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SAND2007-4470
Unlimited Release
Printed July 2007

Method for Creating Gas Standards from Liquid HFE-7100 and FC-72

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Abstract

HFE-7100 and FC-72 fluorinert are two fluids used during weapon component manufacturing. HFE-7100 is a solvent used in the cleaning of parts, and FC-72 is the blowing agent of a polymeric removable foam. The presence of either FC-72 or HFE-7100 gas in weapon components can provide valuable information as to the stability of the materials. Therefore, gas standards are needed so HFE-7100 and FC-72 gas concentrations can be accurately measured. There is no current established procedure for generating gas standards of either HFE-7100 or FC-72. This report outlines the development of a method to generate gas standards ranging in concentration from 0.1 ppm to 10% by volume. These standards were then run on a Jeol GC-Mate II mass spectrometer and analyzed to produce calibration curves. We present a manifold design that accurately generates gas standards of HFE-7100 and FC-72 and a procedure that allows the amount of each to be determined.

TABLE OF CONTENTS

FIGURES

TABLES

ACRONYMS AND ABBREVIATIONS

Abbreviation	Explanation
Ar	argon gas
Cryo	cryofocusing
FC-72	fluorinert electronic liquid
GC	gas chromatography
HFE-7100	Novec 7100 engineering fluid
LEP	lifetime extension program
mmHg	millimeters of mercury (1 mmHg = 1 torr)
MS	mass spectrometry
m/z	mass to charge ratio (Daltons)
N_2	nitrogen gas
ppmv	parts per million by volume
RIC	reconstructed ion chromatograph
SS	Swagelok®
TIC	total ion chromatograph
torr	1 torr = 1 mmHg

INTRODUCTION

This work was performed as part of the weapon lifetime extension program (LEP). HFE-7100[a] and FC-72[b] fluorinert are two fluids used during weapon component manufacturing. HFE-7100 is a solvent used in the cleaning of parts, and FC-72 is the blowing agent of a polymeric removable foam. During the foam-blowing process, the liquid FC-72 volatizes into a gas creating the air spaces of the foam. Over time, the FC-72 vapor exchanges with the surrounding gaseous environment. Accurate determination of FC-72 concentration in headspace gases provides valuable information about this diffusive process and the environment within which weapon components reside. Therefore, gas standards are needed so FC-72 and HFE-7100 gas concentrations can be measured.

Neither HFE-7100 nor FC-72 can be commercially purchased as gas standards. In addition, there currently exists no established procedure for generating gas standards from either of these fluids. Therefore, this report outlines the development of a method for generating HFE-7100 and FC-72 gas standards ranging in concentration from 0.1 ppm through 10% by volume. Standards were made in groups of three representing high, medium, and low concentrations. The highest concentration set contained standards at 1%, 3%, and 10% by volume. The medium concentration set contained standards at 1 ppmv, 10 ppmv, and 100 ppmv. The lowest concentration set contained standards of 0.1 ppmv, 1 ppmv, and 10 ppmv. The medium and low concentration sets were analyzed using a Jeol GC-Mate II mass spectrometer to develop calibration curves. The linearity of these curves was used as a quality check for the standard making process.

This report provides a description of the manifold design with instructions for use, information on the gas analysis by a Jeol GC-Mate II mass spectrometer, and a discussion of results.

EXPERIMENTAL DESIGN

MANIFOLD DESIGN

As discussed in the introduction, the purpose of this project was to generate gas standards from HFE-7100 and FC-72 fluids. The design we developed accomplished this goal by capitalizing on the high vapor pressure of HFE-7100 (202 mmHg at 25°C) and FC-72 (232 mmHg at 20°C). Since evaporation rates are proportional to vapor pressure, HFE-7100 and FC-72 vapor will quickly fill the headspace of a container to their respective pressures. The headspace gas can then be collected and diluted with nitrogen, argon, or other gas to generate a standard of a specific concentration. A general schematic of the system is shown in **Figure 1**. Pictures of the system are shown in **Figures 2** and **3**.

