Cryogenics 48 (2008) 511–514

Contents lists available at ScienceDirect

Cryogenics

journal homepage: www.elsevier.com/locate/cryogenics

^{3}He/^{4}He dilution refrigerator with high cooling capacity and direct pulse tube pre-cooling

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ARTICLE INFO

Article history:
Received 15 May 2008
Received in revised form 1 July 2008
Accepted 16 July 2008

Keywords:
E. Dilution
E. Pulse tube
E. Heat exchangers

ABSTRACT

In the article, a ^{3}He/^{4}He dilution refrigerator (DR) is described which is pre-cooled by a commercial two-stage pulse tube refrigerator (PTR); cryo-liquids are not necessary with this type of milli-kelvin refrigerator. The simple design of the condensation stage of this so-called dry DR is novel and explained in detail. In most dry DRs the circulating ^{3}He gas is cooled by a two-stage PTR to a temperature of about 4 K. In the next cooling step, the ^{3}He flow is cooled and partially liquefied in a Joule–Thomson circuit, before it is run to the dilution refrigeration unit. The counterflow heat exchanger of the Joule–Thomson circuit is cooled by the cold ^{3}He gas pumped from the still of the DR. In the DR described here, the heat exchanger of the Joule–Thomson stage was omitted entirely; in the present design, the ^{3}He gas is cooled by the PTR in three different heat exchangers, with the first one mounted on the first stage of the PTR, the second one on the regenerator of the second stage, and the third one on the cold end of the second stage. The heat load caused by the ^{3}He flow is mostly absorbed by the first two heat exchangers. Thus the ^{3}He flow presents only a small heat load to the second stage of the PTR, which therefore operates close to its base temperature of 2.5 K at all times. A pre-cooling temperature of 2.5 K of the ^{3}He flow is sufficiently low to run a DR without further pre-cooling. The simplified condensation system allows for a shorter, compacter and more economical design of the DR. Additionally, the pumping speed of the turbo pump is no longer obstructed by the counterflow heat exchanger of the Joule Thomson stage as in our earlier DR design.

1. Introduction

In low temperature physics, temperatures below 0.3 K usually are generated by DRs. Several cryo-technology companies worldwide offer DRs commercially. The most important advantage of DRs in comparison with other refrigeration techniques is that they can be operated continuously at high refrigeration capacities for arbitrarily long times. Whereas in the past DRs have always been operated in liquid helium dewars, in recent years so-called dry DRs have been developed. With this type of DR, closed cycle cryocoolers are utilized to pre-cool the dilution unit. Vibrations of PTRs are negligibly small for most experimental applications, and thus PTRs are best suited for pre-cooling a milli-kelvin refrigerator. Cryogens are no longer necessary with these DRs, and the helium dewar of a conventional cryostat is replaced by a simple vacuum jacket. These cryostats can easily be automated, and therefore are convenient to operate. With the price of liquid helium steadily on the rise, dry DRs also offer considerable advantages in running cost.

Our DR has been simplified compared to earlier models [1]. After entering the cryostat, the ^{3}He gas stream is purified and cooled to a temperature of ~50 K in a charcoal purifier at the first stage of the PTR, as in our previous setup. Next, the ^{3}He is further cooled in a heat exchanger affixed to the regenerator tube of the second stage of the PTR. A considerable amount of refrigeration capacity can be provided at the location of this regenerator as has been shown in previous theoretical and experimental work [2–4]. This refrigeration capacity of the second regenerator is available in addition to the one at the cold end of the second pulse tube. The heat exchanger can be designed as a continuous heat exchanger or as a step exchanger [4,5] (or several step exchangers in a row). The step exchanger described in [4] extends into the interior of the regenerator to provide better thermal contact between the gas flow of the PTR with the heat exchanger body. By contrast, the continuous heat exchanger used in our work is mounted at the outside of the regenerator; it can be seen from our experiments that the heat exchange is clearly adequate to pre-cool the ^{3}He. The simple construction of the heat exchanger is especially noteworthy. Two different methods to manufacture this heat exchanger are described in the paper. In the heat exchangers of the first stage and of the regenerator of the second stage, more than 95% of the enthalpy of the ^{3}He can be removed.

