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SUBJECT: Test of Stainless Steel Freeze Jacket to be Used on 1/2-inch
High Pressure and High Temperature Process Lines

TO: I. Spiewak

FROM: B. D. Draper

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INTRODUCTION

Prior to the tests outlined in this report, investigations had been made to determine the effectiveness of different freeze jacket designs on the rate of freezing. Some of the test conditions were as follows:

1. Welded stainless steel tubing and soldered copper tubing on 1/2-in. pipe, in air, with process water at a temperature of 160°F and at pressures of 70,500 and 2000 psi.
2. Wrapped copper tubing on a 3 1/2-in. open end, stainless steel pipe submerged in water at room temperature.
3. Various types of split and integral jackets operating both in air and under water with the process fluid at tap water conditions.

However, it was desired to operate a freeze jacket in air with the process fluid closely following the conditions found in the high pressure fuel circulating loop of a homogeneous reactor, namely, 2000 psi and 300°C. To achieve this, a test loop was built and used in conjunction with the existing refrigeration equipment. Approximately 80 runs were made under varying pressures and temperatures to check the effect of these variables on the maximum leak rate that could be frozen off.

APPARATUS

The refrigeration portion of the test loop is essentially composed of two separate systems. The primary system, using F-12 as a coolant, consists of a 5-HP two stage, low temperature compressor; a water-cooled condenser; a liquid sub-cooler; and an F-12 drier, strainer and temperature-controlled expansion valve. The F-12 is compressed and condensed and then circulated through the sub-cooler and thence through the expansion valve and coils of the F-11 storage tank. The pressure of the compressor suction and discharge lines control the operation of the compressor. The temperature of the primary F-12 refrigerant leaving the F-11 storage tank controls the movement of the expansion valve.

The cold F-11 in the secondary system is circulated at approximately -40°F by a 3/4-h.p. centrifugal pump through a manifold header and back to the cold storage

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tank. A by-pass stream is circulated through the freeze jacket (Fig. 1).

The process loop provides a circulating liquid stream at reactor operating conditions of temperature and pressure. A 3 kw electric immersion heater was used to heat the water and a sprague pump maintained the desired pressure. A hydraulic accumulator was used to smooth out the pulsations of the Sprague pump and a rupture disc assembly was installed as a safety precaution.

At the top of the loop, a 1/2-in. line removed a side stream from the circulating fluid and by means of three throttling valves arranged in series and a rotometer, appropriate leak rates were obtained. Coarse throttling was maintained with two 1/4-in. P and C Co., type 1040A, bar stock valves rated to 10,000 pounds at 150 degrees F. Final adjustment in leak rate was made with a 1/4-in. Republic, stainless steel, micrometer adjustment needle valve, type 0151. The valve was rated to 5000 pounds at 350 degrees F. The freeze jacket was installed on the abovementioned bleed line in a vertical position with the bottom of the jacket located a distance of 12-in. from the circulating loop (Fig. 2). This simulates a condition found on the HRT where the supply lines (167 and 267) to the chemical plant tap off of the main high pressure fuel and blanket circulating loops.

Thermocouples were inserted to the mid-point of the process line, both upstream and downstream from the freeze jacket. Refrigerant temperatures were measured at various points including the inlet and outlet temperature at the freeze jacket. A rotometer installed downstream from the freeze jacket permitted refrigerant flow to be measured.

The freeze jacket was made of 5/16-in., type 347 stainless steel tubing wrapped around and welded to the 1/2-in. stainless steel process line. The overall length of the jacket was 7 1/2-in. Aluminum foil wrapped around the freeze jacket provided insulation.

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RESULTS

In order to determine the effect of pressure and temperature on the freezing ability of the jacket, test runs were made to determine the maximum leak rate that could be frozen under the following conditions:

1. Constant pressure - 2000 psi
Varying temperature - 300, 200, 100, and 24°C
2. Constant temperature - 300°C
Varying pressure - 2000, 1750, 1600, 1500, and 1400 psi
3. Constant temperature - 24°C
Varying pressure - 2000, 1000, and 250 psi
4. Constant temperature - 100°C
Varying pressure - 2000, 1500, 1000, and 250 psi.

Results of test runs made under the first condition of constant pressure and variable temperatures are as follows:

Run I			
At Constant 2000 psi Pressure			
<u>Temperature</u>	<u>Max. Leak Frozen</u>	<u>Elapsed Time</u>	<u>F-11 Temp. at Jacket Inlet*</u>
300°C	11 cc/min	27.2 min.	-39°C
200°C	15 "	15.2 "	-40°C
100°C	22 "	7.0 "	-44°C
24°C	78 "	2.8 "	-41°C

*F-11 temperature is at start of run.

Indications were, at this point, in the experiment that temperature had a rather pronounced effect on the freeze rate. A decrease in temperature from 300°C to 100°C resulted in a 100% increase in the leak rate that could be frozen off. A reduction to room temperature (24°C) increased the maximum freezeable leak rate by a factor of 7.

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Tabulated below are the results of the second series of runs made at constant temperature and variable pressure.

Run II
At Constant 300°C Temperature

<u>Pressure</u>	<u>Max. Leak Frozen</u>	<u>Elapsed Time</u>	<u>F-11 Temp. at Jacket Inlet*</u>
2000 psi	11 cc/min	27.2 min.	-39°C
1750	11	26.6	-39°C
1600	11	25.8	-38°C
1400	11	18.9	-42°C

*F-11 temperature is at start of run.

Although only a 600 pound reduction in pressure could be made as a result of approaching the saturation pressure at 300°C, the maximum leak rate that could be frozen off remained unchanged over this pressure span. In order to look at the effect of a greater pressure variation, it was decided to make a series of runs at room temperature and variable pressure.