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[a] HFE-7100 is composed of methyl nonafluoroisobutyl ether (CAS 163702-08-7) and methyl nonafluorobutyl ether (CAS 163702-07-6).
[b] FC-72 is composed of perfluoro compounds (CAS 86508-42-1)

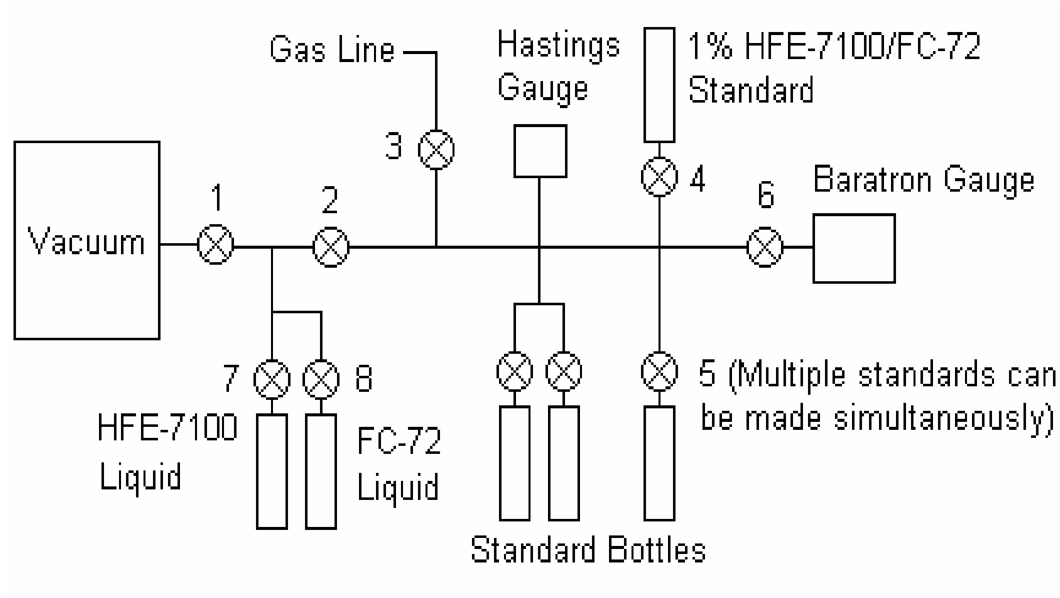

Figure 1: Manifold Schematic

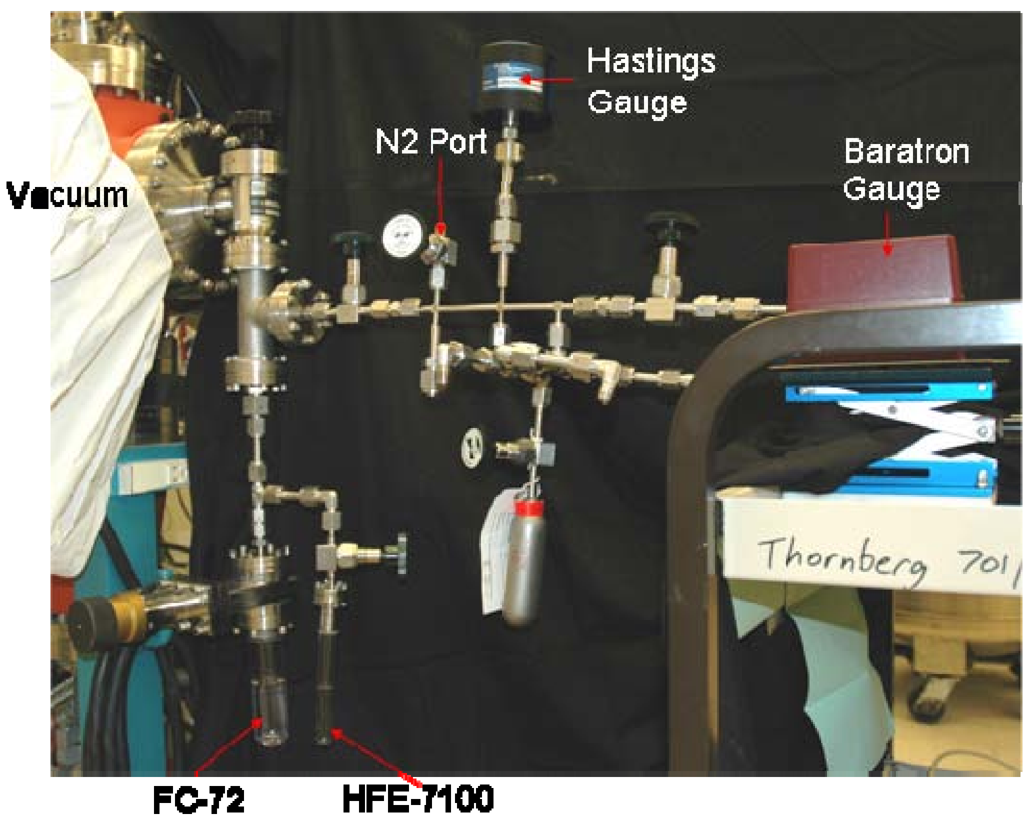

Figure 2: Picture of Manifold

The manifold is comprised of two major components, each with a different function. For the purpose of this report, the first section of manifold will be defined as the region from the vacuum pump system up to valve 2, while the second section is defined as the region from valve 2 to the Baratron gauge. The first section of manifold is where the HFE-7100 and FC-72 fluids are located. Each fluid is enclosed in a separate container that can be isolated from the rest of the manifold. This section acts as a "holding reservoir" so desired amounts of pure HFE-7100 or FC-72 vapor may be introduced to the second section. The second section is where the standards are actually generated. It is here that a gas line for dilution, two gauges to measure pressure, and the standard bottles are attached. Both sections of the manifold can be evacuated through the vacuum pump system.

