and visual clarity, large laboratory glass reactors require high heating and cooling power for effective temperature control. This, of course, is due to massive heat losses from the lack of insulation. The Julabo Presto series circulators is designed for speed, in other words high heat-up and cool-down rates. Further on, we will also demonstrate the temperature stabilization capability of the *Presto LH 50* from exothermic "disturbances", a common requirement in laboratory applications.

The Solution:
Testing was performed with a Julabo *Presto LH 50* (#9 410 150) and the following apparatus:

- QVF Eng. 30 Liter jacketed glass reactor & stirrer.
- Triple insulated metal tubing, 1m (#8 930 210).
- Thermal HL45 heat transfer fluid (#8 940 123).
- 2kW immersible coil heater.
- External Pt100 temperature sensor (#8 981 010).
- Julabo EZTemp™ DAQ software (*free download*).

Julabo LH 50 High Dynamic Temperature Control System.

Test Goal:
The following tests were performed and documented:

#1. Heat-up (+20...+120°C) and Cool-down (+120...-30°C) temperature profiles via external, reactor control.

#2. Lowest Achievable Temperature of the system. Observation of frost formation on the reactor's outer surface.

#3. Time taken to stabilize a 250W exothermic reaction at near ambient temperature, i.e. +20°C.

#4. Time taken to stabilize a 250W exotherm at high temperatures, i.e. +100°C.

Test Summary & Results:
All tests were performed with a stirrer set to approx. 200 rpm. Furthermore, the glass reactor is physically limited to the following:

max. ΔT	< 2°C/min
max. Pressure	7 psi

Figure 1 shows the results of test #1, starting and ending at +20°C. The heat-up curve from +20 to +120°C took 3 hours, whereas the cool-down from +120 to -30°C was accomplished in 5 hours. The average rate of temperature change, the speed, was:

$\Delta T = 0.5$ °C/min.

This rate can be further improved by utilizing a reactor with more rugged physical properties (i.e. max. ΔT greater than 2°C/min). In this test, a temperature "Band" constraint of 30K was implemented in the controller to reduce any risk of reactor damage.

The lowest temperature achieved by the 30L reactor was -34°C. However, as seen below, the reactor was covered with a substantial layer of

Overview of apparatus set-up. Notice the white frost surrounding reactor (far right) in test #2 .

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frost, illustrating the tremendous amount of heat loss during the test. Figure 2 shows the *T* (°C) versus *t* (min) curve during a 250W simulated exotherm in the reactor via a heater coil. The "exothermic reaction" increased the reactor to a 25.7°C peak, and the recovery time was approx. 120 minutes to return to +20°C setpoint. The result of test #3 is 2 hours.

Figure 3 displays the results of a 250W exotherm at +100°C setpoint. The reactor peaked at 104.3°C during the "exothermic reaction", but recovered in under 2 hours.

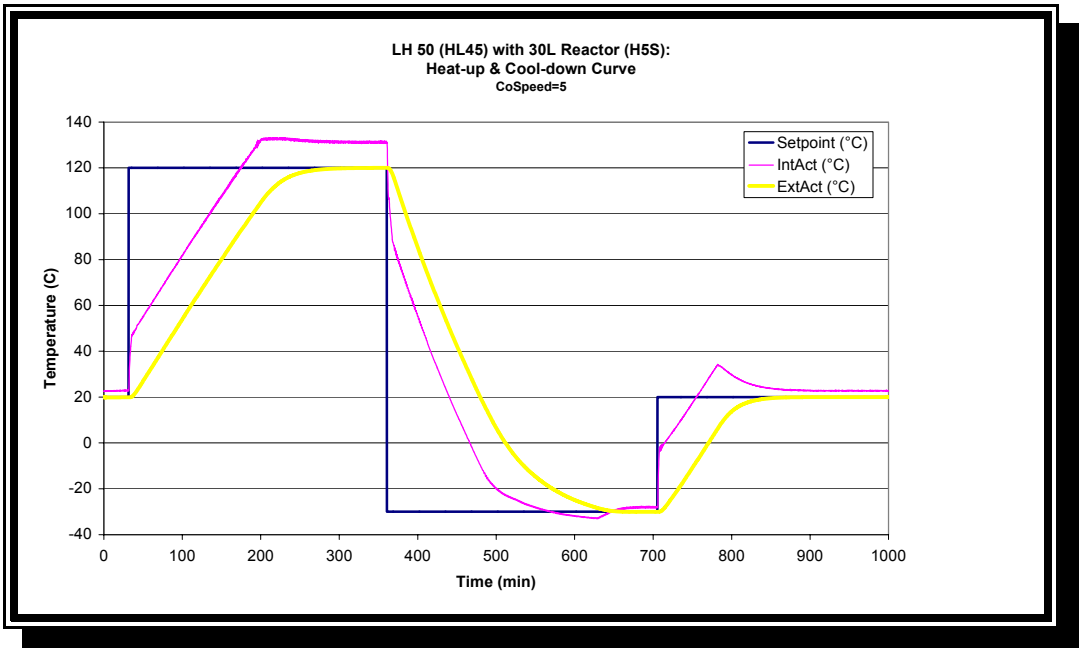

Figure 1: Heat-up and Cool-down Curve for the Presto LH 50. This test was limited to, | IntAct - ExtAct | < 30°C. "IntAct" refers to the temperature of the circulating medium (Thermal HL45 Silicone Oil), whereas "ExtAct" the reactor temperature.

