

# Autocascade refrigeration system: Experimental results in achieving ultra low temperature

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## SUMMARY

In this paper the experimental results of an autocascade refrigeration system for achieving ultra low temperature are presented. The plant is used to preserve tissue and cells. When the air temperature is equal to  $-150^{\circ}\text{C}$  in  $0.25\text{ m}^3$  space, the required refrigeration power is about 250 W. The influence of the most meaningful variables is discussed with regard to the design of the plant. The experimental results show an acceptable time to reach the steady state in dependence of the finality of the plant. The working substance is a non-azeotropic mixture consisting of hydrofluorocarbon (HFC) refrigerants in addition to argon and methane. Copyright © 2009 John Wiley & Sons, Ltd.

KEY WORDS: autocascade refrigeration; cryogenic temperature; two-stage temperature

## 1. INTRODUCTION

Ultra low temperature is usually needed for long-term preservation of tissue, cells or genes, a semiconductor fabricating process, etc. Particularly, in case of biological materials such as cells, if they are kept at temperature of  $-130^{\circ}\text{C}$  or less than that corresponding to recrystallization temperature of ice, water contained therein is not crystallized but is in an amorphous state. Thus, since it is not likely that a cell membrane is destructed, the term of preservation therefore can be greatly prolonged over 10 years. In order to

achieve ultra low temperature of about  $-150^{\circ}\text{C}$  two ways are possible: employing a multi-stage cascade refrigeration cycle or using the liquid nitrogen having liquefaction temperature of  $-196^{\circ}\text{C}$ . As the liquid nitrogen is used up only once, it is necessary to refill it for another use, causing both an increase in cost increasing and reducing practicality. Considering these problems a cryogenic refrigerator has been proposed. It employs a two-stage cascade mixed refrigerant refrigeration circuit for achieving lower temperature in a low-temperature side refrigeration circuit by using a high-temperature side refrigeration

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circuit [1–4]. In the last years the autocascade refrigeration systems using non-CFC refrigerant have been analysed [5]. In this paper an innovative proposed plant working with a non-HCFC component mixture is presented. Moreover, the plant illustrated in this paper differs from commercially available systems with reference to the bigger useful refrigerated space.

## 2. EXPERIMENTAL APPARATUS

As shown in Figure 1, there are two separate high- and low-temperature side refrigeration circuit, which in turn are connected with each other through a cascade condenser [6].

The cascade condenser serves both as an evaporator for the high-temperature side refrigeration circuit and as a condenser for the low-temperature refrigeration circuit. The high-temperature refrigeration circuit is used to achieve a lower temperature in the low-temperature side refrigeration circuit. Pure refrigerant is used in the high-temperature stage while a non-azeotropic

mixture works in the low-temperature stage. The mixed refrigerant stream, as a result of being compressed and cooled, is partially condensed, and the two phases are separated in the first phase separator. The liquid and overhead vapor flow as separate streams to the first exchanger (HX1), where heat is transferred to the returning refrigerant stream. The vapor is thereby condensed and flows from this heat exchanger to the second phase separator, where the phases are again separated. The liquid phase leaving the first phase separator is reduced in pressure before being returned through the first exchanger to the warm end. The overhead vapors and liquid bottoms from the second phase separator are sent as separate streams to the second exchanger (HX2) for further cooling. The liquid leaving the second phase separator is again reduced in pressure before being returned to the second exchanger as a refrigerant stream. The vapor overhead from the second phase separator is cooled and totally condensed in the second and third exchangers and is then reduced in pressure to supply the final level of refrigeration in flowing back through the

