

Ace Glass 30L Jacketed-Reactor controlled by Julabo *FP52-SP with HST*

Prepared by staff engineers at Julabo USA, Inc.

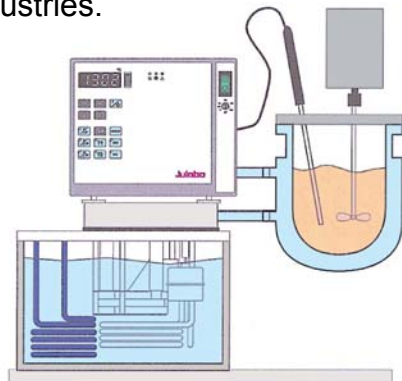
capacities from its temperature controller.

with an *HST Booster Heater* (#8810010) as a solution for



The Objective:

Achieving & maintaining a constant temperature in a large 30 Liter Jacketed-Glass Reactor vital in the Chemical, Petrochemical & Pharmaceutical Industries.



Conceptual Drawing of a Glass Reactor Application controlled by a Julabo Circulator.



The Motivation:

Distillations, Chromatography, and Chemical Reactions in general are accomplished at very specific temperatures in reactor vessels. Glass reactors are popular for such applications due to their superior resistance to chemical corrosion. It is also common to find these glass reactors used without any insulation, therefore, demanding higher heating and cooling



Julabo FP50-HP Benchtop Circulator maintaining temperature in a stirred, Jacketed-Reactor



The Solution:

Using Julabo Heating & Refrigerated Circulator *FP52-SP* (#9320752)



Julabo FP52-SP Heating & Refrigerated Circulator



6kW HST Booster Heater Accessory

temperature control, and Julabo *EZTemp™* software (*free*) for Data Acquisition

of both the internal (*FP52*) & external (Reactor) temperature. The HST heating element assembly replaces the bath lid when in use with the *FP52-SP* Circulator.

Test Goal:

The Reactor was tested for two temperature ranges, one above the ambient temperature of +20°C, and the other below. For practical purposes, the selected reactor medium were as follows (Including their respective test temperature ranges):

- i) Ethanol (T=+20...-10°C)
- ii) Water (T=+20...+80°C)

The key to this test is to optimize the PID control parameters, through several iterations, to obtain the most stable temperature control.

Test Summary:

Tests conducted at Julabo USA, Inc. with an *FP52-SP with HST*

Ace Glass 30L Jacketed-Reactor controlled by Julabo *FP52-SP* with *HST*

were able to meet the above requirements within a reasonable time frame:

- i) <250 mins. for +20...-10°C
- ii) <300 mins. for +20...+80°C

Internal bath temperatures were purposefully limited to the following ranges due to inherent limitations of the tested Glass Reactor and the reactor medium:

- i) with Ethanol,
 $I_{\min} = -20^{\circ}\text{C}$ & $I_{\max} = +40^{\circ}\text{C}$.

- ii) with Water,
 $I_{\min} = -20^{\circ}\text{C}$ & $I_{\max} = +100^{\circ}\text{C}$.

In both tests, the *FP52-SP with HST* was set to “External Control”. The External Sensor was a 24" x 1/4"OD Stainless Steel Pt100 RTD immersed at least 5" into the reactor medium.

A total of 45 Liters of Julabo Thermal H10S Silicon Oil (#8940114) was utilized as the circulating medium or bath fluid (~15 Liters filled the Glass Jacket, whereas ~30 Liters filled the Julabo *FP52*'s bath).

Additionally, Julabo Triple Insulated Metal Tubings were used to transport & insulate the circulating medium.

Figure 1 illustrates the test apparatus in a controlled lab



Figure 1: Test Set-up Consisting Mainly of, i) Julabo *FP52-SP+HST* in the foreground, ii) Ace Glass 30L Reactor in the background on left, and iii) Julabo Messbox Data Acq System in the background on right.

environment. The test data was obtained via the multi-channel Julabo Data Acquisition System (Messbox™) used jointly with *Watch&Control 2000* software on a lab PC.

