

## **CRYOCOOLER CONTAMINATION STUDY**

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### **ABSTRACT**

Eliminating or reduction of contamination is essential for the success of cryocoolers as far as performance and cooler life is concerned. Contaminants in a cooler can be defined as any foreign material that results in the rubbing of mechanical parts or blockage of flow, which in turn reduces cooler life and / or degrades performance. In this paper, we will limit the scope of our study to gaseous and liquid contaminants, which may freeze and are detrimental to cooler performance. It was found that most of the contaminants in BEI's coolers consist, carbon dioxide, water, cleaning solvents (acetone and alcohol), nitrogen and oxygen. Five 0.5 watt coolers (B512) were used for the study. These coolers were baked out continuously at 71°C for one week. Gas analysis was performed on the cooler working gas every day during the one-week period. Outgassing curves have been constructed on the contaminants listed above. Based on the analysis, optimum bake-out time of the cooler components can be derived. Optimum purge and fill procedure will also be discussed in this paper.

### **INTRODUCTION**

Due to the presence of narrow passages in cryocoolers (clearance gaps and torturous paths in regenerators), any contaminant would tend to interfere with the gas dynamic or mechanism of the cooler operation, which in turn results in the degradation of performance or shortening of cooler life. In this paper, we will concentrate on the study of gaseous and liquid contaminants. Using a Varian 3800 Gas Chromatography (GC) Analyzer (which is connected to a Dell Workstation), the following contaminants were identified and tabulated in Table 1. The Varian GC Analyzer uses a Thermal Conductivity Detector (TCD) and a Flame Ionization Detector (FID) to analyze contaminants. TCD detects gases like nitrogen, oxygen, carbon dioxide, and water vapor. FID on the other hand detects hydrocarbons. Also included in Table 1 are the retention time of various contaminants.

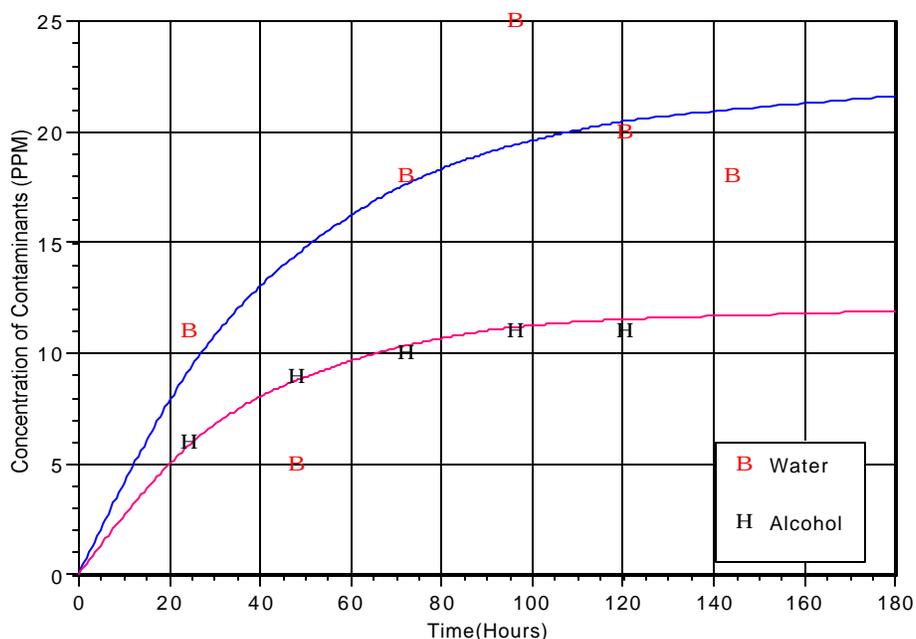
**Table 1.** List of contaminants with retention time.

