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The Coefficient of Thermal Expansion of Fluorinert as a Function of Volume Percent Absorbed Air

By J. M. Reilly

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C. W. Bean, Project Leader

Project Team:
F. N. Larsen
J. M. Reilly

Technical Communications **Bendix** **Kansas City Division**

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The relationship between the coefficient of volumetric thermal expansion of liquid Fluorinert FC-86 and the volume percent of absorbed air was examined experimentally. A special test apparatus was built for this purpose. A floating liquid seal was used to isolate the Fluorinert sample from the atmosphere. This prevented gas absorption during thermal cycling yet allowed the sample to expand and contract freely during testing. It was found that the coefficient of thermal expansion is not influenced by the percentage of absorbed air.

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The Bendix Corporation
Kansas City Division
P. O. Box 1159
Kansas City, Missouri 64141

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SUMMARY

The coefficient of volumetric thermal expansion (CVTE) of Fluorinert FC-86 (with respect to the volume percentage of absorbed air) was examined. The experiments conducted provided information to support a project sponsored by Lawrence Livermore National Laboratory (LLNL), Livermore, CA. A test method was developed to determine the CVTE of Fluorinert samples with various percentages of absorbed air contents. Since Fluorinert absorbs air quickly, a test apparatus was designed and built that allowed the Fluorinert to expand and contract freely during thermal cycling yet kept the Fluorinert sealed from the atmosphere. The CVTEs from 12 to 40°C of Fluorinert samples with 1, 9, 20, and 32 volume percentages of absorbed air were measured. All values were within ±4 percent of $17.7 \times 10^{-4}/°C$. The 4 percent spread was less than the experimental error; therefore, it was concluded that the CVTE of Fluorinert is not influenced by the volume percent of absorbed air.

DISCUSSION

SCOPE AND PURPOSE

The cost and procedure for filling a transfer mechanism depends heavily upon whether or not the volume percentage of absorbed air in Fluorinert FC-86 affects its coefficient of volumetric thermal expansion (CVTE). The testing described was conducted to supply Lawrence Livermore National Laboratory (LLNL) with another source of CVTE versus percentage of absorbed air data, providing them with a basis for choosing a procedure for filling the transfer mechanism.

ACTIVITY

Fluorinert is a liquid manufactured by the 3M Corporation. It has the property of being able to absorb large quantities of air, higher than 40 percent by volume at standard temperature/pressure. Because of some of its applications, it was necessary to measure experimentally the coefficient of volumetric thermal expansion (CVTE) as a function of the volume percentage of absorbed air.

Theory

The volumetric expansion of a liquid subject to temperature and pressure changes is given by the equation

$$\Delta V = V_o \beta \Delta T - V_o B \Delta P \ , \qquad (1)$$

where

β = CVTE and

B = isothermal bulk modulus.

Solving for β yields

$$\beta = \frac{\Delta V + V_o B \Delta P}{V_o \Delta T} \ . \qquad (2)$$

An isobaric process presents the most convenient method of measuring β, reducing Equation 2 to

$$\beta = \frac{\Delta V}{V_o \Delta T} \qquad (3)$$

and no corrections are needed for pressure variations.

Approach

Degassed Fluorinert, when exposed to the atmosphere, tends to absorb air quickly. This presents the problem of how to allow the Fluorinert to expand and contract freely while keeping it isolated from the atmosphere. The approach used was to seal the Fluorinert sample with a floating liquid seal. Specifically, a less dense liquid, insoluble in Fluorinert, was allowed to float on the Fluorinert during thermal cycling. This created a liquid piston that provided a constant pressure environment to the sample (atmospheric pressure plus the pressure resulting from the weight of the floating liquid), and also provided adequate sealing so that air could not be absorbed during testing. The liquid barrier, however, would allow degassing of the Fluorinert at higher temperatures. (The floating liquid is analogous to a diode.) Since the solubility of air in Fluorinert decreases with increasing temperature, all data were taken after the Fluorinert was exposed to the highest test temperature. This ensured that any degassing took place prior to recording data, while the floating barrier assured that no additional air would be absorbed during testing. It also enabled all data on a given Fluorinert sample to be taken at a constant volume percentage of absorbed air and at a constant pressure.

