



# Transient phenomena initiating phase transition in dilution refrigerator



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## ABSTRACT

We have analysed the transient processes in a Dilution Refrigerator (DR) leading to phase separation of  $^3\text{He}$ – $^4\text{He}$  mixture into concentrated and dilute solutions using an extended SIDFO (Simulation of Integrated Dilution refrigerator For Optimisation) Pradhan et al. (2013) analysis tool. The evolution of the phase separation interface along the concentrated channel and its arrival in the mixing chamber for a given physical situation has been thoroughly examined. The significance of the  $^3\text{He}/^4\text{He}$  composition in the mixer, the flow rate and the still power on the phase separation is presented here. The consequences of several relevant thermodynamic parameters and the composition of isotopic mixture for successful function of the DR is also discussed in this paper.

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## 1. Introduction

The dynamic processes in a dilution refrigerator during cool down, especially phase separation are of fundamental importance for gaining insight and extracting information on the various thermodynamic parameters responsible for the optimal functioning of the machine. On condensing the isotopic gas mixture in the refrigerator, it takes a few hours to reach temperature of 1.2–1.5 K. Based on the desired base temperature and cooling power, on isotopic mixture of  $^3\text{He}$  and  $^4\text{He}$  is prepared. For a predetermined gas composition,  $^4\text{He}$  becomes superfluid at that temperature and  $^3\text{He}$  begins to condense. After a short while, phase separation initiates at the high temperature end of concentrated channel of the heat exchanger and eventually the interface begin to propagate toward the mixing chamber. All these phenomena are primarily relies on the effectiveness of the condenser, heat exchanger, initial isotopic gas composition, still heater power and the circulating system. The time variation of all these attributes have been analysed to provide a stable steady-state condition of the dilution refrigerator with optimum base temperature and designed cooling power.

The functioning of a dilution refrigerator primarily concerns with the condensation of circulating gas mixture in the 1 K pot and occurrence of phase separation within the mixing chamber (MC). In the temperature range 1.2–1.5 K within the 1 K pot  $^4\text{He}$  becomes superfluid while  $^3\text{He}$  gas at that pressure and temperature remains in the liquid phase. In view of the fact that with the passage of time, the temperature of still gradually falls thereby

reducing the vapour pressure substantially. This will result in low flow rate which will prevent the circulating gas from causing phase separation. Extended SIDFO enables us to estimate that for a gas mixture bearing 30%  $^3\text{He}$  with a still temperature of 0.6 K, phase separation occurs at the inlet of the heat exchanger.

This article represents an extension of earlier work [1] and the SIDFO tool used therein has been upgraded to accommodate the stages of development preceding the steady state condition with time and to obtain the base temperature from a homogenous mixture of liquid  $^3\text{He}$  and superfluid  $^4\text{He}$ . Simulation and modelling show that the phase-separation process advances with time along the concentrated channel landing up at the mixing chamber. To the best of our knowledge, no comprehensive study to investigate the time dependent analysis of transitory phases has been attempted so far. Selection of still heater power and still temperature so as to set up the right conditions during cool-down and the effect of initial  $^3\text{He}/^4\text{He}$  concentration on optimal performance of the DR have been explored in this work using extended SIDFO.

## 2. Materials and methods

To study the operation of dilution refrigerator during transient operating conditions, SIDFO is extended by introducing time variables. This has been done on the basis of mass- and energy-balance approach involving time-dependent relationship between pressure, volume, temperature and  $^3\text{He}$  concentration. Subtle changes occurring in the process of cool down phenomena ahead of reaching base temperature turn out to be rather complex. Formulation for the simulation process requires fundamental understanding of the various stages of cooling and the appropriate fluid properties

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as a function of temperature. The  $^3\text{He}/^4\text{He}$  mixture with different proportion is introduced into the system at about 1000 mbar of pressure and continuously cools down on exchanging heat with the 1.5 K superfluid inside the 1 K pot. The model can reasonably predict the cool-down time and time required for reaching to steady state. The simulator SIDFO is articulated by stringing together each component of the system and numerically solved before integrating together [1]. Additional auxiliary structured modules are added to create the extended SIDFO in order to modify the process flow sheet easily with minimal recoding. By modular, we mean that the code which represents each piece of equipment is assembled into one sub-routine. The simulation helps to diagnose and identify the system performance, control and operation for a variety of input conditions. The primary goal of the simulation is to calculate the values of the temperature, pressure and  $^3\text{He}$  concentration of the  $^3\text{He}$ - $^4\text{He}$  mixture over time with reasonable accuracy for a set of probable input conditions. For all practical reasons, it is desirable to have phase separation in the mixing chamber for optimal performance. Hence the programme precisely indicates the location of the phase-separation interface during cool-down under different operating conditions. For an initial concentration of  $^3\text{He}$  in the homogenous mixture, the phase separation temperature and  $^3\text{He}$  concentration in the separated mixture is calculated using auxiliary modules.