Contaminants	Detector	Retention Time (min)
Nitrogen / Oxygen	TCD	0.48
Carbon Dioxide	TCD	1.78
Water	TCD	5.5
Methane	FID	0.51
Acetone	FID	0.83
Alcohol	FID	1.22

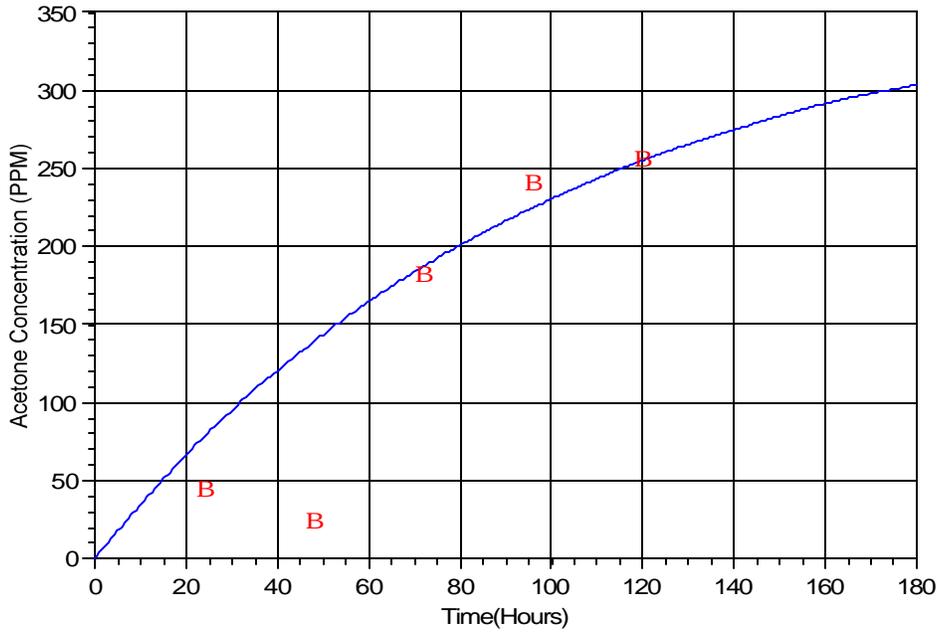
The Varian GC Analyzer was calibrated using known amount of nitrogen, oxygen, carbon dioxide and methane. Sample of water vapor, acetone and alcohol were prepared at BEL. Acetone and alcohol are used as cleaning solvents, and the rest of the above gases are readily available in the atmosphere. A Hayesep column (N80/100) and a DB wax capillary (15m x 0.53 mm) were used in the GC analyzer. The column temperature was ramped up from 35°C to 140°C in five minutes and held at 140°C for another five minutes. The retention times of nitrogen and oxygen are too close to be distinguished.

### Outgassing Study

Five B512C coolers<sup>1-3</sup> were used for the contamination study for one week. The coolers were randomly selected from the production line. They were placed in an oven connected to the Varian GC Analyzer. The oven temperature was set at 71°C. Gas samples were injected into the GC Analyzer every 24 hours and the levels of contaminants recorded. Out of the five readings (from the five coolers) the maximum and minimum values were discarded and the average of the three medium values was recorded. Water, acetone, and alcohol tend to be absorbed onto the surfaces inside the cooler and outgas for an extended period of time showing a steady increase of concentration in the gas sample as shown in Figures 1 (water and alcohol) and 2 (acetone).



**Figure 1.** Concentration of water and alcohol as a function of time.



**Figure 2.** Acetone concentration as a function of time.

On the other hand, gases like nitrogen, oxygen and carbon dioxide do not get absorbed and outgassed, at least not substantially. The rate of increase in contaminant concentration ( $m$ ) was found to follow the following equation.

$$m = B \cdot T \cdot (1 - e^{-t/A}) \quad (1)$$

Where  $A$  and  $B$  are constants,  $T$  is the ambient bake-out temperature in degrees Celsius and  $t$  is time in hours. Values of  $A$  and  $B$  for water, alcohol and acetone can be found in Table 2. The outgassing rate ( $dm/dt$ ) can then be obtained by taking the derivative of Equation 1.