The results are shown below.

Run III
At Constant 24°C Temperature

<u>Pressure</u>	<u>Max. Leak Frozen</u>	<u>Elapsed Time</u>	<u>F-11 Temp. at Jacket Inlet*</u>
2000 psi	78 cc/min	2.8 min.	-41°C
1000	106	2.3	-43°C
250	**135	2.2	-43°C

*F-11 temperature at start of run.

**This was about the highest leak rate that could be maintained at a steady state. The maximum rate that could be frozen may be somewhat higher than this value.

Although the maximum leak rate that can be frozen off at 250 psi may be somewhat greater, as explained above, the rate of 135 cc/min. obtained is about twice the rate at 2000 psi. This would indicate that pressure has an effect on the freezing rate, even though Run II indicated otherwise

In order to resolve the question, Run IV was made at constant temperature of 100°C and with pressures ranging from 2000 psi to 250 psi. The following results were obtained.

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Run IV
At Constant 100°C Temperature

<u>Pressure</u>	<u>Max. Leak Frozen</u>	<u>Elapsed Time</u>	<u>F-11 Temp. at Jacket Inlet*</u>
2000 psi	22 cc/min	7.0 min.	-44°C
1500	23	16.8	-43°C
1000	24.5	11.5	-43°C
250	23	4.8	-44°C

*F-11 temperature at start of run.

It can be seen from Run IV that the maximum leak rate which could be frozen off remained almost constant for all the pressures checked. This substantiates the results of Run II. However, at 2000 psi, the maximum flow of both Runs II and IV were appreciably smaller than the flow of Run III at 2000 psi. This larger flow of Run III may have caused the maximum leak rate which could be frozen off to increase upon a reduction in pressure.

CONCLUSIONS

1. At reactor operating conditions of 2000 psi and 300°C, only a small leak rate, 11 cc/min, could be frozen off. A reduction in temperature from these operating conditions resulted in an increase in the maximum freezeable leak rate. A reduction in pressure from operating conditions resulted in no appreciable increase in the maximum leak rate that could be frozen.

2. For an average F-11 temperature increase of 2 degrees centigrade through the freeze jacket and a flow of 1.7 gpm, the heat removal capacity of the jacket is approx. 16 Btu/min or 1/12 tons of refrigeration (See Appendix). From a survey of numerous runs, the heat given up by the process fluid varies between 4 - 6 Btu/min. Hence, the efficiency of the freeze jacket in air is approximately 30%.

3. At the beginning of the freezing operation, it is recommended that the temperature of the secondary refrigerant entering the freeze jacket be at least -40°C. It has been observed that with a refrigerant temperature 2 degrees warmer, i.e., -38°C, leak rates frozen off at -40°C could not be frozen at the warmer temperature. A 2 or 3 degree variation at this low level thus appears to be important.

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4. The following table may be of use to determine whether a leak can be frozen off at the reactor. For example, if at 300°C and 2000 psi the freezing operation has been progressing for an hour and an ice plug has not been formed, it is safe to assume the leak is too great to be frozen since the time to freeze the maximum leak at these conditions was less than 30 minutes on the test mockup. Other conditions may be judged accordingly from use of the table.

TABLE I

<u>Temperature</u>	<u>Pressure Span</u>	<u>Time to Freeze Maximum Leak</u>
300°C	2000-250 psi	27.2 min.
200°C	2000 psi	15.2
100°C	2000-250 psi	7.0
24°C	2000-250 psi	2.8

5. It is recommended that the freeze jacket be made as long as practical. Tests on a 12-in. long jacket with 18 coils indicated that the maximum leak rate that could be frozen off was twice the value of that obtained with the 7 1/2-in. long jacket and 11 coils at 2000 psi and 300°C. It should be noted however, that the end of the longer jacket was kept at a distance of 12-in. from the circulating loop for purposes of comparison with the 7 1/2-in. long jacket. If lengthening a jacket on the reactor places the end less than 12-in. from the circulating process line, the increase in freezing ability may not be as pronounced.

The following table shows a comparison between the 7 1/2-in. and 12-in. freeze jackets for the few conditions of pressure and temperature checked.

TABLE II

<u>Jacket Length, Inches</u>	<u>Process Line Pressure, psi</u>	<u>Process Line Temperature, °C</u>	<u>Max. Leak Frozen-cc/min</u>
7 1/2	2000	300	11
12	2000	300	22
7 1/2	2000	200	15
12	2000	200	36

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APPENDIXCalculation of Heat Removal Capacity
of Freeze Jacket

F-11 flow on rotometer - 37.5%

Full scale flow for F-11 - 4.48 gpm

$$.375 \times 4.48 \text{ gpm} = 1.7 \text{ gpm}$$

$$1.7 \frac{\text{Gal.}}{\text{Min.}} \div 7.48 \frac{\text{Gal.}}{\text{Ft}^3} = .225 \text{ ft}^3/\text{min.}$$

At -40°F specific volume of F-11 = .00988 ft³/lb

$$\text{Density} = 101.2 \text{ lb/ft}^3$$

$$\text{Then } .225 \frac{\text{Ft}^3}{\text{Min.}} \times 101.2 \frac{\text{lb.}}{\text{Ft}^3} = 22.8 \text{ lb/min.}$$

For 2 degree temperature rise of F-11 through jacket at -40°F, the heat gained equals .70 Btu/lb.

$$\therefore 22.8 \frac{\text{lb.}}{\text{Min.}} \times .70 \frac{\text{Btu}}{\text{lb.}} = 16 \text{ Btu/min.}$$

16 Btu/min = 1/12 tons of refrigeration.

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FIGURE 1

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FIGURE 2