Before any standard can be generated, the room air in the headspace of each fluid container must be removed. If the headspace is not pure, then the pressure used to mix each standard will not accurately reflect the vapor pressure of the pure fluid. Removal of the room air is accomplished by freezing each fluid with liquid nitrogen, allowing the headspace to be evacuated by the vacuum pump system, thawing the fluid with the vacuum closed, and repeating several times. Detailed instructions can be found in the appendix as **Section 1** (**Headspace Purification**). After the room air contamination has been removed and the fluid has equilibrated to room temperature, any of the three standard sets can be generated.

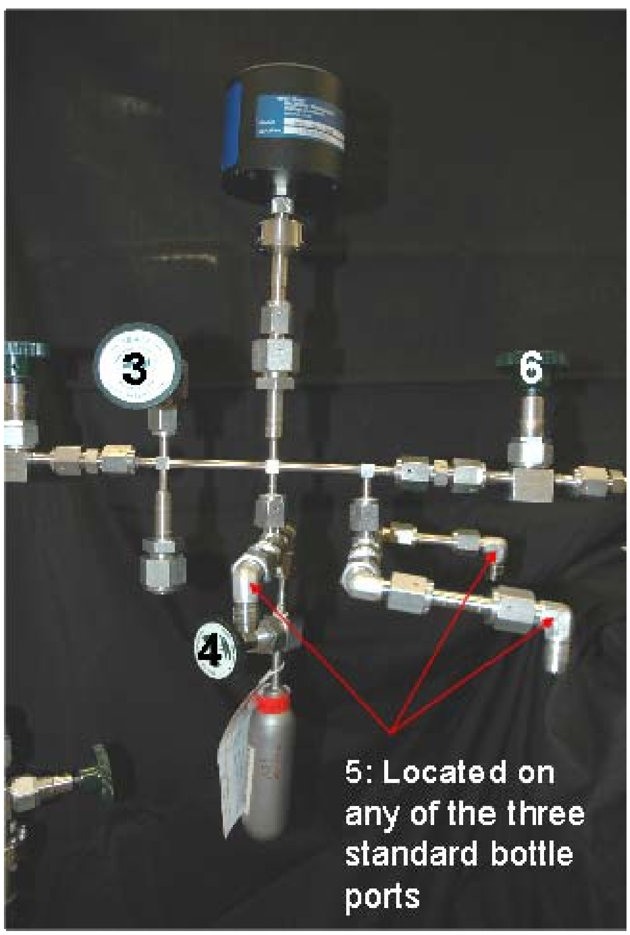

Figure 3: Enlarged View of Valve System

The first standard set produced contained the 1%, 3%, and 10% by volume HFE-7100/FC-72 mixtures and was produced using a 1000 (± 0.12% of reading) torr Baratron gauge. The total pressure of each standard was 990 torr with HFE-7100 and FC-72 having individual pressures of the following: 9.9 torr, 1%; 29.7 torr, 3%; 99 torr, 10 %. Further details are presented in **Table 1** with the associated theory calculations in the appendix. The appendix also contains step-by-step instructions in **Section 2 (Mixing the 1%, 3%, and 10% HFE-7100 and FC-72 Standards)**.