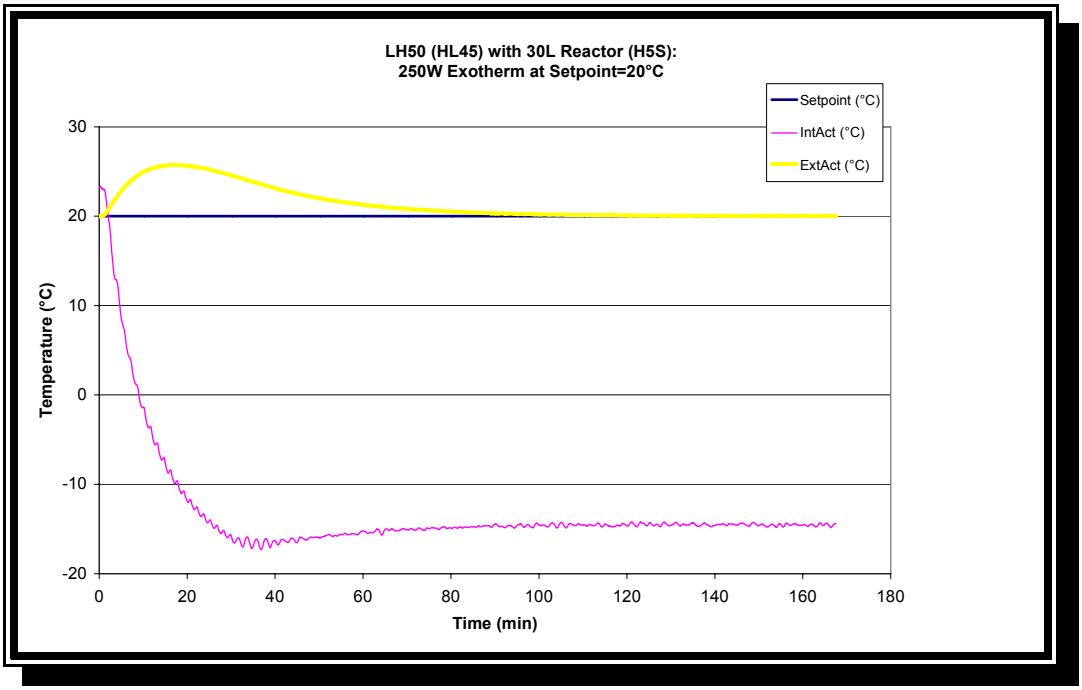

Figure 2: Response of LH 50's control system counteracting a 250W exotherm at +20°C reactor temperature. Reactor returns to setpoint in 2 hours. | IntAct - ExtAct | < 50°C.

Test Conclusions:

- Lowest achievable temperature in the reactor with *Presto LH 50* and a 30L Uninsulated Glass Reactor: -34°C.
- Highest temperature deviations from a 250W exotherm are 5.7°C and 4.3°C for setpoints of +20°C and +100°C, respectively.
- Recovery time from a 250W exotherm are approx. 2 hours for both setpoints of +20°C and +100°C.

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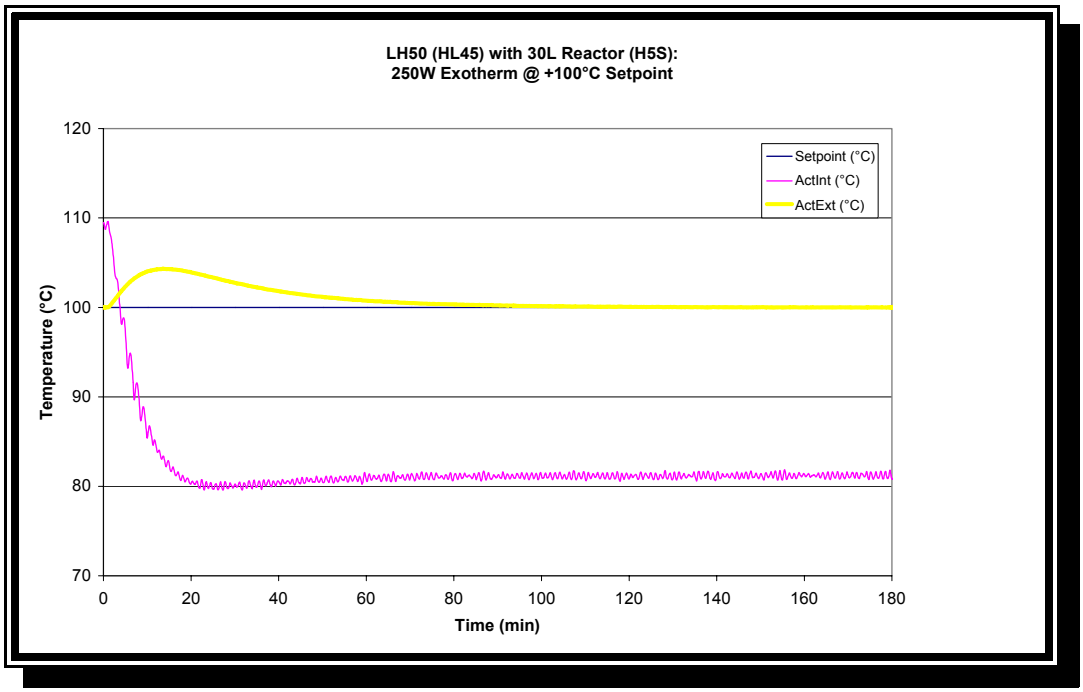

Figure 3: Response of LH 50's control system counteracting a 250W exotherm at +100°C reactor temperature. Reactor returns to setpoint in under 2 hours. | IntAct - ExtAct | < 50°C.