$$dm/dt = (B \cdot T/A) \cdot e^{-t/A} \quad (2)$$

**Table 2.** Outgassing constants

	A	B
Water	45	0.31
Alcohol	36.4	0.17
Acetone	100	5.13

Note that within the temperature range of this study, the temperature dependence of outgassing appears to be linear. Care should be taken in applying these equations at temperatures above 71°C. Figure 3 shows the outgassing rate of water and alcohol and Figure 4 shows the outgassing rate of acetone. Extensive bake out of cooler components and hot purge of coolers are essential to prevent contamination. Data like that of Figure 1

and  $\lambda$  help to determine if the bake out and not purge processes are adequate. Based on experience, the maximum amount of each contaminant allowable in a cooler was determined. This value must not be exceeded during the entire life of the cooler. Looking at Figure 1 and 2, one realizes that the rise in contaminant concentration tends to peak out after 200 hours or so, which can then be considered as the end-of-life value. This end-of-life value, therefore, must not exceed the maximum allowable contaminant concentration, obtained from experience.

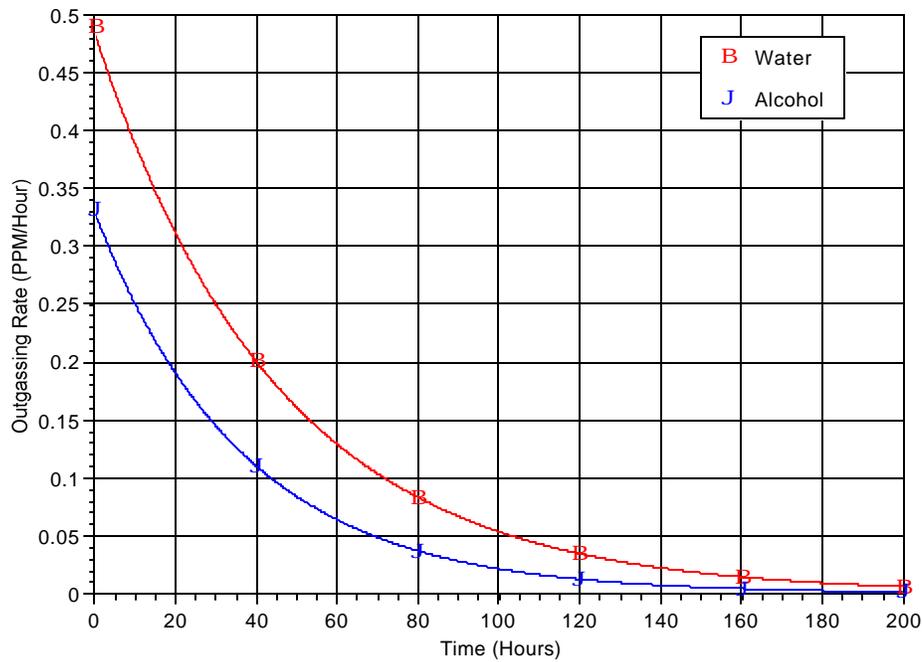


Figure 3. Outgassing rates of water and alcohol.