Table 1: Summary of Manifold Pressures for 1%, 3%, and 10% Standards

	Pressure of HFE-7100	Pressure of FC-72	Combined Pressure	Total Pressure
1%	9.9 torr	9.9 torr	19.8 torr	990 torr
3%	29.7 torr	29.7 torr	59.4 torr	990 torr
10%	99 torr	99 torr	198 torr	990 torr

Standards with a concentration of less then 1% were also desired. However, generating dilute standards from pure vapor is difficult and provides many opportunities for error. Therefore, FC-72 or HFE-7100 vapor was converted to 1, 10, and 100 ppmv standards through a double-dilution process from a 1% HFE-7100/FC-72 standard. The 1% standard can be made via two processes. It can be made through the steps outlined in Section 2 or can be made through the process described in **Section 3 (Mixing a 1% HFE-7100 and FC-72 Standard for Further Dilution)**. When making low concentration standards, it is recommended the process described in Section 3 be utilized. This is because one of the main advantages of this manifold design is that all standards in each set can be made by opening and closing valves in a specified order. No gas samples bottles or gauges need to be exchanged during the process. This decreases the likelihood of a leak and contamination becoming introduced. Unlike the high concentration standards generated in Section 2, these low concentration standards are generated using a 10 (± 0.12% of reading) torr Baratron gauge and a 1000 (± 1.5% of reading) torr Hastings gauge. A combination of gauges was required because the Hastings gauge has a high uncertainty at low pressures, while final pressure of the standards (760 torr) exceeded the capabilities of the 10 torr Baratron gauge. The total pressure for each standard was 760 torr with the 1% HFE7100/FC-72 standard providing the following pressures: 0.076 torr, 1 ppmv; 0.76 torr, 10 ppmv; 7.6 torr, 100 ppmv. Further details are presented in **Table 2** and the associated calculations are in the appendix. The appendix also contains step-by-step instructions for making these standards as **Section 4 (Mixing the 1, 10, and 100 ppmv HFE-7100 and FC-72 Standards).**

Table 2: Summary of Manifold Pressures for 1, 10, and 100 ppmv Standards

	Pressure of 1% Mixture in Manifold	Total Pressure
1 ppmv	.076 torr	760 torr
10 ppmv	0.76 torr	760 torr
100 ppmv	7.6 torr	760 torr

The final standard set contained the 0.1, 1, and 10 ppmv HFE-7100/FC-72 standards using a 10 (± 0.12%) torr Baratron gauge. These standards were also produced through a multiple dilution process. The 10 ppmv standards were generated by diluting a 1% standard while the 0.1 and 1 ppmv standards were generated by diluting a 100 ppmv standard. A summary of manifold pressures is visible in **Table 3** with the associated theory calculations in the appendix. Detailed instructions can be found in **Section 5 (Mixing the 0.1, 1, and 10 ppmv HFE-7100 and FC-72 Standards)** of the appendix.

Table 3: Summary of Manifold Pressures for 0.1, 1, and 10 ppmv Standards

	Pressure of 100 ppmv Standard in Manifold	Pressure of 1% Standard in Manifold	Total Pressure
0.1 ppmv	0.76 torr	-	760 tor
1 ppmv	7.6 torr	-	760 tor
10 ppmv	-	0.76 torr	760 tor

GAS ANALYSIS

Samples were analyzed using cryofocusing gas chromatography/mass spectrometry (Cryo/GC/MS). Each prepared standard was connected to inlet 1 of an Entech model 7100 pre-concentrator. Two (1, 10, and 100 ppmv standard set) or ten (0.1, 1, and 10 ppmv standard set) cubic centimeters of sample were concentrated and injected into a Agilent 6890N gas chromatograph (J&W Scientific, DB5-MS, 0.25 µm I.D., 0.25 µm film thickness, 60 meters long) with a split injection ratio of 5:1. Helium (UHP grade) was used as the carrier gas for the GC at a flow rate of 1.6 ml/minute. The oven temperature profile is summarized as **Table 4** with a total run time of 51.8 min with a 3 min filament delay. The separated analytes were detected using a Jeol GC-Mate II mass spectrometer that was scanned over a 10-220 m/z range. Blanks (house N_2) were run between samples.