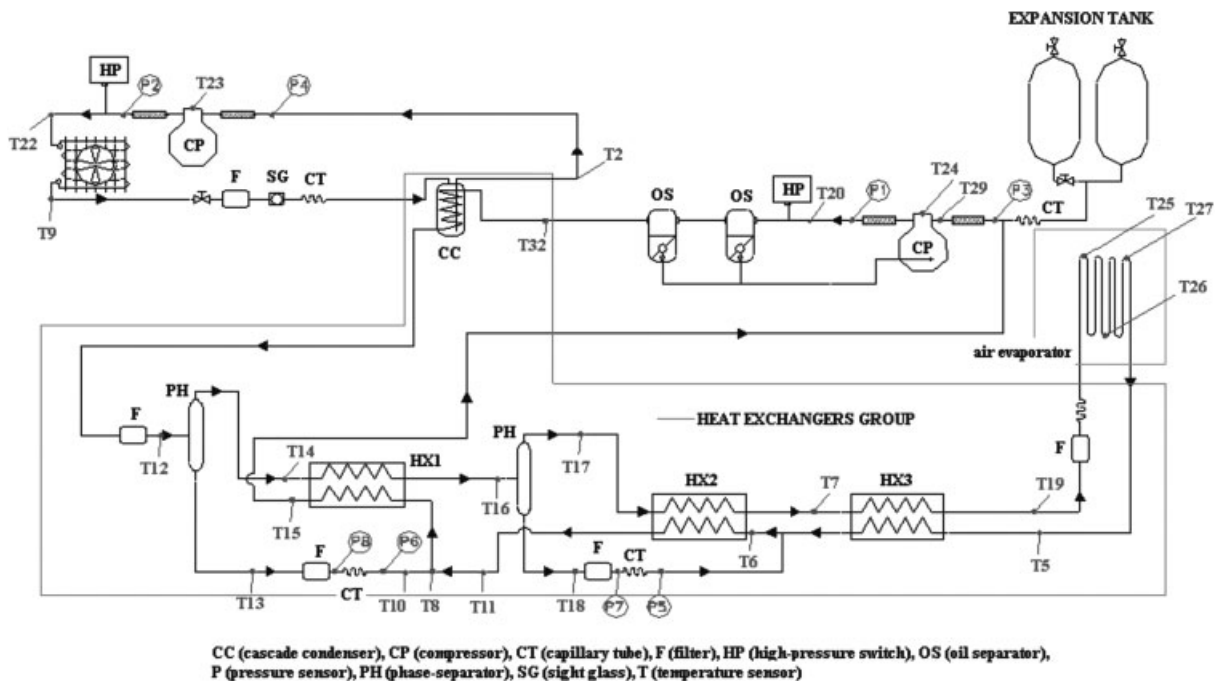


Figure 1. Sketch of the autocascade plant.

third exchanger (HX3). The total combined refrigerant stream from the first exchanger flows to the compressor, where it is recompressed for recirculation.

The heat exchangers HX1, HX2 and HX3 are double-pipe type working counter flow: the refrigerant evaporates in the annulus.

In the refrigerant processes, the multi-component refrigerant mix is repeatedly partially condensed, separated, cooled, expanded and warmed.

On the low-temperature circuit two oil separators and the expansion tank are inserted to avoid dangerous pressure levels at the shut-down of the plant. The oil used is polyester type with viscosity equal to ISO 22.

The temperatures were measured by means of thermocouples (T-type) electrically insulated and the pressures were monitored, thanks to piezoelectric sensors. A vacuum-insulated panel (VIP) has been used to insulate the refrigerated space at  $-150^{\circ}\text{C}$ ; with reference to this panel the results obtained by means of a thermographic method [7] have shown a thermal conductivity three times smaller than the equal thickness of polyurethane foam.

### 3. REFRIGERANT MIXTURE DESIGN

An autocascade refrigeration system is based on the following concept. Consider two refrigerants boiling at different temperatures at the same pressure. Mixing the higher boiling temperature refrigerant in liquid phase with the other refrigerant, the resulting mixture boils off in the range between the first refrigerant and the second refrigerant.

The refrigerant mixture employed in the low-temperature stage consists of seven components: R507, R245fa, R116, R23, R14, R740 and R290. The percentage of the components of the mixture is close. Successively the role of each refrigerant in the mixture considered is explained. The R507 and the R245fa are liquid after the first separator; the R507 helps with oil return, the R245fa also helps with oil return, but it aids in the heat transfer within the condenser. The R116 and the R23 are

liquid after the second separator; in particular, the R116 increases the heat transfer coefficient of the mixture. Considering the R14 and the methane, the resulting mixture can achieve a boiling temperature of  $-135^{\circ}\text{C}$ . Adding the argon to the mixture composed by the R14 and the methane it is possible to create a mixture boiling in the range between the mixture (R14 and methane) and the argon. Obviously also in the high-temperature stage the working substance is an HFC refrigerant. In the mixture working in the low-temperature circuit, methane and argon allow the suitable final expansion in the air evaporator as regards the temperature. As both methane and argon exhibit a critical pressure very different with respect to the others mixture components, it is not possible to find out an analytical expression for both thermodynamic and thermophysical properties of the entire mixture [8,9]. Consequently it has been necessary to study the non-azotropic mixture without methane and argon; these gases have been separately considered because binary interaction parameters are not available for very wide boiling mixtures. There are little or no experimental data for binary mixtures of the hydrocarbon (argon and methane) plus refrigerant components (R507, R245fa, R116, R23, R14). We have tried to add predictive schemes that estimate the interaction parameters when no data are present in Refprop [8] but, mixing fluids with such different volatilities, the prediction scheme is not very good. To overcome this obstacle two separate calculations have been considered: in the first one only R507, R245fa, R116, R23 and R14 and in the second one only the argon and the methane have been contemplated. No problem was encountered while separating the components with knowledge of leakage about the binary interaction parameters by means the program REFPROP: this hypothesis leads to acceptable results as reported below.