Apparatus

A custom-built 50 mL jacketed buret was used to measure volume and volume changes of the Fluorinert (Figure 1). A heating/cooling medium of soap water was circulated through the jacket by a Haake Model FE heater/circulator. Cooling was provided by a chilled ethylene glycol water solution circulated through the heater/circulator cooling coil. Temperature measurements of the Fluorinert were made with a 5 mil K type thermocouple interfaced to a Keithly 191 digital multi-meter through an Omega ice point reference. All volume percentages of dissolved air measurements were made with an Aire-ometer, manufactured by Seaton and Wilson, Incorporated.

Procedure

Initial Preparation

Using a 25 mL buret, the 50 mL jacketed buret was calibrated to determine the volume of "dead space" between the stopcock and the 50 mL graduation. The dead space was 3.5 mL. The volume of liquid in the 50 mL buret was obtained from the meniscus reading by the equation

$$\begin{aligned} \mathrm{Vol} &= 3.5 + (50 - \mathrm{reading}) \\ &= 53.5 + \mathrm{reading}\ . \end{aligned} \tag{4}$$

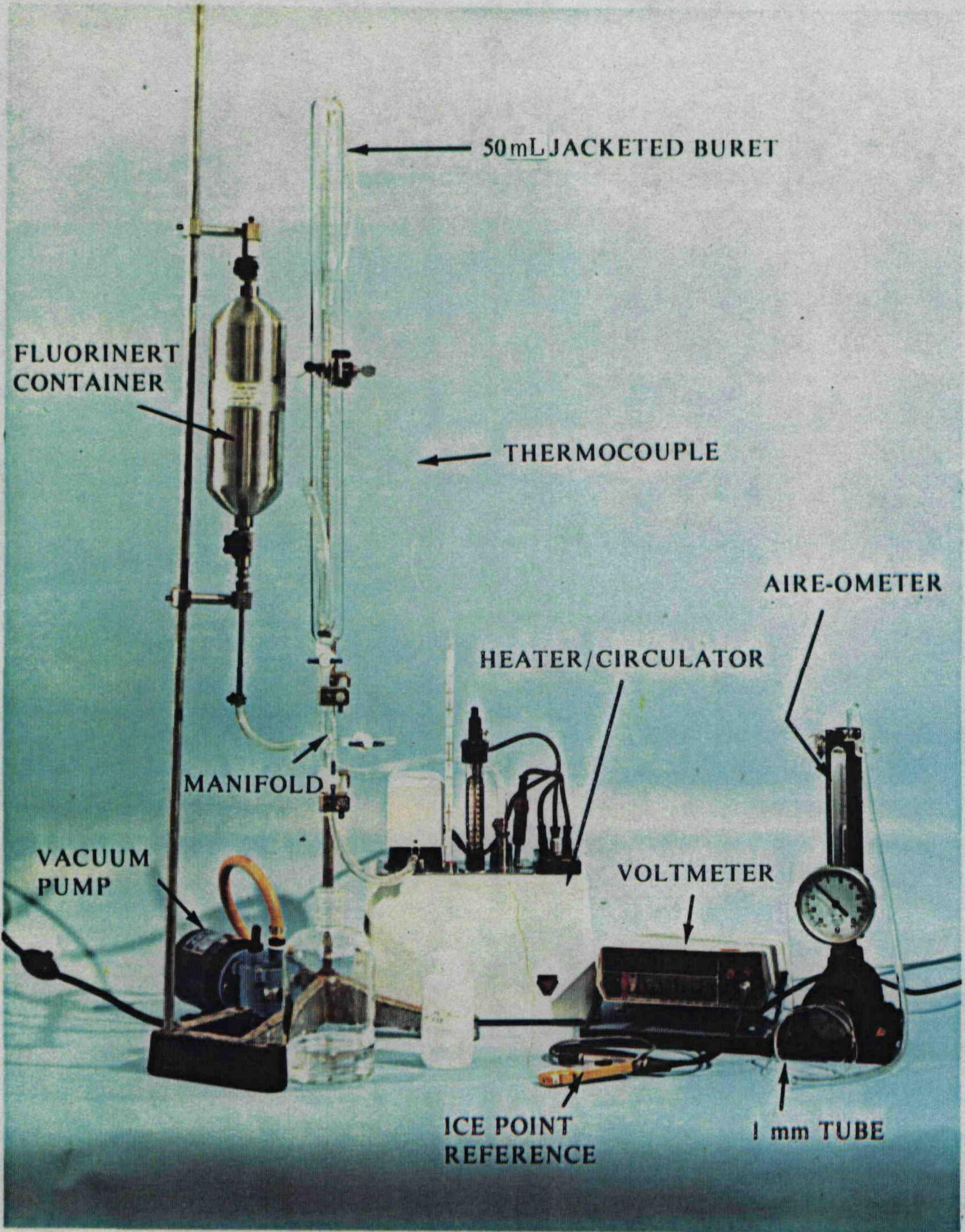

Figure 1. Thermal Expansion and Degassing Apparatus

Sample Loading

The 50 mL jacketed buret first was loaded with 25 mL of Fluorinert. Two different loading techniques were used; one for saturated Fluorinert and one for degassed Fluorinert.

Saturated Fluorinert. Loading the buret with saturated Fluorinert consisted of pouring the Fluorinert into the top of the buret until the meniscus was above the 28.5 mL graduation. Ethylene glycol (5 mL) was added to form the floating seal. Fluorinert then was drained from the bottom of the buret until the Fluorinert/ethylene glycol interface reached the 28.5 mL graduation, leaving 25 mL of Fluorinert in the buret with the 5 mL seal.

Degassed Fluorinert. A vacuum transfer technique was used to load the buret with degassed Fluorinert. A vacuum line was attached to the top of the buret. The vacuum pump was started and left running, pulling a vacuum of approximately 600 mmHg. The stopcock between the buret and the manifold was opened. The valve on the Fluorinert bottle then was used to meter the fluid into the buret. Since the Fluorinert would boil and degas because of the vacuum in the buret, the fluid was metered slowly. When an adequate amount of Fluorinert had entered the buret, the stopcock was closed. The vacuum line was disconnected from the top of the buret and 5 mL of ethylene glycol was added immediately. The Fluorinert then was drained until 25 mL was left for testing.