### 2.1. Process description

The dilution refrigeration consists of five distinct units which are interconnected to make a complete and closed system. They are the condenser (1 K-pot), the still, the heat exchanger, the mixing chamber and the gas circulating system. Schematics of the process flow diagram for the system are given in Fig. 1. The room-temperature circulating pump extracts the mixture from the still and returns it to the dilution unit through successive cooling stages at 77 K and

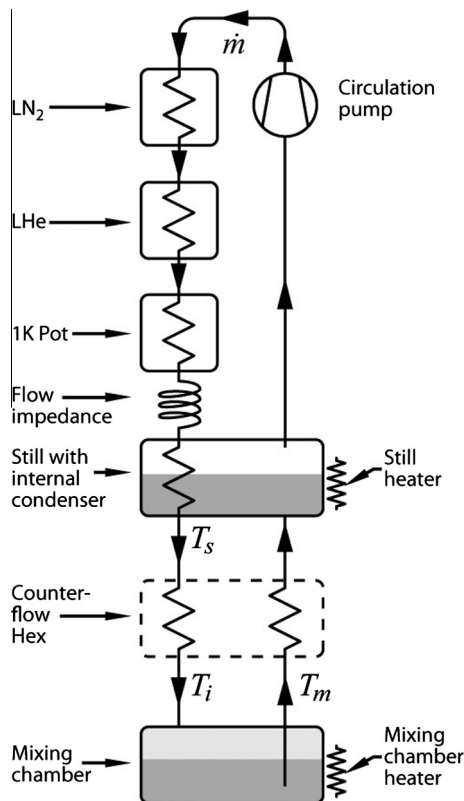


Fig. 1. Schematic flow diagram of dilution refrigerator considered for simulation.

4 K temperatures before it enters the condenser. Simulation relies on the experimental data with a condensation temperature of 1.2 K with a cooling power of about 18 mW [2].

Initially the incoming isotopic gas mixture is continuously cooled and condensed by exchanging heat with the 1 K pot, filling the colder parts of the refrigerator to a temperature close to 1.2 K. This may take a few hours depending on the efficiency of the condenser and the presence of residual heat, however small it is. As time progresses the  $^3\text{He}$  content of the circulating gas increases and the  $^4\text{He}$  largely settles down inside the dilute channel starting from MC to still. The secondary flow impedance in the concentrated line after the still ensures the mixture is fully liquefied before entering the HeX.

The still thus filled with  $^3\text{He}/^4\text{He}$  solution is pumped out to ensure continuous circulation and maintain dynamic equilibrium at low temperature. This sucked gas is pushed back into the cryostat through the discharge port of the pump. The concentrated mixture leaving out of the condenser is further cooled by the still before it is directed to the main heat exchanger. The heat exchanger plays the key role of reducing the temperature of the incoming concentrated  $^3\text{He}$  to a temperature close to that of the mixing chamber by exchanging heat with the returning dilute solution from the MC. Finally, the concentrated  $^3\text{He}$  mixture enters the MC where the interface between the concentrated and dilute phases occurs. The  $^3\text{He}$ -rich concentrated phase floats above the  $^4\text{He}$ -rich dilute phase due to the density difference between the two liquid phases.

The transient cool-down of dilution refrigerator comprises of following discrete steps.

1. Filling of the dilution insert with the homogenous mixture of  $^3\text{He}$ - $^4\text{He}$  with a concentration ranges from 25% to 40% of  $^3\text{He}$ .
2. The condensation of gas mixture by heat exchange with the 1-K pot.
3. The circulation of the mixture by pumping thereby reducing the vapour pressure at the still and bringing down the temperature further.
4. Maintaining the still temperature through still heater to ensure adequate flow and initiation of phase separation.
5. The phase separation interface gradually reaches the mixing chamber and cools further to the base temperature by dilution of  $^3\text{He}$  atoms in  $^3\text{He}$ - $^4\text{He}$  mixture.

For optimal performance of the dilution refrigerator, the total amount of mixture and ratio of  $^3\text{He}/^4\text{He}$  in the mixture must be tuned to obtain phase boundaries within the MC at a desired position. In order to avoid degradation of cooling power, an extended SIDFO helps to diagnose and avoid phase transition in the HeX. While considering the  $^3\text{He}/^4\text{He}$  ratio inside the DR, the dead volume of the circulating pump presumed to be negligible compared to the total volume of the closed circulating system. After the occurrence of the phase separation, osmotic drive facilitates the continuous flow of  $^3\text{He}$  across the phase boundary within the MC and produces desired cooling effect.

### 2.2. Modelling details

Each component of the dilution refrigerator is conveniently represented in the form of a control volume as already described in the literature [1]. It is important to determine the position of the phase separation for possible sets of operating conditions. The circulation rate is effectively controlled by the still heater and this in turn controls the heat extraction rate in the MC. Because of the large specific heat of the dilute  $^3\text{He}$  returning from the MC to the still coupled with marginal thermal load from the incoming concentrated phase keeps the still on cooling.

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