Regarding the separation processes the chemical potential (Gibbs function) has been used because approximately in a mixture the chemical potential of a component can represent its escape tendency [10].

In Figure 2 the thermodynamic pressure enthalpy diagram of the mixture studied without

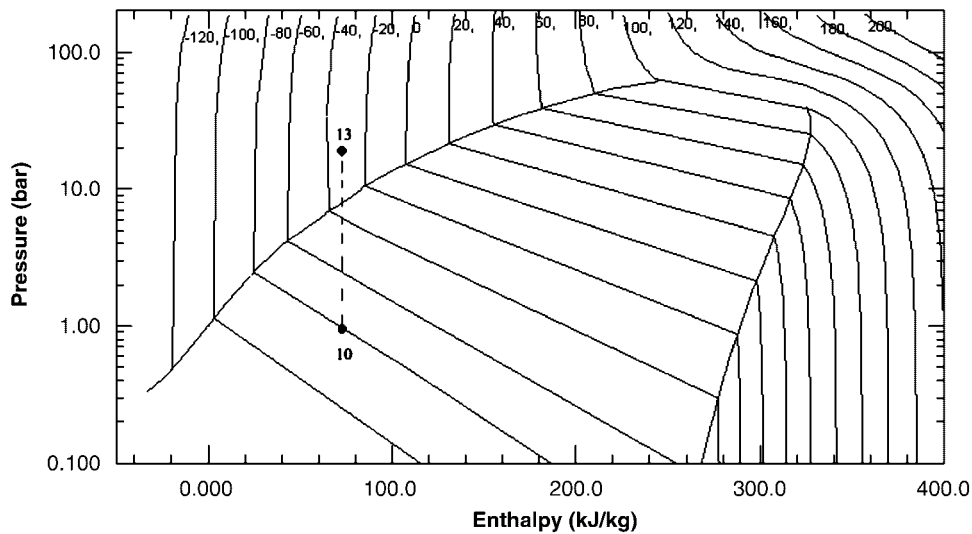


Figure 2. Pressure enthalpy thermodynamic diagram of the mixture in liquid phase (composition calculated at the exit of the first phase separator).

considering argon and methane is presented; in detail it shows the pressure enthalpy diagram of the mixture in liquid phase, whose composition has been calculated at the exit of the first phase separator. The throttling process starting by the point 13 is visible (see Figure 1); the temperature value readable in the diagram has been found like the experimental measurements revealing that the correctness of the hypothesis is based on without considering methane and of argon in the mixture simulation with reference to the liquid phase; these two substances in the processes occur at the first and at the second, the phase separator does not participate to throttling remaining in gas phase: this results allows a good accuracy in the prevision of the temperature and of the pressure after the throttling processes and, then, in the calculation of the properties used to evaluate the heat transfer coefficients regarding the annulus of the heat exchangers HX1 and HX2 (double-pipe type). For this reason smaller accuracy has been experimented calculating the temperatures for the refrigerant mixture partially condensed in HX1 and in HX2; analogous result with regard to the mixture flowing in HX3 and in the air evaporator. Only in the steady-state conditions and with reference to both the air evaporator and HX3, the results of the simulation are in agreement with

the experimental measurements because the properties of the argon–methane mixture flowing alone are analytically valuable as specified above.

As a further example, in Figure 3 the thermodynamic pressure enthalpy diagram of the mixture at the inlet of the second phase separator is reported. The refrigerant thermodynamic states at point 14 and at point 15 are evidenced. Also the points 6 and 9 are identified in the diagram. The great ‘temperature-glide’ of the mixture permitting the correct separation phase along the refrigerant circuit can be seen in Figure 3.

#### 4. SYSTEM COMPONENTS DESIGN

The system components design presents a lot of difficulties; in particular the size of both the capillary tubes [11,12] and the double-pipe heat exchangers (diameter and length) are critical points. In the autocascade refrigeration plant presented there are five capillary tubes. It has been easy to design the capillary tube mounted at the exit of the condenser at the high-temperature stage and at the discharge of the expansions tank, significant problems have been linked to the design of the capillary tubes in the low-temperature stage. The length and diameter of these tubes should be