Fluorinert samples with an intermediate volume percentage of absorbed air were prepared by loading the buret with a degassed sample of Fluorinert sealed with ethylene glycol. A small diameter (approximately 1 mm) plastic tube fixed to the Aire-ometer was lowered in the burret until it reached the bottom. The Aire-ometer was used to bubble air through the Fluorinert until the desired volume percentage of absorbed air was reached. The Fluorinert then was drained until 25 mL was left for testing.

Temperature Cycling

The thermocouple was lowered into the Fluorinert and the Haake circulator/heater was set to 40°C. After the system reached thermal equilibrium and any degassing had occurred, the Fluorinert level and temperature were recorded, and the circulator/heater was set to 12°C (the capacity of the cooling system). When the system stabilized, the Fluorinert level and temperature were recorded again. The sample was allowed to return to room temperature, and the Fluorinert level and temperature were recorded for a third time. Thus, all three sets of readings were taken with the same quantity of absorbed air.

Volume Percent of Absorbed Air Measurement

The volume percent of absorbed air was measured after the sample had returned to room temperature. The end of the small diameter plastic tube was lowered through the ethylene glycol seal into the Fluorinert. A sample carefully was drawn out into the Aire-ometer and the percent of absorbed air was determined according to the manufacturer's instructions. The reported value was derived by averaging two such samples.

Data Reduction

The CVTE was determined using the equation

$$\beta = \frac{V_{T_H} - V_{T_L}}{V_{T_o} - \beta'(V_{T_o})(T_o - 27^\circ C) \quad T_H - T_L} \tag{5}$$

where

β = corrected CVTE with the reference volume at 27°C,

T_H = high temperature,

T_L = low temperature, and

T_o = room temperature,

and

$$\beta' = \frac{V_{T_H} - V_{T_L}}{V_o(T_H - T_L)} \tag{6}$$

where

β´ = CVTE with the reference volume at room temperature.

Results

The CVTE and the absorbed air percentages for six Fluorinert samples are listed in Table 1. Temperature and volume data also are presented. Figure 2 depicts the CVTE as a function of absorbed air percent. The average value of the CVTE for the six samples is $17.7 \times 10^{-4}/^\circ C$.