Finally, the ^{3}He stream is cooled in a heat exchanger attached to the second stage of the PTR. The remaining enthalpy to be absorbed there is small, and thus the second pulse tube runs near its base temperature of 2.5 K, almost independent of the ^{3}He flow

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doi:10.1016/j.cryogenics.2008.07.004

rate of our DR. We will see from the enthalpy diagram of ^{3}He that its temperature of 2.5 K at a pressure of 0.5×10^5 Pa to 1×10^5 Pa is sufficient for the operation of a DR.

2. Experimental setup

In Fig. 1, a sectional drawing of the apparatus is shown. For cooling from room temperature to 10 K, the dilution unit is situated in a separate inner vacuum can with H_2 exchange gas. This inner vacuum can is thermally anchored at the second stage of our PTR (Cryomech, PT4-05 [6]) via a set of copper braided straps. These compensate the small cyclical expansions and contractions of the cold head during operation, and also compensate thermal expansion effects of the cryostat during cool-down. Furthermore, the braided straps attenuate the temperature oscillations (of about 0.15 K) of the cold head by a factor of four before they reach the inner vacuum can in our setup. The second stage of the PTR has a cooling capacity of 0.5 W at a temperature of 4.2 K; the first stage, which has to cool a big radiation shield, provides 22 W at 55 K.

The ^{3}He is cooled by the PTR in three steps, before it enters the dilution unit through a flow restriction (Fig. 1). The first cooling step occurs in a charcoal trap which is thermally anchored at the first stage of the PTR. The cold trap is necessary to remove contaminations of air, water and pumping oil from the gas flow; the temperature of the ^{3}He is ~50 K when it leaves the cold trap. Next, the gas flow is further cooled in a heat exchanger attached to the regenerator of the second stage of the PTR. We have tested two different types of heat exchangers in our cryostat. The first type was made from eight half-shells of brass which were clamped to the outside of the steel tube of the regenerator; the ^{3}He input line was soft soldered to the half-shells. This heat exchanger has been described in [7]. A big advantage of this concept is that the heat exchanger can easily be mounted to the PTR in a fully installed DR without removing the PTR from the cryostat. A sketch of the heat exchanger is shown in the insert of Fig. 2.

The second type (insert of Fig. 2) is almost identical to heat exchangers which have been used previously in dry miniature helium liquefiers [8,9]. Sixteen turns of stainless steel tubing (2 mm i.d.) were soft soldered to the regenerator. The soldering was carried out at Cryomech. The entire length of the regenerator tube was used to fabricate the heat exchanger. In the next cooling step, the ^{3}He stream is cooled in a simple heat exchanger which is bolted to the cold end of the second pulse tube. This heat exchanger is made of a copper stud where a CuNi capillary is soft soldered to (insert of Fig. 2). After the third pre-cooling step, the ^{3}He was directly run to the flow restriction of the dilution unit ($z = 0.4 \times 10^{18}$ m^{-3}) without further pre-cooling. This is in contrast to all of our earlier DRs, where the ^{3}He was always pre-cooled in another heat exchanger which was placed in the pumping line of the still. In the new concept, the flow of ^{3}He on the low pressure side of the DR is no longer constricted by this heat exchanger. The condensation process is described in detail in Section 3 using the enthalpy-pressure diagram. A separate pressure line was inserted into the DR to allow for exact pressure measurements on the flowing ^{3}He before it entered the flow restriction (Fig. 1).

The dilution unit was made at the WMI; its design was straightforward, consisting of a still, a continuous heat exchanger, two step heat exchangers and a large mixing chamber. A similar dilution unit was described in [10]. In order to pump the ^{3}He from the still, two turbo pumps (Pfeiffer TMH 1601) were used in parallel, backed by three Alcatel 2033H rotary pumps.

At the outlet of the heat exchangers of the second regenerator, thermometers (Cernox [11]) were available to monitor the temperature of the ^{3}He stream. In addition, the temperatures of the first stage of the PTR, of the cold trap and of the second stage of the