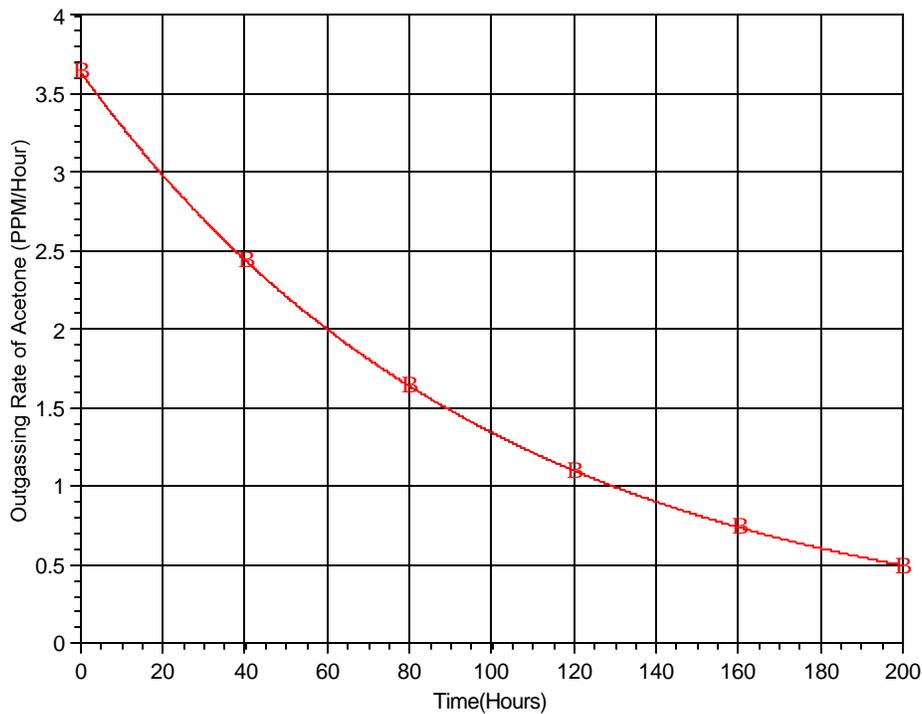
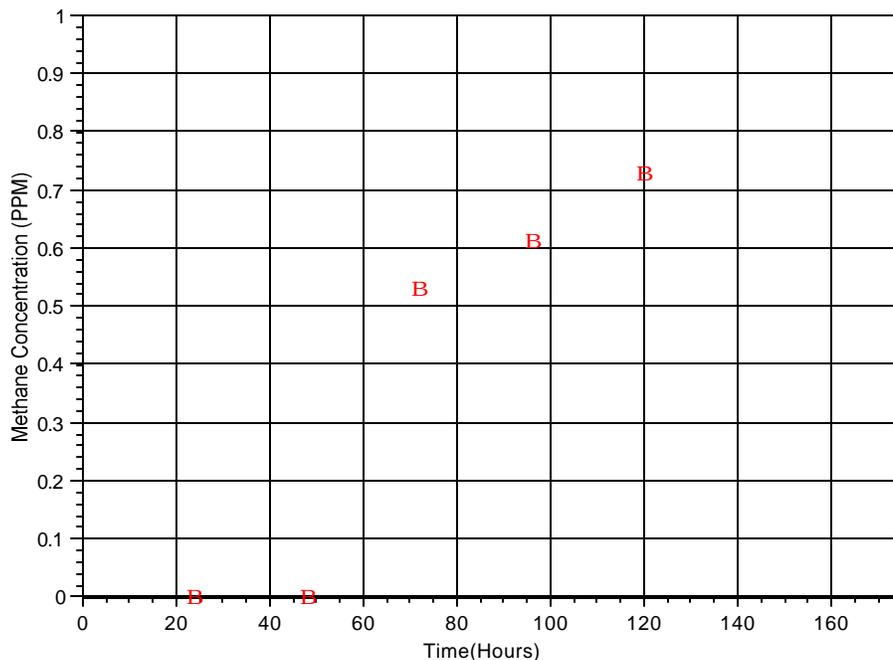


Figure 4. Outgassing rate of acetone.

However, testing each cooler for 200 hours is a time-consuming process. To shorten test time, one can test the cooler for a couple of hours and extrapolate the result (using Equation 1) to the end-of-life value at 200 hours. This end-of-life value can then be used to fine-tune the bake-out and hot-purge processes to optimize the process.

One interesting observation was the build-up of an unidentified contaminant as the cooler ages. This contaminant has a retention time of approximately 0.5 minutes and it shows up in the Flame Ionization Detector of the GC Analyzer. From the short retention time, one can speculate that the contaminant is a light hydrocarbon. By characterizing various light hydrogen carbons it was determined that the contaminant was methane. The presence of methane together with the disappearance of alcohol in other BEI coolers with long run-time, tends to suggest that alcohol in the coolers decomposes into methane. Figure 5 shows the accumulation of methane in the coolers under study, however the level is quite low (below 1 PPM) after one week. Although 90% of outgassing tends to be completed in 200 hours, the decomposition of alcohol into methane tends to take much longer, as supported by the trace amount of methane detected compared to the relatively high concentration of alcohol after 200 hours. Thus to limit the build up of methane, one must limit the initial concentration of the alcohol in coolers.

Outgassing is a function of surface area on which the contaminants are being absorbed. The findings in this paper can also be applied to other models of BEI coolers<sup>1-8</sup> via scaling if the internal surface area can be estimated.