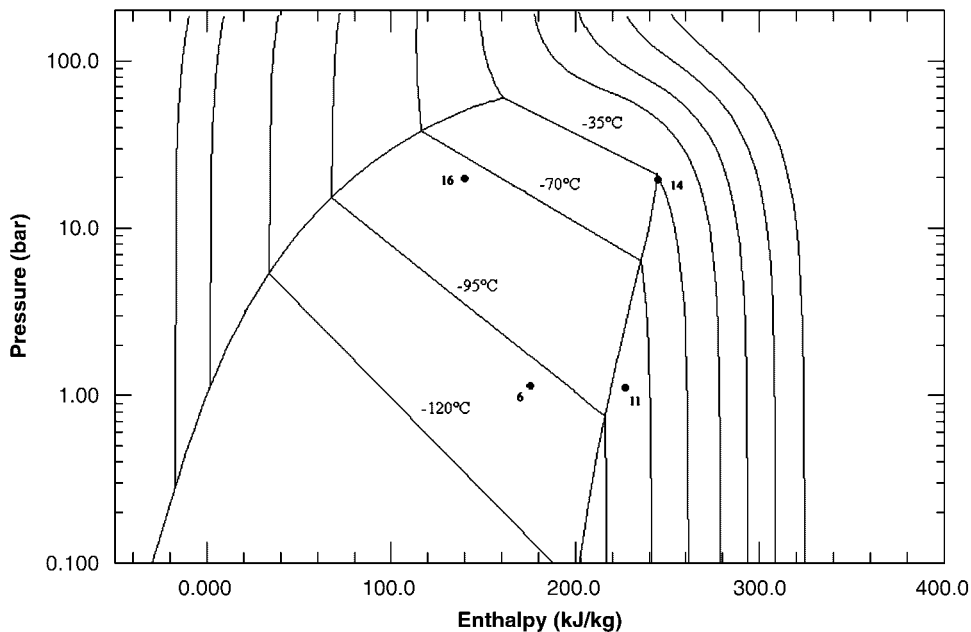


Figure 3. Pressure enthalpy thermodynamic diagram of the mixture at the inlet of the second phase separator.

adjusted so as to ensure an acceptable time to reach the steady state and a correct final temperature in the refrigerated space. Both aspects are influenced only by the last capillary tube that serves the air evaporator; while the remaining capillary tubes have to ensure both a circuits balance and a correct refrigerant temperature at their exit:  $-75^{\circ}\text{C}$  for the capillary tube at HX1 and  $-95^{\circ}\text{C}$  for the capillary tube at HX2.

In Figure 4 are reported the refrigerant temperatures at the points 5, 7 and 19 when the last capillary tube used is very short (2.00 m) and when it has a large diameter (1.88 mm). Both the short length and the large diameter of the capillary tube allow a great mass flow rate; consequently a great heat quantity can be removed in small time, so that the steady-state condition can be reached quickly. Because of the large capillary tube diameter an incorrect throttling process occurs hence it is impossible to condense a correct quantity of methane and argon since the temperature (points 5 in Figure 1) remains too high. Besides as a consequence of the high temperature level in the plant some mixture components does not condense in the phase separator completely and circulate maintaining

the suction pressure at the compressor at high level (about 3 bar). In Figure 5 the refrigerant pressure at points 7 and 8 is reported. The high-pressure difference (pressure drop) reveals a high refrigerant mass flow rate circulating into the plant when the last capillary tube is too short with a larger diameter; correct size of the last capillary tube (2.5 m length, 1.1 mm internal diameter) allows a pressure drop of about 0.3 bar.

As regards the heat exchangers design it has to be noted that the available correlations to calculate the heat convective transfer coefficient have been set up with reference to the refrigerant mixtures presenting a very small temperature glide [13,14]. These uncertainties augmented by the mixture properties unknown in some conditions (partial condensation in HX1 and HX2, enthalpy variations in HX3 and in the air evaporator in unsteady-state conditions) have caused a long test period to get the plant ready. Besides it is not correct to design the components with reference to the steady-state condition: their size could be too small. A lot of attempts to find out the correct dimensions of the heat exchangers have been carried out. An example regarding HX1 is reported below.