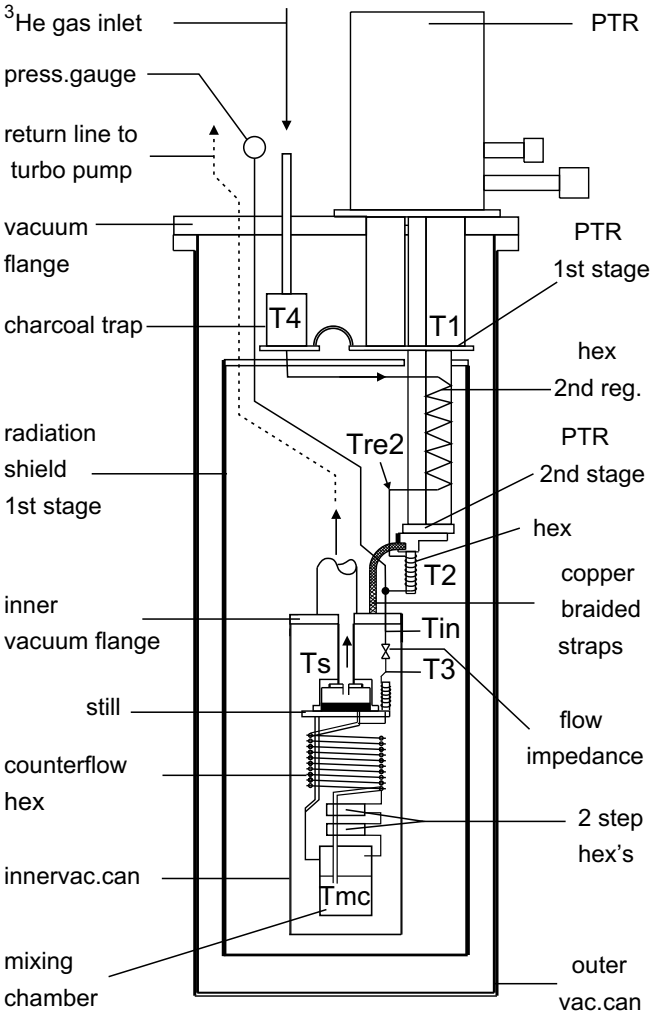

Fig. 1. Sectional sketch of the DR. The ^{3}He of the dilution cooler is directly run from the three heat exchangers of the PTR to the dilution unit without further pre-cooling. Temperature sensors are placed at all critical spots of the refrigerator. Total length of the cryostat is 1.20 m.

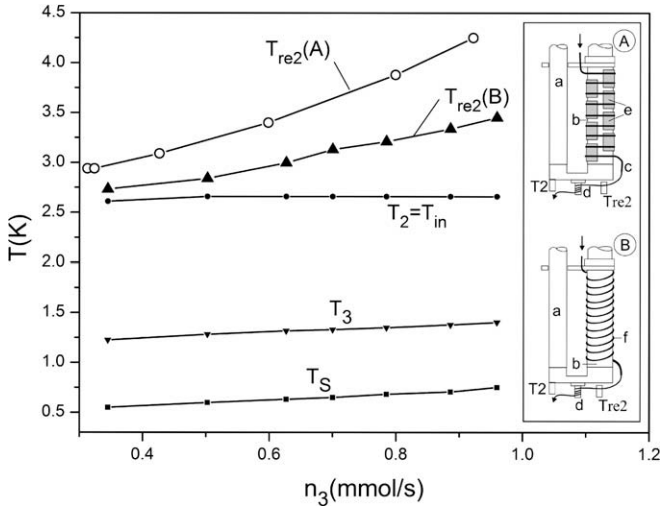

Fig. 2. Temperatures of the condensation process as a function of the ^{3}He flow rate n_3. T_{re2}(A) and T_{re2}(B) are the ^{3}He temperatures after the heat exchangers A or B at the second regenerator (see insert). T_2 is the temperature of the 2nd stage. T_{in} and T_3 are the ^{3}He temperatures before and after the flow restriction, and T_s is the still temperature. Insert: a – 2nd pulse tube; b – 2nd regenerator; c – CuNi tube; d – heat exchanger; e – half-shells; f – stainless steel tube.

PTR are monitored during the experiment (PT100, Cernox). Furthermore, the temperatures at both ends of the flow restriction could be measured with thick film resistance thermometers, as well as the temperatures of the still and of the mixing chamber (Fig. 1). The mixing chamber thermometer and the still thermometer had been calibrated with a ^{3}He melting curve thermometer [12].

3. Results

To condense the ^{3}He/^{4}He mash at the beginning of an experiment, the ^{3}He/^{4}He mash was circulated through the cryostat with the rotary pumps at an outlet pressure of only 0.1 MPa, whereby part of the gas was continuously liquefied. The liquefied fraction of the ^{3}He/^{4}He was steadily replaced by gas from the storage tanks. The rate of liquefaction was 50 std. l/h of gas; a compressor was not needed for the condensation process.