**Figure 5.** Detection of methane in coolers.

## Room Temperature Purge and Fill

As mentioned earlier, gases like nitrogen, oxygen, carbon dioxide do not tend to outgas in the cooler. However, since these gases are readily available in the atmosphere, they must be carefully purged out of the coolers to prevent contamination. Thus, before the coolers are charged with helium gas, the coolers are generally purged with helium gas. The optimum number of purges must be determined so that the least number of purges or the shortest purge process would guarantee acceptable levels of these gases.

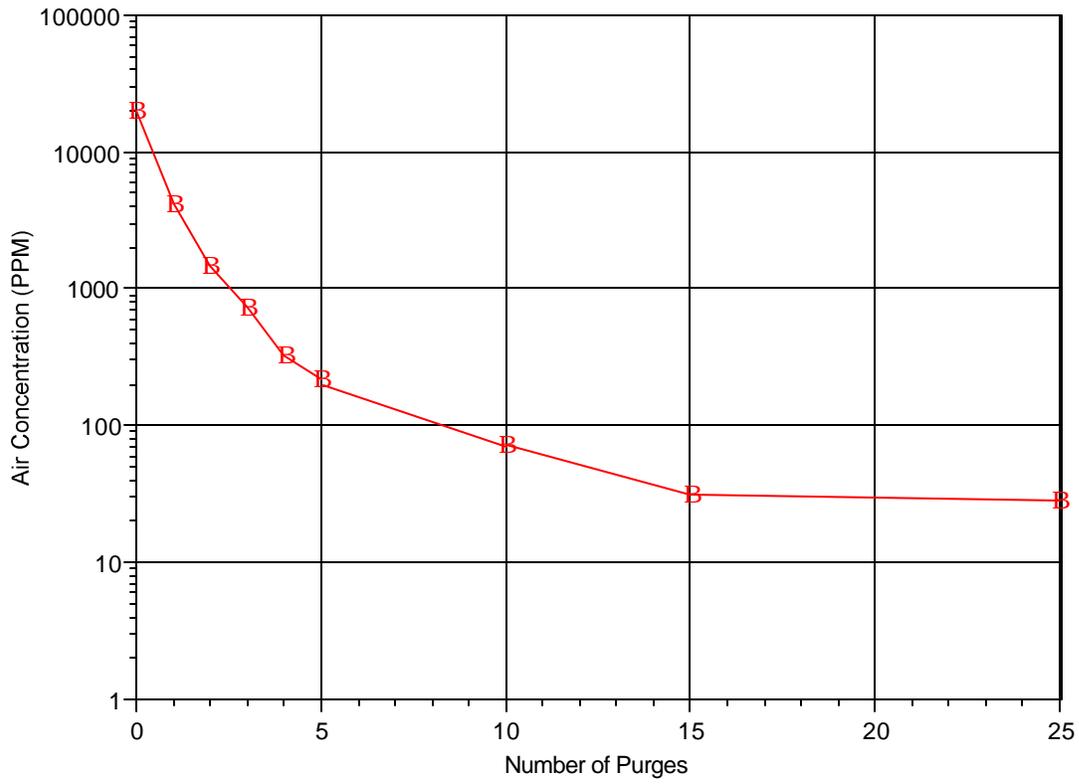
A separate study was conducted to determine this optimum value. A cooler was selected for this purpose. The expander was separated from the compressor and the cooler was allowed to come to equilibrium with the ambient air overnight. The cooler was then reintegrated and the appropriate number of purges applied. The cooler was then kept in an oven for two hours at 71°C before injecting the working gas into the GC Analyzer for evaluation. The process was then repeated with a different purge number. Since the retention times of nitrogen and oxygen are not distinguishable, we will group the gases together and call them “air”.