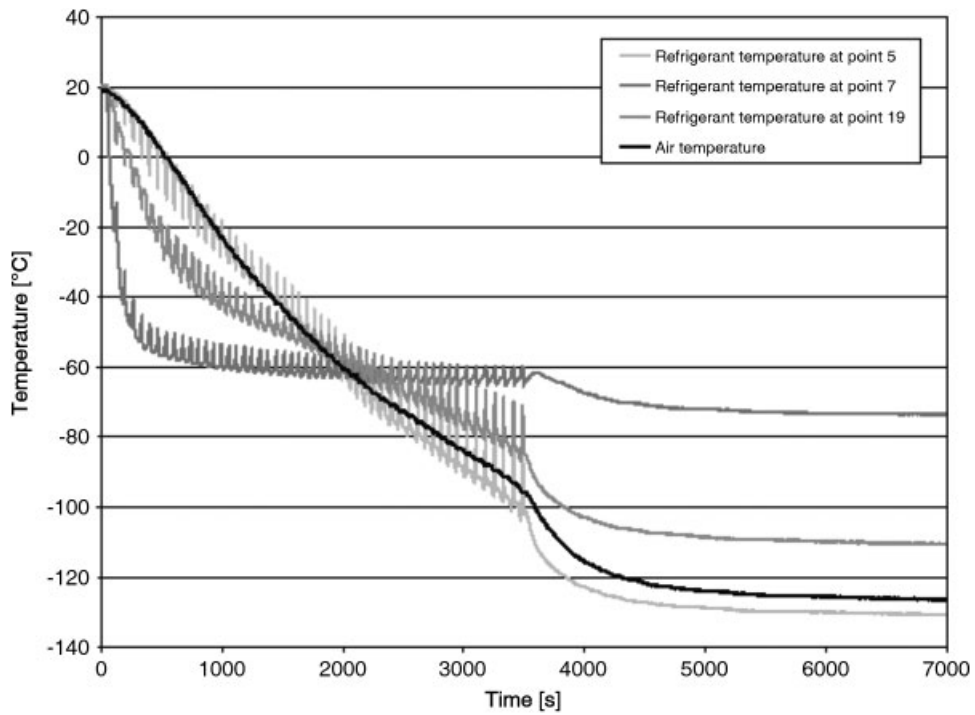


Figure 4. Refrigerant temperatures at the points 5, 7 and 19 when the last capillary tube used is very short.

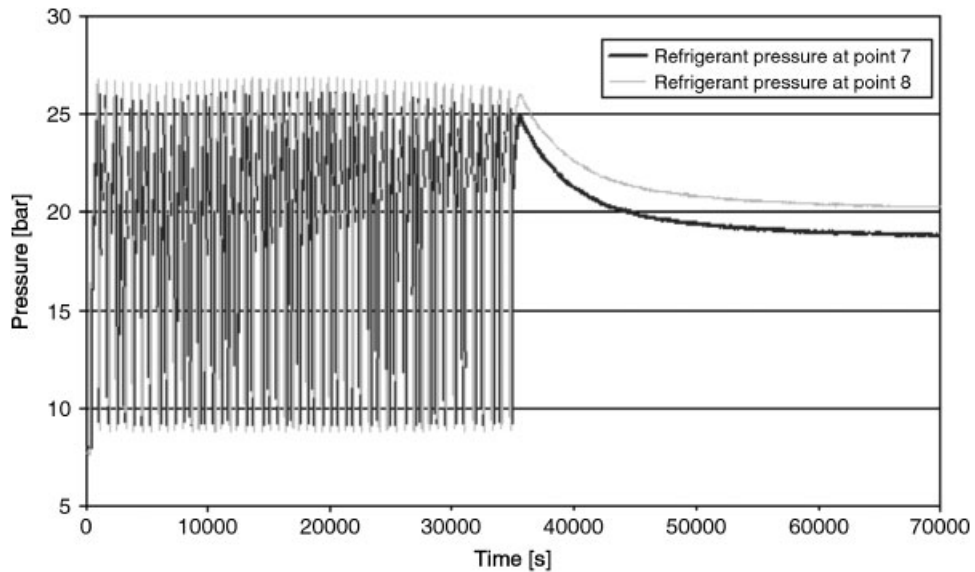


Figure 5. Refrigerant pressure at points 7 and 8.

One of the most critical point in the HX1 design was the selection of the external diameter of the internal tube where the condensing mixture flows

(from 10 to 12 mm). In this case the external diameter of the external tube is equal to 20 mm to balance the heat transfer coefficients between the



two refrigerant streams flowing in the annulus and in the internal tube. Augmenting the diameter a better process in the first phase separator has been verified, thanks to the decrease in pressure drop in the internal tube heat exchanger HX1 allowing a more correct condensing refrigerant mass flow rate.

In the heat exchangers HX2 and HX3, the external diameter of the external tube is equal to 16 mm while the external diameter of the internal tube is equal to 10 mm.

Keeping constant the phase-separator dimension, the selection of the refrigerant velocity at the phase-separator inlet is important: the velocity has to allow the formation of the correct composition mixture in liquid and gas phases. Only in this case it is possible to obtain a very low temperature at the points 6, 7 and 19; later a very low final evaporation temperature is obtainable.

In Figure 6 is reported the refrigerant temperature at point 7 with a smaller diameter of the internal tube of HX1. To verify the improvement it is possible to compare these results with the trend of the temperature at point 7 measured with the correct diameter for the heat exchanger HX1 (Fig. 6).