In Fig. 2, the ^{3}He pre-cooling temperatures T_{re2}(A) and T_{re2}(B) after the second regenerator are plotted as a function of the throughput n_3, where A and B stand for the two versions of heat exchangers depicted in the insert of Fig. 2. The heat exchanger, where a stainless steel tube is soldered to the regenerator tube, produces slightly lower pre-cooling temperatures. In addition, the temperatures of the ^{3}He at the inlet (T_{in}) and at the outlet of the flow restriction (T_3) are given; and finally, the temperature of the liquid ^{3}He/^{4}He in the still (T_s) is included in Fig. 2. T_{in} equals precisely the temperature of the second stage of the PTR (T_2); T_2 and T_{in} are not affected at all by the heat input of the ^{3}He flow in our experimental range of flows. This suggests that the PTR could cope with an even higher ^{3}He circulation rate. T_3 varies between 1.2 K and 1.4 K, and T_S between 0.55 K and 0.75 K in our experiment.

In Figs. 3 and 4 we give an example in a ^{3}He enthalpy diagram on how the condensation process occurs in our DR. The enthalpy diagram was compiled with He3Pak 1.20, software published recently [13]. After the ^{3}He gas leaves the heat exchanger at the second regenerator (point 1; p = 0.91 bar, T = 3.21 K), it is condensed and cooled at constant pressure to T = 2.66 K (point 2) in the heat exchanger attached to the second pulse tube. In the impedance that follows, the liquid ^{3}He is expanded at constant enthalpy to point 3 which is in the two-phase regime. The temperature T_3 (1.35 K) has been measured; the pressure at point 3 is the corresponding vapor pressure. Subsequently, the ^{3}He is led to a heat exchanger in the still; its cooling capacity is used to condense the gaseous fraction of the ^{3}He and to cool the liquid to the temperature of the still (point 4). Admixtures of ^{4}He to the ^{3}He flow were always below 5 percent and are not considered in the following.

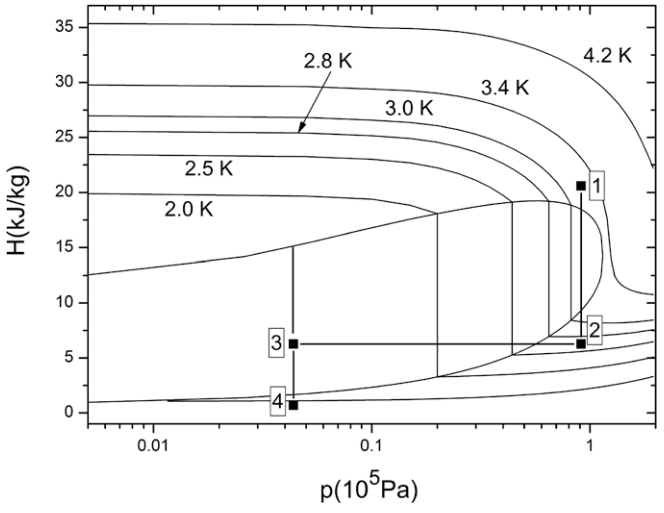

Fig. 3. Enthalpy-pressure diagram of ^{3}He in a semi-logarithmic plot. An example of the liquefaction process in our cryostat is depicted in the graph (points 1–4). For details see text (10^5 Pa = 1 bar).

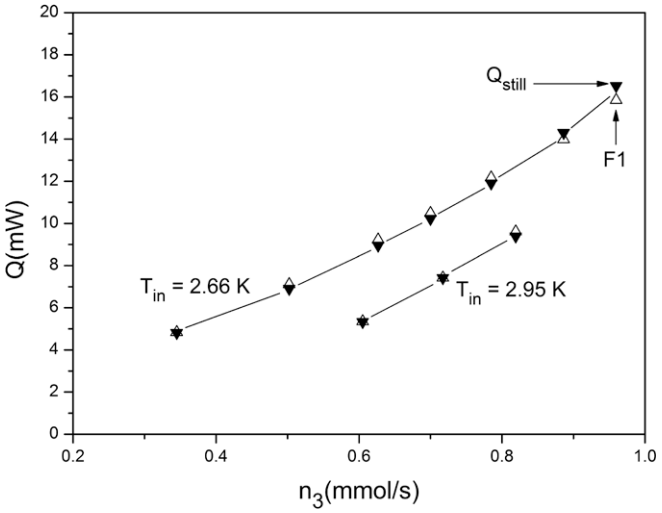

Fig. 4. Heat applied at the still Q_S and $F = n_3 * [H_3(p_{out}, T_{out}) - H_3(p_{in}, T_{in})]$ as a function of the ^{3}He flow n_3 for two different inlet temperatures T_{in}. For details see text.