Figure 2 shows the 30L Reactor with a close-up of the Reactor on the inside. The Reactor is supported by a metal structure provided by the manufacturers, and features PTFE components besides glass.

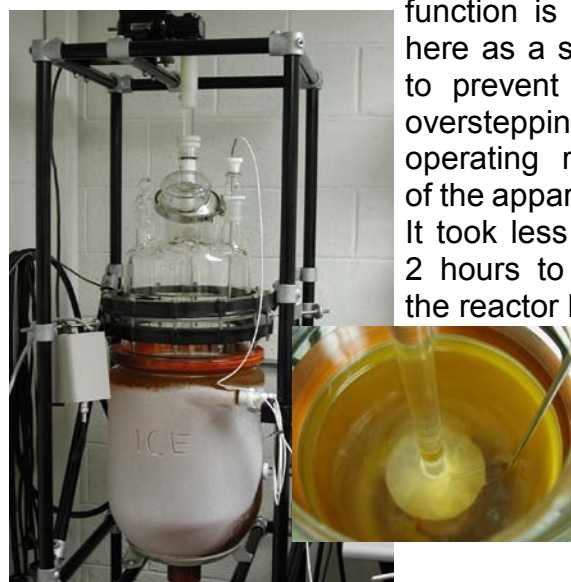


Figure 2: The 30L Jacketed-Glass Reactor with a stirrer and an external Pt100 sensor. (Inset, right) Inside view of the Reactor in operation.

Test Results & Conclusion:

Due to the minimum and maximum temperature limit settings on the Julabo instrument, the rate of temperature heat-up (to +80°C) and cool-down (to -10°C) were not optimized. Therefore, for future reference, by changing the apparatus (Glass Reactor and circulating medium) to those capable of wider temperature ranges, the rate of temperature change in the reactor could increase.

Figure 3 shows the test results for cooling Ethanol from +20...-10°C in about 4 hours. Notice that internal bath temperature of the *FP52*, the circulating medium, never exceeded the preset limit. This internal limit function is used here as a safety to prevent from overstepping the operating range of the apparatus. It took less than 2 hours to heat the reactor back

to +20°C.

Ace Glass 30L Jacketed-Reactor controlled by Julabo FP52-SP with HST

Figure 4 displays the test results for heating Water from +20...+80°C in about 5 hours. It is worthwhile to note here that since Water has a much higher heating capacity than a heat transfer fluid, like Silicon Oil (almost 3 times less heating capacity), this heat-up time is closer to a worst-case scenario.