To explain the trends shown it is necessary to consider the particular working plant dynamics in the unsteady state. The mixture partially condenses in the internal tube of the heat exchanger while the mixture evaporates in the annulus. The rise in pressure during the start phase is dependent on the length of the internal tube diameter: a smaller diameter allows a rapid increment of the pressure while a slower increment of the pressure occurs using a greater diameter of the internal tube. It has to be noted that during the start phase when the pressure reaches a value of 29 bar, the pressure control shuts down the low stage, which restart when the pressure decreases to 11.5 bar; at each on-off cycle the temperatures of the point 19, 25, 26, 27, 5 decrease. The on-off cycles preserve the low stage compressor maintaining a low discharge temperature and avoid dangerous pressure levels. In view of the previous considerations, a smaller diameter of the internal tube causes an intense fluctuation of the temperature of point 7, whereas by employing a correct diameter the fluctuation is smoothed. Obviously the trend of the temperature of the point 7 is the same as that of the pressure.

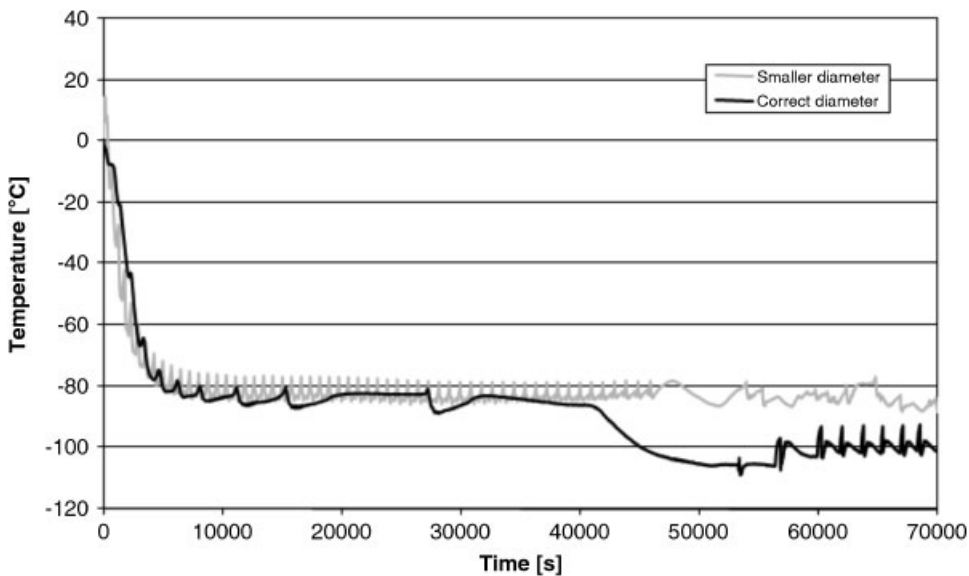


Figure 6. Refrigerant temperature at the point 7 for two different diameters of the internal tube of the heat exchanger HX1.

A photo of the heat exchangers assembled pertaining to the low-temperature stage is shown in Figure 7.

## 5. RESULTS

As regards the high-temperature stage, it is interesting to verify the correctness of the evaporation temperature at steady-state condition. In Figure 7 the evaporation temperature during both the unsteady and steady conditions is reported. When the entire mixture flows in the low-temperature stage in the steady state, the evaporation temperature rises at about  $-38^{\circ}\text{C}$ ; the superheating at the suction of the compressor is fixed to  $7^{\circ}\text{C}$  by the capillary tube and, thanks to the refrigeration power produced, the refrigerant mixture flowing in the low-stage temperature reaches the correct quality at the inlet of the first phase separator. In this type of plant the performances are strongly a function of the inlet condition of the refrigerant mixture to the first phase separator [3]. During the unsteady state the evaporation temperature is lower than the temperature in the steady state as a consequence of the

smaller refrigerant mass flow rate flowing in the circuit of the low-temperature stage. The high-temperature stage is air condensed: the performance has been reproduced equally when the external air temperature is equal to  $30^{\circ}\text{C}$ .

With reference to the refrigerant temperatures in the low-temperature stage, the only temperatures varying in the plant during the unsteady state are pertaining to the points 19, 25, 26, 27 and 5. From the start and about for 8 h, the refrigerant temperature at the outlet of the air evaporator is greater than the temperature at the exit of HX3; afterwards the refrigerant temperature at point 5 becomes smaller than the temperature at point 19; this occurs, thanks to the condensation and the consequent evaporation of some components of the mixture in the air evaporator. The process continues until the condensation of argon and of methane occurs completely: at this point the steady state is reached.

The refrigerant mixture temperature at the outlet of the evaporator and the air temperature in the refrigerated space versus time are reported in Figure 8.

