



## Experimental investigation of low-pressure refrigerant mixtures for micro cryogenic coolers<sup>☆</sup>

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### ABSTRACT

Micro Cryogenic Coolers (MCCs) can achieve very small sizes and high efficiencies when operating with a refrigerant mixture, but micro-scale compressors have a limited pressure output. Four refrigerant mixtures were designed to operate between 0.4 MPa and 0.1 MPa, and tested in a MCC system both with and without pre-cooling. For comparison, two pure refrigerants were tested as well. Without pre-cooling, each mixture exhibited considerably lower cooling power than the design value. With pre-cooling, the mixtures exhibited unsteady cooling temperatures accompanied by flow pulsations after a period of time. The low cooling power, unsteady temperatures, and time required for the pulsations to occur are analyzed in terms of composition change due to liquid hold-up in the annular and intermittent flow regimes.

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### 1. Introduction

MCCs are gaining attention because their small volume, low noise, fast response, and portability make them attractive alternatives to conventional cryocoolers [1]. In the field of MCCs, those operating with a Joule–Thomson (J–T) cycle have the potential to be 10× smaller than Stirling MCCs, and 10× more efficient than thermoelectric coolers [2]. Refrigerant mixtures enable both benefits. Mixtures are designed to have a large glide between the bubble point and the dew point, such that the refrigerant in the heat exchanger of the J–T cooler is 2-phase flow. The 2-phase flow enhances heat transfer between the high-pressure and low-pressure streams, allowing the size of heat exchangers to be reduced. Mixtures are also designed to have a high specific cooling power, resulting in high cooling system efficiency [3]. Previous refrigerant mixtures typically use driving pressures of around 2 MPa [4–6], with some as low as 1.6 MPa [7]. However, to achieve a micro-system, one must use a micro-compressor, which may be limited to 0.4 MPa. Previous work from our group has shown that mixtures driven by 0.6 MPa can be effective at cooling to 177 K [8].

A number of methods have been proposed for choosing the components of a refrigerant mixture. These methods have been reviewed in Venkatarathnam's monograph on cryogenic mixed refrigerant processes [9]. The original patents of Alfeev et al. [10]

and Boiarski et al. [11] chose compositions based on experimental observations. In a patented by Little [12], refrigerants compositions are chosen to ensure the constant-pressure lines corresponding to the high- and low-side pressures of the cooler be parallel on a temperature–enthalpy diagram. Radebaugh showed that the specific cooling power of a refrigerant mixture in a J–T cooler with an ideal heat exchanger is given by the isothermal enthalpy difference between the high pressure and low pressure streams  $(\Delta h|_T)_{\min}$  which represents a function that can be optimized by varying the component compositions [13]. Another method, patented by Alexeev and Quack [14], finds compositions that maximize the specific refrigeration effect  $(\Delta h_{\min})$ , in a manner similar to Radebaugh [13]. A third method, reported by Gong et al. [15], maximizes the Carnot efficiency ( $\eta$ ) of the refrigerator, which is given as:

$$\eta = \frac{\Delta h_{\min}}{\text{specific compressor work}} \left( \frac{T_0}{T} - 1 \right) \quad (1)$$

This method is conceptually similar to optimizing the coefficient of performance (COP), as discussed in [13]. Mixtures designed to maximize the specific cooling effect will have some components entering the warm end of the heat exchanger in a liquid phase. Boiarski referred to these as liquid refrigerant supply (LRS) systems, and differentiated them from gas refrigerant supply (GRS) systems in which all components of the mixture are in a single gas phase as they enter the cryostat [16]. GRS systems are simpler to build, but have lower specific cooling power.

One issue that arises when using refrigerant mixtures is the consistency of the composition. It has been shown that the composition of the mixture changes at different locations within the

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cryocooler, with the cold-end favoring lighter (low boiling-point) components [17]. When the refrigerant is in 2-phase flow, the liquid phase is a different composition than the vapor phase. Depending on the 2-phase flow regime, this can separate the components in a cooler. In microchannel 2-phase flow, the regimes most often encountered are annular and intermittent [18]. These can prove problematic: in annular flow the high liquid hold-up causes drastic composition changes, and in intermittent flow the varying flow-rates can cause oscillations in temperatures. We face a dilemma between the desire of reaching high cooling capacity at low pressure, on the one hand, and the desire for compactness (MEMS construction). The compactness restricts the thermodynamic feasibility to reach high cooling capacity at low compression ratios, due to the fluid flow regimes encountered at that size scale.

We designed four hydrocarbon mixtures to operate between a high pressure of 0.4 MPa and a low pressure of 0.1 MPa, designed for the temperature range 140–300 K (Mix 1), 140–275 K (Mix 2), 160–300 K (Mix 3), and 200–300 K (Mix 4). Each of the mixtures is of the LRS type. These mixtures were used in a MCC system with a fiber-base cooler and a miniature compressor, described in [8]. For comparison, the system is also run with two pure hydrocarbons: propane and isobutane. The pure hydrocarbons perform as expected. The cold-tip temperature of the isobutane went as the boiling point of isobutane at the low-side pressures. The low temperature of the cold-tip with propane was limited to 242 K, which is the temperature that balances the small cooling power of the vapor with the background heat loads of the MCC. When the propane lines were cooled to 275 K (below propane's boiling point at 0.6 MPa of 281.1 K), the MCC temperature dropped to the temperature associated with the low-pressure boiling point of propane. In this situation, as with that of isobutane, the MCC acts as one would expect for a vapor-compression refrigerator.