In our cryostat, experiments were carried out at two different pre-cooling temperatures (2.66 K and 2.95 K). The highest pre-cooling temperature possible, T_{in} (max), can be derived from the enthalpy balance of the condensation stage

$$H_3(p_{in}, T_{in}) + Q_s/n_3 = H_3(p_{out}, T_{out}). \quad (1)$$

Here, H_3 is the enthalpy, and p_{in}, T_{in} (p_{out}, T_{out}) are the pressure and the temperature of the ^{3}He flow entering (leaving) the dilution unit. Q_S is the heat supplied to the still. The enthalpies can be calculated (e.g. using He3Pak), but $H_3(p_{out}, T_{out})$ can also be calculated from the gas law:

$$H_3(p_{out}, T_{out}) = \frac{5}{2} * R * T_{out}, \quad (2)$$

where R is the gas constant and $p_{out} = 0$.

Before we calculated T_{in} (max), it was verified that the experimental parameters of the cryostat were in agreement with Eq. (1). P_{in} was measured with the pressure gauge explained in the previous chapter; p_{out}, T_{in}, $T_{out} = T_S$ and n_3 were all measured. Thermal relaxation times are long in the PTR–DR cooler; in order to stabilize all the temperatures in the refrigerator, waiting times of about 6 hours between measuring points were needed. Eq. (1) was written as $Q_S = n_3 * [H_3(p_{out}, T_{out}) - H_3(p_{in}, T_{in})]$ and Q_S and $\{n_3 * [H_3(p_{out}, T_{out}) - H_3(p_{in}, T_{in})]\}$ were plotted as a function of n_3. Two sets of experimental data were available, one set with T_{in} = 2.66 K, and one with T_{in} = 2.95 K. To measure the second data set, the second pulse tube was heated with a constant thermal load of 70 mW. For the entire range of ^{3}He throughputs we find that Eq. (1) is satisfied. For values of Q_s = 0, $T_s = T_{out}$ = 0.75 K, p_{in} = 0.12 MPa, He3Pak yields a value of T_{in}(max) = 3.35 K. All commercial two-stage PTRs have base temperatures well below 3 K, and should therefore be able to pre-cool a DR without an intermediate pre-cooling stage.

In Fig. 5, the base temperatures of the mixing chamber are given. The lowest data point was taken with just the forepumps circulating the ^{3}He. With the plain dilution unit that was used for the experiments, the base temperature was between 8 mK and 20 mK, depending on the ^{3}He flow rate.

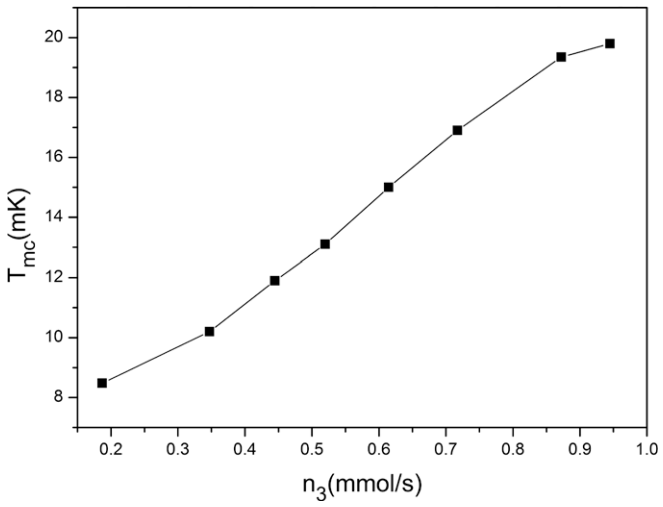

Fig. 5. Base temperatures of the mixing chamber as a function of the ^{3}He flow rate.