Table 4: GC Temperature Profile

Temp (°C)	Hold Time (min)	Rate (°C/min)
-40	2	-
-40 to 30	-	9
30 to 120	-	3
120 to 150	-	3
150	2	-

RESULTS AND DISCUSSION

Data analysis was performed using version 2.02 of TSS 2000 software (Shrader Analytical and Consulting Laboratories). Components were identified using library matching and spectrums are shown in **Figures 4** and **5**. Reconstructed ion chromatogram(s) (RIC) were then applied to the total ion chromatograph (TIC) to further differentiate HFE-7100 and FC-72 from other compounds (retention times of 6.68 min and 11.72 min, respectively). For the HFE-7100, an RIC of m/z equal to 69.04 ± 0.4 Daltons was applied. For the FC-72, RICs of m/z equal to 69.04 ± 0.4 Daltons and 81.06 ± 0.4 Daltons were used. An example of an FC-72 RIC is shown as **Figure 6**. Calibration curves were generated by plotting peak intensity (obtained from the RIC and TIC, if possible) for each standard against gas concentration (ppmv) and applying a linear regression.

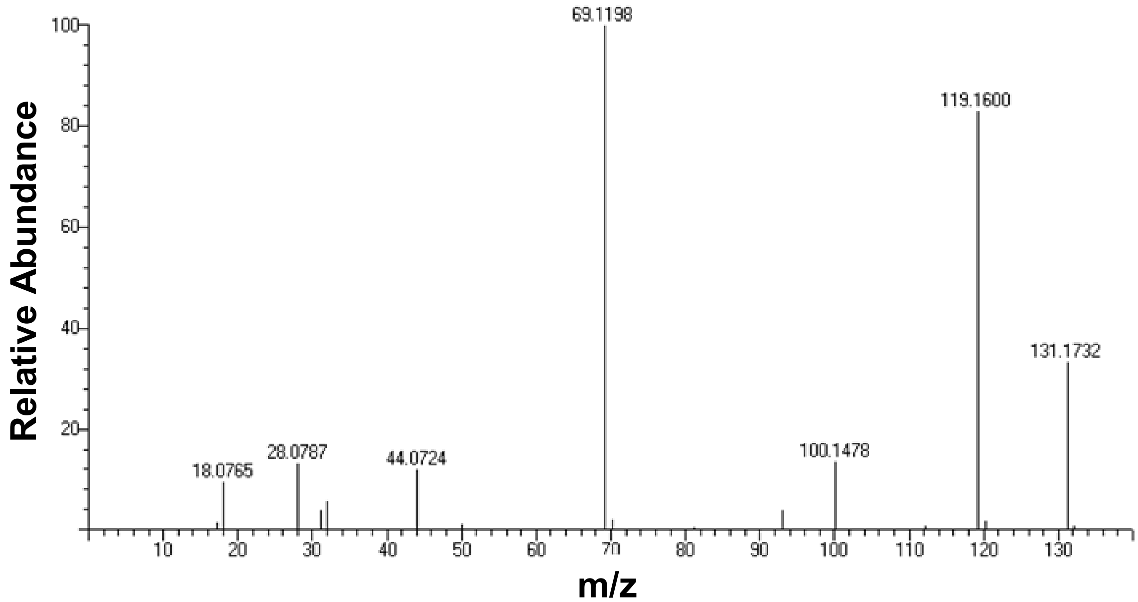

Figure 4: Mass Spectrum of HFE-7100

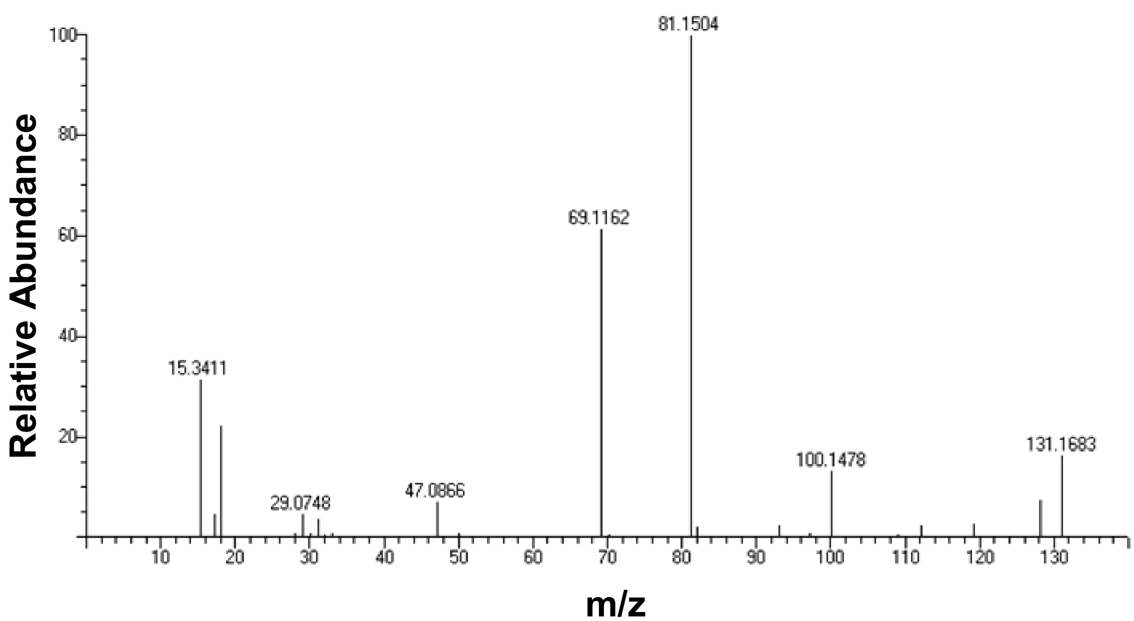

Figure 5: Mass Spectrum of FC-72

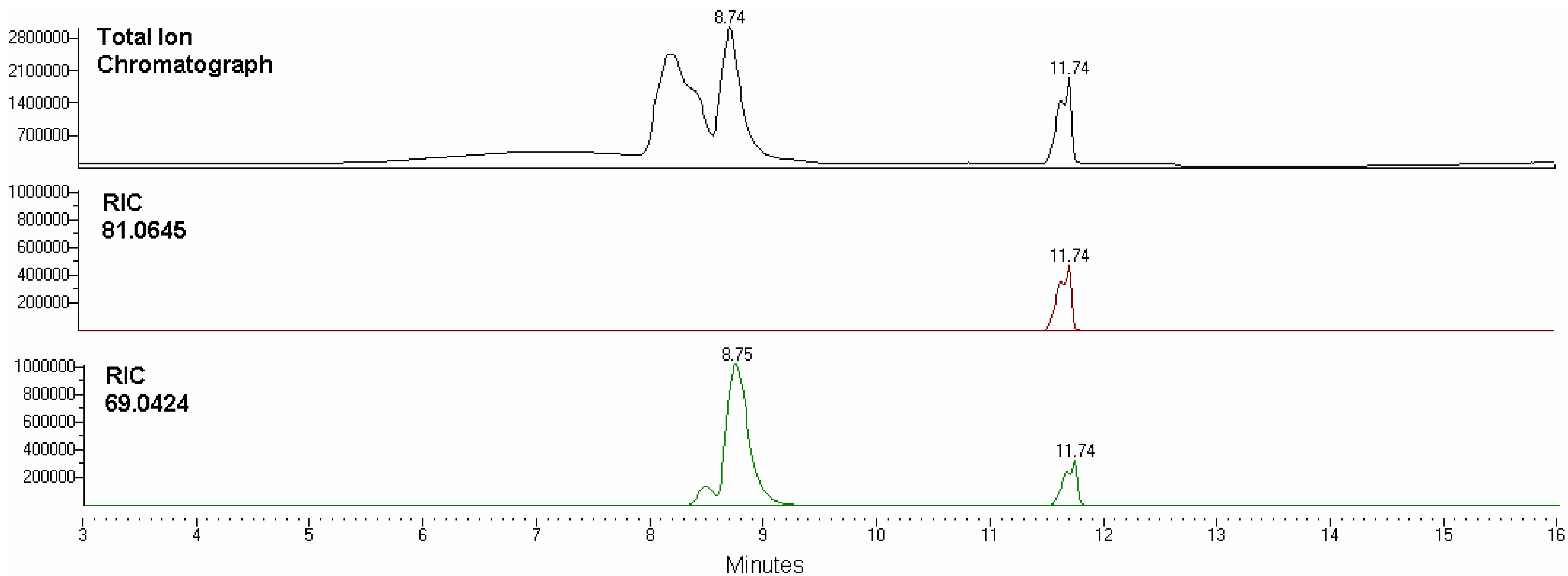

Figure 6: Reconstructed Ion Chromatograph