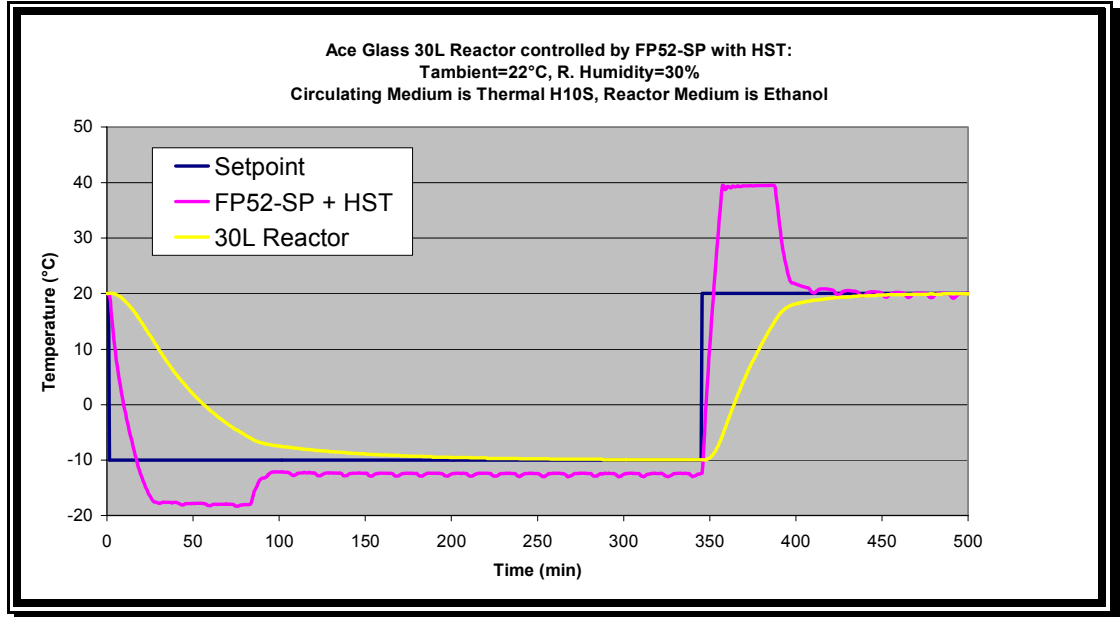


Figure 3: Temperature (°C) vs. Time (minutes) Plot of the Test Results when Using Ethanol in the Reactor.

The results found in Figures 3 & 4 are the best control schemes obtained from several iterations of PID parameters, for a large uninsulated glass reactor. Temperature control may be further optimized via a simulation program of the control model.

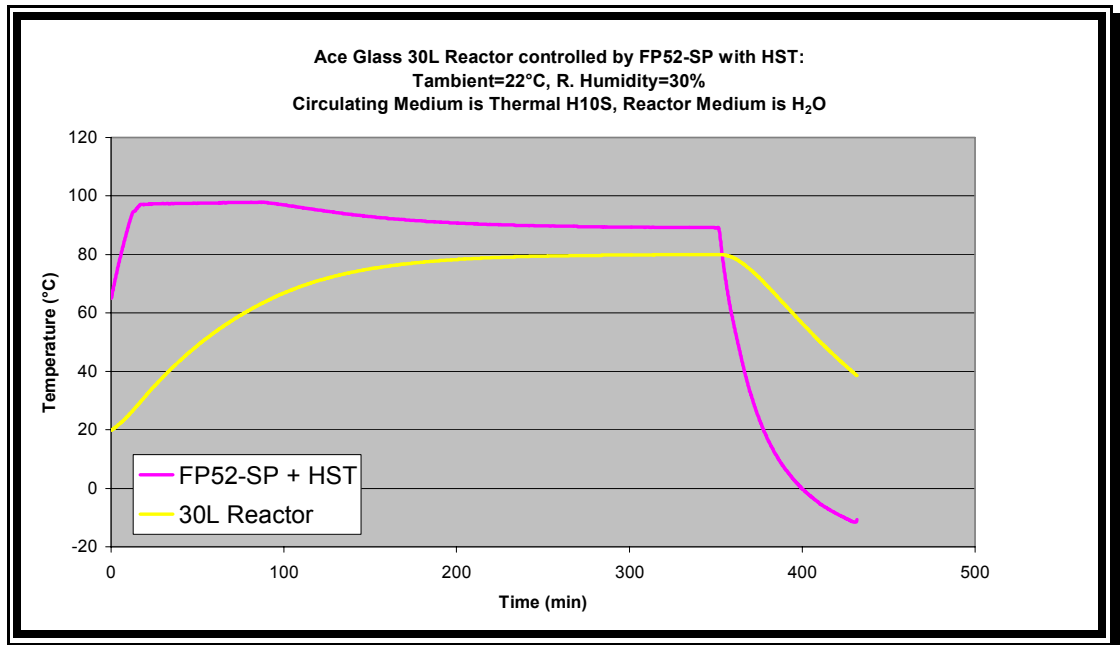


Figure 4: Temperature (°C) vs. Time (minutes) Plot of the Test Results Using Water in the Reactor.