A maximum air temperature vertical gradient of  $1.5^{\circ}\text{C}$  has been measured in the refrigerated space

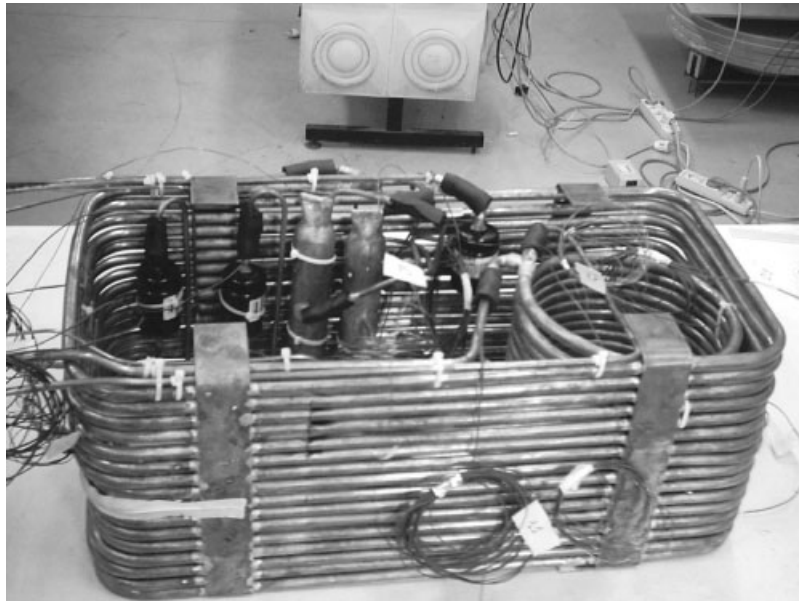


Figure 7. Evaporation temperature during the unsteady and steady conditions (high-temperature stage).



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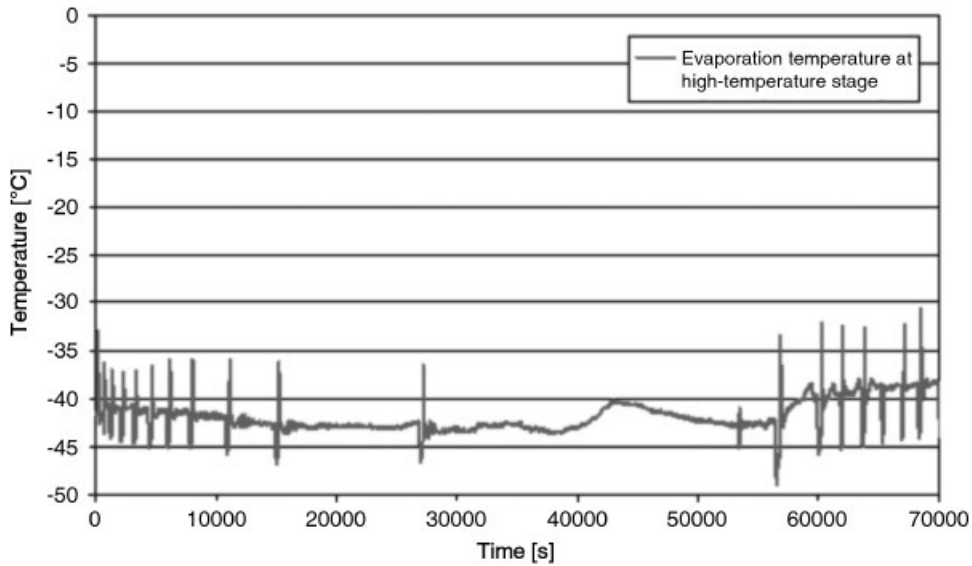


Figure 8. Refrigerant mixture temperature at the outlet of the air evaporator and the air temperature in the refrigerated space versus time.

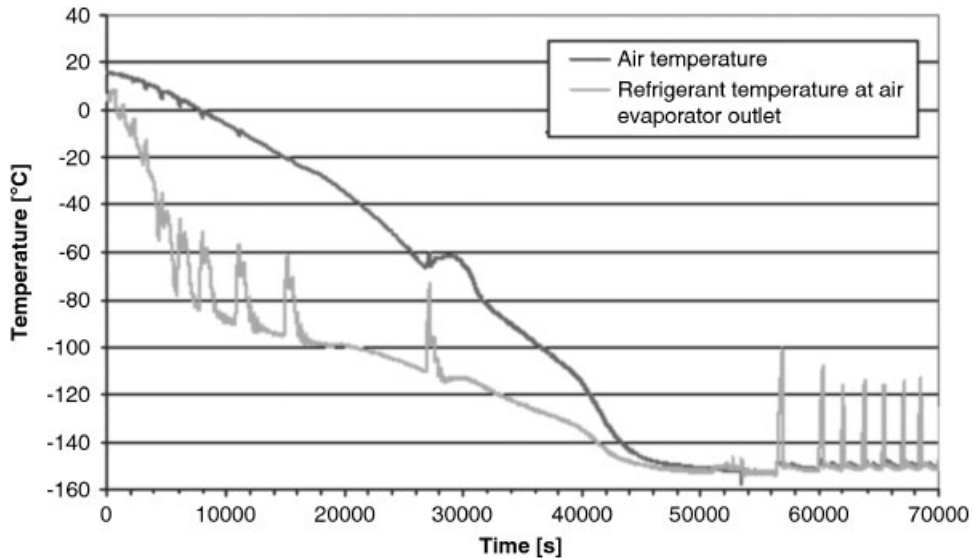


Figure 9. Refrigerant pressures at the suction and at the discharge of the compressor of the low-temperature stage versus time.