For the 1, 10, and 100 ppmv standard set, a calibration curve and data summary for HFE-7100 are shown as **Figure 7** and **Table 5**, and for FC-72 as **Figure 8** and **Table 6**. For the 0.1, 1, and 10 ppmv standard set, a calibration curve and data summary for HFE-7100 are shown as **Figure 9** and **Table 7,** and for FC-72 as **Figure 10** and **Table 8**. Data analysis was not performed for the 1%, 3%, and 10% standard set because these higher concentrations saturated the detector.

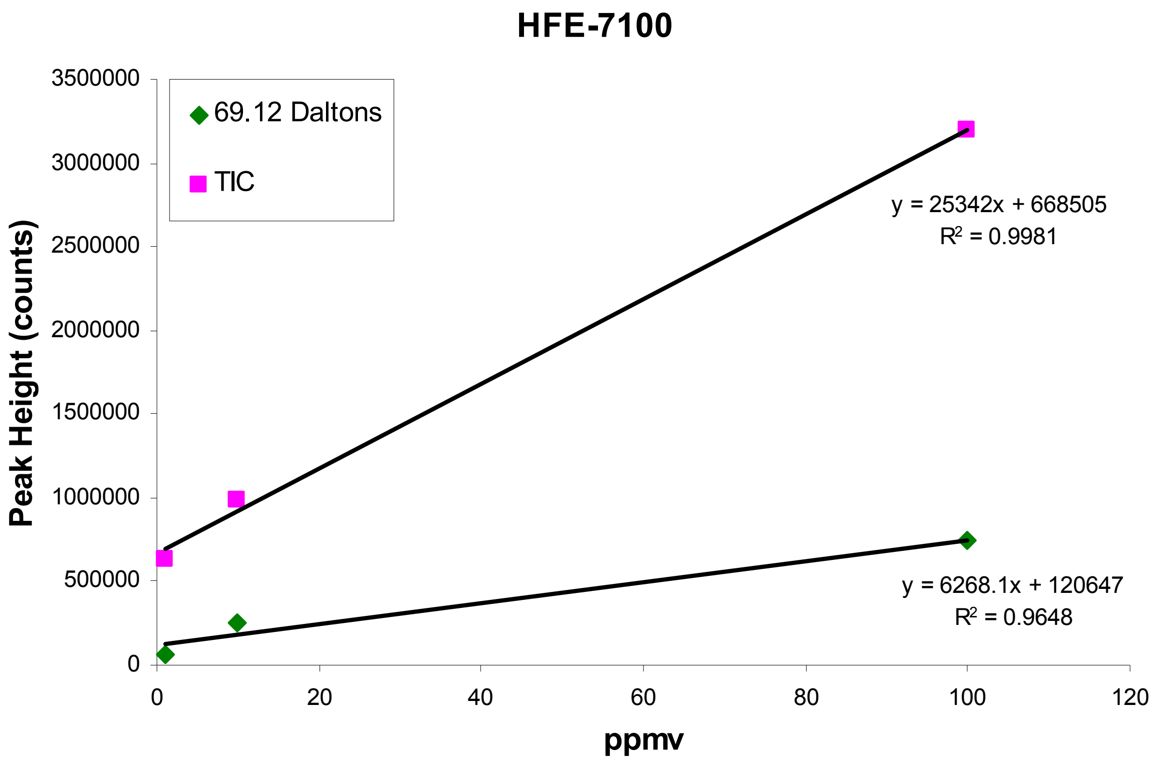

Figure 7: HFE-7100 Calibration Curve for 1, 10, and 100 ppmv Standards

Table 5: Data Used to Generate HFE-7100 Calibration Curve for 1, 10, and 100 ppmv Standards