If the volumetric expansion is assumed to be constant, the high and low values from samples 2 and 5 are in error by -2.3 and 4.0 percent, respectively, which is less than the resolution error of the instrument. With the apparatus used, the CVTE

Table 1. CVTE and Absorbed Air Percentages for Six Fluorinert Samples

Sample Number	Absorbed Air (Volume Percent)	Fluorinert Temperature (°C)	Fluorinert Level (mL)	B_{Vo} at 27°C
1	32	39.9	25.00	17.6×10^{-4}
		12.0	23.80	
		26.7	24.40	
2	1	40.9	25.60	17.3×10^{-4}
		13.2	24.40	
		28.2	25.05	
3	1	40.7	25.60	17.5×10^{-4}
		13.3	24.40	
		29.1	25.10	
4	31	40.8	25.50	18.1×10^{-4}
		13.1	24.25	
		28.0	24.95	
5	20	40.7	25.50	18.4×10^{-4}
		13.4	24.25	
		68.2	24.90	
6	9	40.0	25.60	17.4×10^{-4}
		13.6	24.45	
		27.1	25.00	

appears to be independent of the absorbed air percentage and equal to 17.7×10^{-4}/°C.

Error Analysis

The basic equation for calculating β is:

$$\beta = \frac{V_{T_H} - V_{T_L}}{V_o(T_H - T_L)} \; . \qquad (7)$$

Since V_{T_H} and V_{T_L} were measured with the same buret, and T_H and T_L were measured with the same thermocouple system, the error analysis is simplified and performed on the equation