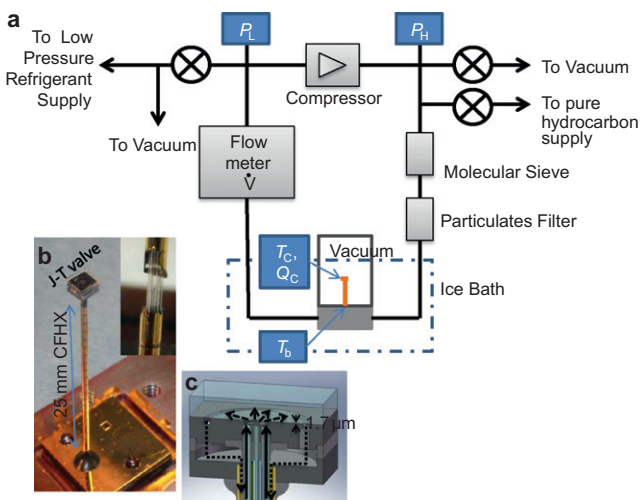
However, the mixtures do not cool according to their design. Without pre-cooling, the MCC running the mixed refrigerant experiences steady flow and cooling powers that are much lower than the designed values. With pre-cooling, after a period of time, the flow experiences pulsations. The low cooling power and time required for the transition to pulsating flow are analyzed in terms of composition change due to liquid hold-up in annular and intermittent flow regimes, as discussed in Section 5 of this article.

2. Materials and methods

This study considered two pure hydrocarbon refrigerants (propane and isobutane) and four custom mixtures. The single-component hydrocarbons had a purity rated by the vendor to be >99.9%. The compositions of the four mixtures are given in Table 1. These compositions were chosen to maximize  $(\Delta h/T)_{\min}$  over the temperature range of interest, and the optimization was carried out by the software NIST4 [19]. The refrigerants were provided by a commercial gas supplier and analyzed for composition, with actual composition listed in Table 1.

**Table 1**  
The design composition and manufactured composition of each of the four refrigerant mixtures studied.

|       | Design composition (mol fractions) | Actual composition (mol fractions) |       | Design composition (mol fractions) | Actual composition (mol fractions) |
|-------|------------------------------------|------------------------------------|-------|------------------------------------|------------------------------------|
| Mix1  | 34% Methane                        | 34.2% Methane                      | Mix 3 | 24% Methane                        | 24.1% Methane                      |
|       | 20% Ethane                         | 19.8% Ethane                       |       | 36% Ethylene                       | 36.0% Ethylene                     |
|       | 18% Ethylene                       | 15.2% Ethylene                     |       | 14% Propane                        | 14.0% Propane                      |
|       | 16% Isobutane                      | 14.9% Isobutane                    |       | 10% Isopentane                     | 10.0% Isopentane                   |
|       | 12% Isohexane                      | 15.9% Isohexane                    |       | 16% Pentane                        | 15.9% Pentane                      |
| Mix 2 | 34% Methane                        | 33.9% Methane                      | Mix4  | 8% Methane                         | 7.93% Methane                      |
|       | 22% Ethane                         | 22.0% Ethane                       |       | 46% Ethane                         | 45.88% Ethane                      |
|       | 20% Ethylene                       | 20.0% Ethylene                     |       | 14% Propane                        | 14.0% Propane                      |
|       | 12% Isobutane                      | 12.0% Isobutane                    |       | 4% Butane                          | 3.99% Butane                       |
|       | 12% Isopentane                     | 12.1% Isopentane                   |       | 28% Pentane                        | 28.2% Pentane                      |



**Fig. 1.** (a) The MCC test loop, which includes measurements of low-side pressure ( $P_L$ ), high-side pressure ( $P_H$ ), flowrate ( $\dot{V}$ ), MCC cold-end temperature ( $T_c$ ), heat applied to the MCC cold-end ( $Q_c$ ), and base temperature ( $T_b$ ). (b) Photograph of the MCC, and (c) cross-sectional drawing of the J-T valve, showing path of high-pressure refrigerant (solid line) as it expands (dashed line) to its low-pressure state (dotted line).

The test setup is shown in Fig. 1. This test method has been described elsewhere [8], but is briefly repeated here. The key components are a miniature oil-free compressor and micro cryogenic cooler. The compressor is formed by coupling a commercial piston oscillator with a pair of custom micro-fabricated check-valves. The MCC consists of a warm-end micro-coupler, a tubes-in-tube counter-flow heat exchanger (CFHX) formed by hollow-core glass fibers, and a microfabricated J-T valve with a restriction of 1.7  $\mu\text{m}$ , as described in [7]. One gram of 0.3 nm molecular sieve is included between the compressor and the MCC to remove any trace moisture contaminant and thereby reduce the likelihood of icing. Refrigerant is compressed to high pressure by the miniature compressor, passes through the molecular sieve and a 7  $\mu\text{m}$  particulates filter, and into the MCC, where it expands to low pressure, and through a mass flowmeter. With the mixtures, the low-side pressure was kept open to a supply tank regulated at 0.100 MPa. The high-side pressure is determined by the performance of the compressor, which is a function of the flow-rate and the compressibility of the refrigerant in question, but it is typically between 0.4 and 0.7 MPa.

With the pure hydrocarbons, the system was fed with an initial charge, and all of the valves were closed during operation. With the isobutane, the system was fed with refrigerant regulated at 0.35 MPa into the high side while the low side was still under vacuum. The propane was fed through the high-pressure side, with an initial equilibrium pressure of 0.47 MPa, which was sufficient to develop an operating pressure of 0.63 MPa on the high side with a pressure of 0.10 MPa on the low side.

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