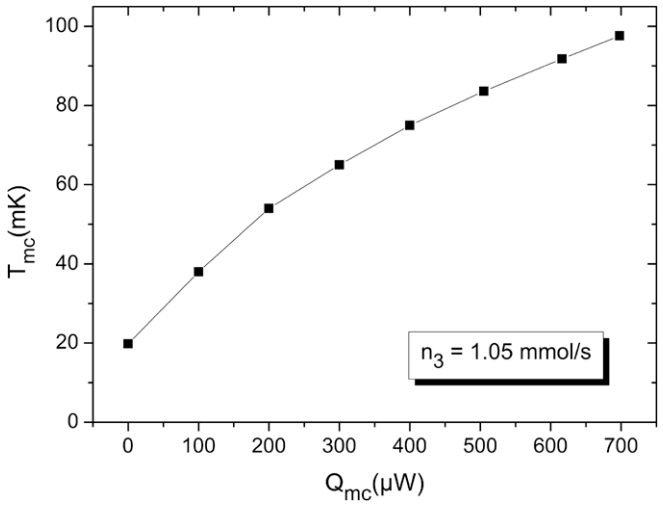

Fig. 6. Cooling capacity of the mixing chamber for a high flow rate of 1.05 mmol/s.

Finally, refrigeration capacity measurements are depicted in Fig. 6. For a ^{3}He flow rate of 1.05 mmol/s, which is close to the maximum flow possible with our DR, we found a refrigeration power of 0.7 mW at a mixing chamber temperature of 100 mK. For this test, the heater and the thermometers were attached to the outside bottom plate of the mixing chamber which had a big silver sponge that provided the thermal contact to the ^{3}He/^{4}He mixture inside.

4. Summary

Constructing dry DRs is becoming simpler. We show in the paper that a powerful dry DR can be pre-cooled by a PTR without an intermediate cooling stage. To cool the dilution unit, a standard PTR was utilized with a heat exchanger at its second regenerator that is easy to make. The second stage of the PTR always ran close to its base temperature of 2.5 K, independent of the ^{3}He circulation rate of the DR. It is demonstrated that the limiting temperature of the PTR, below which the direct pre-cooling is possible, is 3.35 K. The maximum cooling power of the DR was 0.7 mW at a temperature of the mixing chamber of 100 mK. In the future, one can expect that dry DRs with direct pre-cooling will become serious competitors to DRs with intermediate Joule–Thomson pre-cooling.

Acknowledgement

The author thanks A.T.A.M. de Waele for helpful comments and J. Hoehne of VeriCold Inc. for support.

References

[1] Uhlig K. ^{3}He/^{4}He dilution refrigerator with pulse tube pre-cooling. Cryogenics 2002;42(2):73–7.
[2] Radebaugh R, Marquardt E, Gary J, O'Gallagher A. Regenerator behavior with heat input or removal at intermediate temperatures. Cryocooler 11, 2001:409–18. <http://www.cryogenics.nist.gov/Papers/ICC11paper4regen.pdf>.
[3] Zhu S, Ichikawa M, Nogawa M, Inoue T. 4 K pulse tube refrigerator and excess cooling power. Adv Cryo Eng 2002;47A:633.
[4] Ravex A, Trollier T, Tanchon J, Prouvé T. Free third-stage cooling for two-stage 4 K pulse tube coolers. Cryocooler 14, 2006:157–5. <http://www.conferences.library.wisc.edu/index.php/icc/article/download/21/21>.
[5] Prouvé T, Godfrin H, Gianèse C, Triqueneaux S, Ravex A. Pulse-tube dilution refrigeration below 10 mK for astrophysics. J Low Temp Phys 2008;151(3/4):640–4.
[6] Cryomech Inc., 113 Falso Drive, Syracuse, NY 13211, USA.
[7] Uhlig K. Condensation stage of a pulse tube pre-cooled dilution refrigerator. Cryogenics 2008;48(3/4):138–41.
[8] Wang C. Helium liquefaction with a 4 K pulse tube cryocooler. Cryogenics 2001;47(7):491–6.
[9] Thummes G, Wang C, Heiden C. Small scale^{4}He liquefaction using a 4 K pulse tube cooler. Cryogenics 1998;38(3):337–42.
[10] Uhlig K. Dry dilution refrigerator with high cooling power. Adv Cryo Eng 2007;53B:1287.
[11] Lake Shore Cryotronics, Inc., 575 McCorkle Blvd, Westerville, OH 43082, USA.
[12] Rusby R, Fellmuth B, Engert J, Fogle W, Adams D, Pitre L, Durieux M. Realization of the ^{3}He melting pressure scale, PLTS 2000. J Low Temp Phys 2007;149(3/4):156–75.
[13] Huang Y, Chen G, Lai B, Wang S. p-H and T-S diagrams of ^{3}He from 0.2 K to 20 K. Cryogenics 2005;45(10/11):687–93.