$$\beta = \frac{\Delta V}{V_o \Delta T} \; . \qquad (8)$$

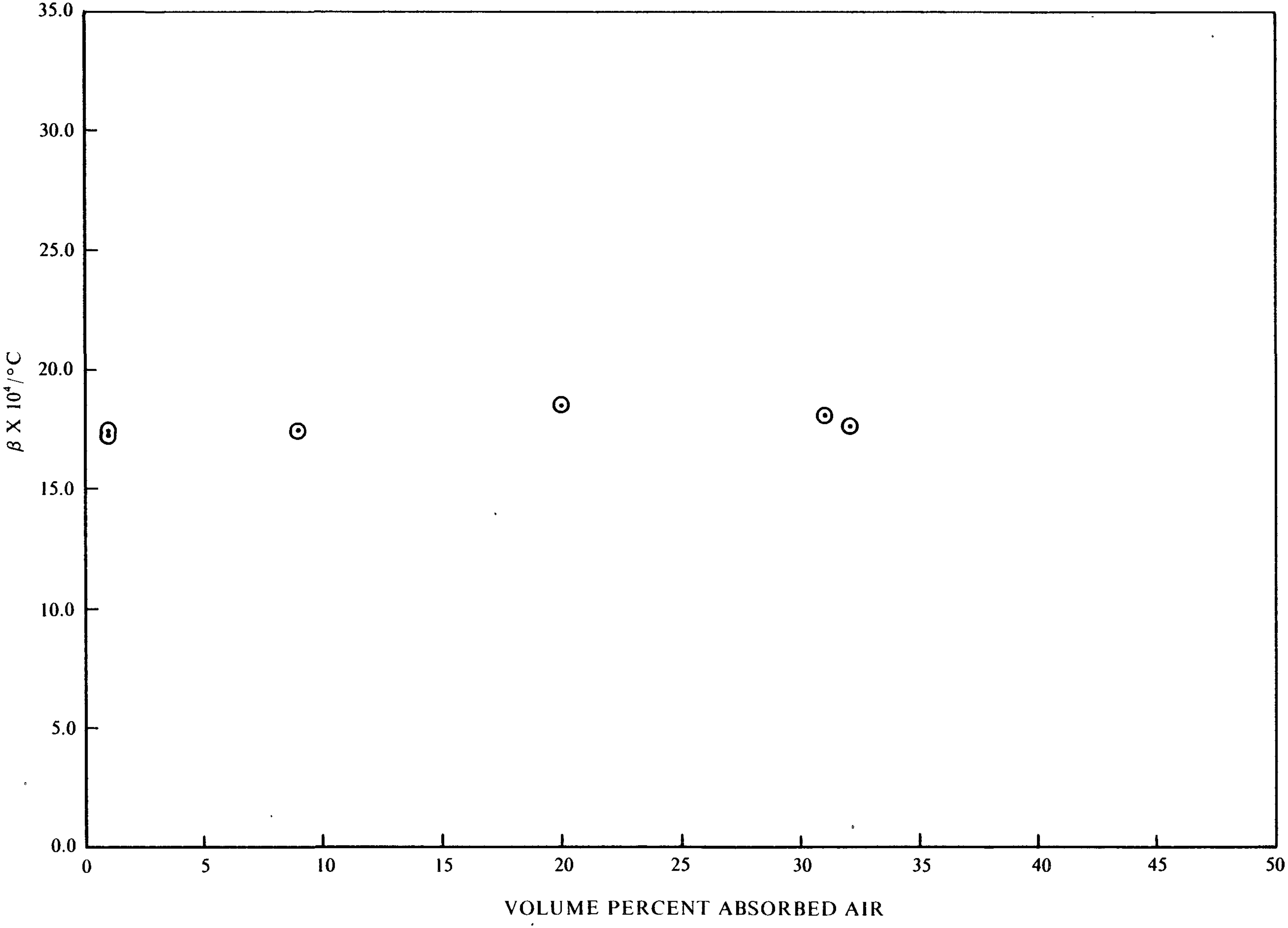

Figure 2. CVTE as a Function of Absorbed Air Percentages

The sensitivity equation becomes

$$\varepsilon_{\beta} = \varepsilon_{\Delta V} - \varepsilon_{V_o} - \varepsilon_{\Delta T} \, . \tag{9}$$

The sensitivity factors (seen to be ±1) were derived from the equation

$$S_X = \frac{\frac{\partial \beta}{\partial X} X}{\beta} \tag{10}$$

with S_X being the sensitivity of the error in β, $\varepsilon\beta$, to an error in X, ε_X.

Since all errors are independent, the error in β will be related to the errors in the measurements by the equation

$$\varepsilon_{\beta} = \sqrt{\varepsilon_{\Delta V}{}^2 + \varepsilon_{V_o}{}^2 + \varepsilon_{\Delta T}{}^2} \, . \tag{11}$$

Error in ΔV. The buret used to measure volume (ΔV) had a capacity of 50 mL with 0.1 mL graduations. Readability and precision errors were assumed to be half of the smallest division. The error in ΔV was estimated to be

$$\varepsilon_{\Delta V} = \sqrt{4(0.05)^2} = 0.1 \text{ mL} \, . \tag{12}$$

The factor of four comes from two readability errors plus two precision errors. Typically, ΔV was 1.2 mL giving

$$\varepsilon_{\Delta V} = \frac{0.1}{1.2} = 8.3 \text{ percent} \, . \tag{13}$$

Error in V. The room temperature volume (V) was measured with the same 50 mL buret. When the reading was taken, there was a 0.05 mL readability error and a 0.05 mL precision error. Also, there were additional errors in V as a result of the measuring of the "dead space" between the stopcock and the 50 mL graduation. This volume was measured by filling the dead space with liquid from a 25 mL buret also with 0.1 mL graduations. The error introduced from the calculation of the dead space involves two readability errors and two precision errors from the 25 mL buret:

$$\varepsilon_{V_o} = \sqrt{6(0.05)^2} = 0.12 \text{ mL} \, . \tag{14}$$

Typically, V was 25 mL giving

$$\varepsilon_{V_o} = \frac{0.12}{25} = 0.48 \text{ percent} . \tag{15}$$

Error in T. It was assumed that the error introduced when reading temperature was 0.5°C. Since two readings were involved for each ΔT,

$$\varepsilon_{\Delta T} = \sqrt{2(0.05)^2} = 0.71°\text{C} . \tag{16}$$

Typically, ΔT was 28°C giving

$$\varepsilon_{\Delta T} = \frac{0.71}{28} = 2.5 \text{ percent} . \tag{17}$$

Experimental Error. The error (β) in the final result becomes

$$\varepsilon_{\beta} = \sqrt{\left(\frac{8.3}{100.0}\right)^2 + \left(\frac{0.48}{100.0}\right)^2 + \left(\frac{2.5}{100.0}\right)^2} = 8.7 \text{ percent}. \tag{18}$$

Thus, the experimental reading is within ±9 percent of the actual value more than 95 percent of the time.

ACCOMPLISHMENTS

An apparatus was built for testing the coefficient of volumetric thermal expansion (CVTE) of liquid samples. A method was found for sealing liquid samples that must be isolated from the atmosphere during testing but also must retain the ability to expand and contract freely. The apparatus was used to find the CVTE of Fluorinert FC-86 containing various percentages of absorbed air.