along the height (68 cm). During the unsteady working the refrigerant mixture drawn out by the expansion tank, flowing along the circuit that is at high temperature, allows the correct condensation

of some mixture components; for this reason the refrigerant pressure rises and the plant is stopped at 29 bar. Afterwards the pressures balance until a value of 11.5 bar when the plant restarts. In Figure 9

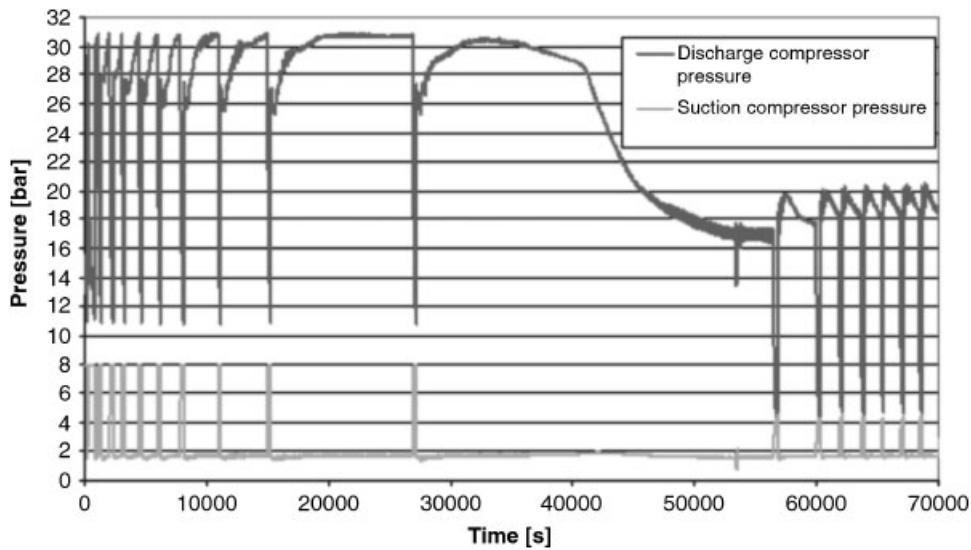


Figure 10. Photo of the heat exchangers assembled (low-temperature stage).

are reported the refrigerant pressures at both the suction and the discharge of the compressor of the low-temperature stage.

The steady-state condition is reached in about 14 h and then the plant works according to the temperature selected on the thermostat. The following cycle time is a function of only the thermal insulation of the refrigerated space: the long working time as to the stop time of the plant suggests a better insulation of the air evaporator.

When the thermal level in the plant allows the correct condensation of the mixture components, the discharge pressure decreases to about 20 bar. It has been noted that when the low-temperature stage is stopped by the thermostatic control, the high-temperature stage continues to work producing liquid refrigerant in the cascade condenser to serve the low-temperature stage at the restart. At this cryogenic temperature level no significance can be attributed to the energetic performances of the entire system. Usually analog temperature level can be obtained by means of liquid nitrogen using a complex arrangement in terms of plant components. With reference to the energetic performance, as expected the COP valuated like the ratio between the refrigeration power is necessary to maintain the space at  $-150^{\circ}\text{C}$  and the mean electrical power required

by the two compressors is very low. Because of the unknown refrigerant mass flow rate in the last evaporator, the evaluation of the refrigeration power cannot be evaluated as energy balance on the evaporator refrigerant side; consequently an easy mathematical model of the refrigerated space insulated with the VIP panels was used (Figure 10).

## 6. CONCLUSIONS

In this paper an autocascade refrigeration system for achieving ultra low temperature ( $-150^{\circ}\text{C}$ ) is presented. The system presents a high-temperature stage and a low-temperature stage connected with each other through a heat exchanger (evaporator/condenser). In the low-temperature side only HFC refrigerants types are allowed in addition to argon and methane; also in the high-temperature side a HFC refrigerant is used as refrigerant. Typical aspects of the complex design process have been evidenced with reference to the heat exchangers and to the capillary tubes. A lot of tests have been carried out to verify the absence of instabilities in the steady-state conditions: the plant has operated for a long time without troubles. The calculated value of the COP is very low, but this system

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requires only the electric energy to work avoiding the complex plant necessary to refrigerate by means of the liquid nitrogen.

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