Figure 6 is a plot of air concentration vs. number of purges at half-a-minute intervals. After about 15 purges, the decrease in air concentration levels off at a value (approx. 30 PPM) which is way below the acceptable level of 100 PPM (Ref. 9). When conducting the purge and fill test, it became quite evident that the higher the number of purges and the longer the duration of the purge process the cleaner the working gas became. The test was thus repeated with different combinations of purge duration and number of purges. It was found that when plotting the air concentration vs. product of purge number and total purge duration, the curve follows that of an exponential decay, as shown in Figure 7. Within the limits of this study (maximum number of purges = 20, minimum of purges = 4, maximum total purge time = 10 minutes, minimum total purge time = 1 minute) the data tend to fall on this exponential curve and are quite insensitive to the combination of purge number and total purge duration. This information is quite useful if one wants to shorten the purge process. In order to obtain the same result of a 10 minute process, with 20 purges at a 30 second interval ( $20 \times 10 \text{ min} = 200$ ), one can adopt a 7 minute process with 28 purge at a 15 second interval ( $28 \times 7 \text{ min}$  equals to 196).

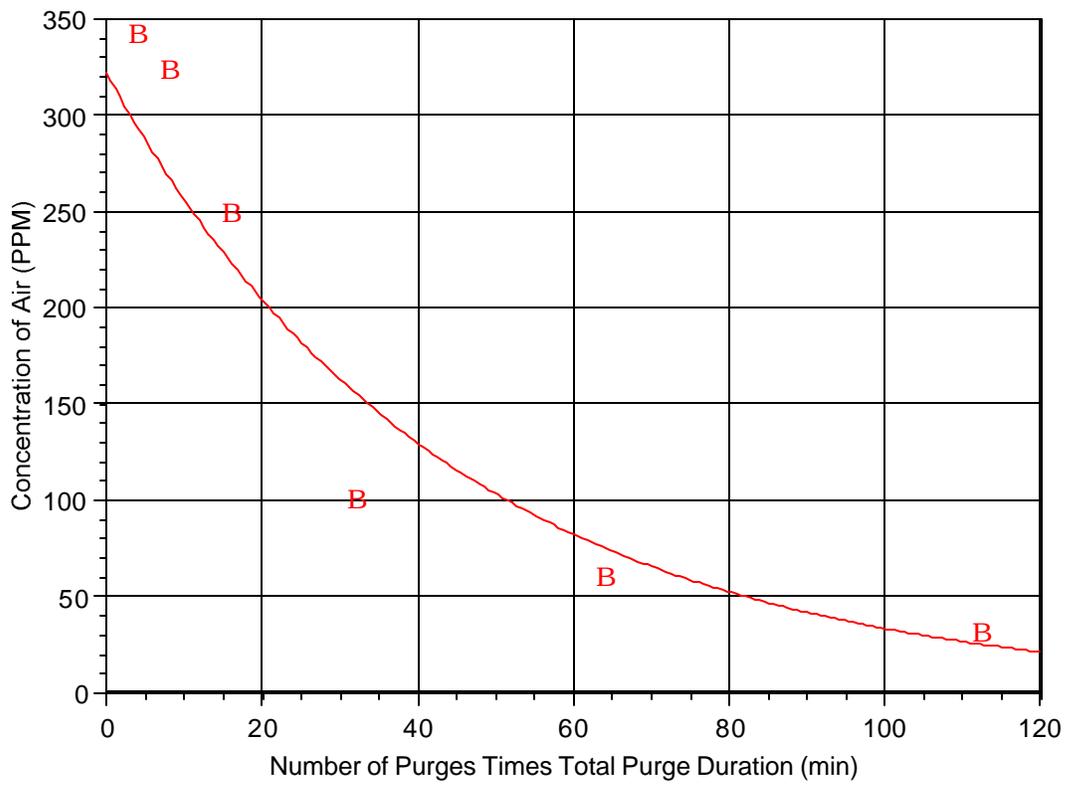
The shortest purge interval used in this study was 15 second. It is not recommended to decrease the purge interval to below 15 seconds for the air inside the cooler (especially in the regenerator) will not have enough time to equilibrate with the purge gas.

## CONCLUSIONS

The outgassing characteristics within the BEI B512C coolers have been studied. Outgassing equations for water, alcohol, and acetone have been suggested. The dependence of outgassing on ambient temperature should be further investigated. An optimized purge and fill process has also been proposed to eliminate air and moisture in coolers.



**Figure 6.** Air concentration vs. number of purges.



**Figure 7.** Study of purge duration and number of purges.

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