Concentration (ppmv)	69.12 ± 0.4 Daltons (counts)	TIC (counts)
1	64742	636092
10	251718	985458
100	741238	3196944

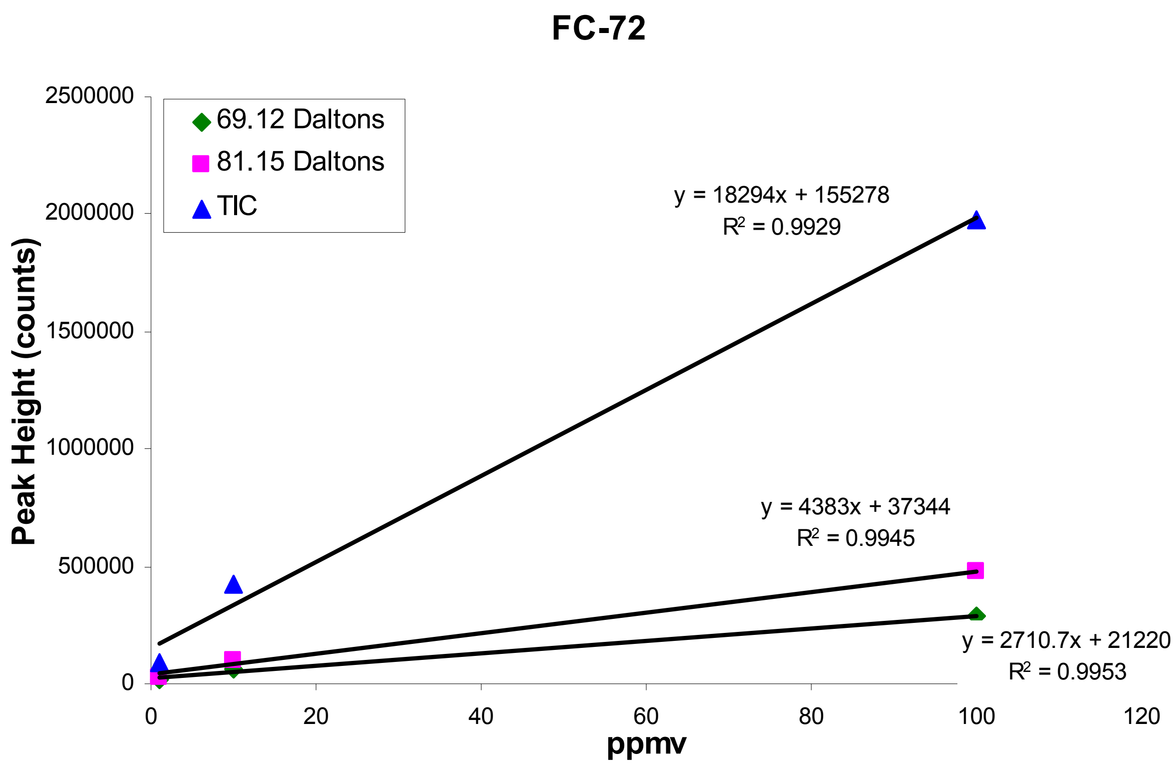

Figure 8: FC-72 Calibration Curve for 1, 10, and 100 ppmv Standards

Table 6: Data Used to Generate FC-72 Calibration Curve for 1, 10, and 100 ppmv Standards

Concentration (ppmv)	69.11 ± 0.4 Daltons (counts)	89.15 ± 0.4 Daltons (counts)	TIC (counts)
1	14230	24854	93424
10	58998	99734	426382
100	291318	473958	1976674

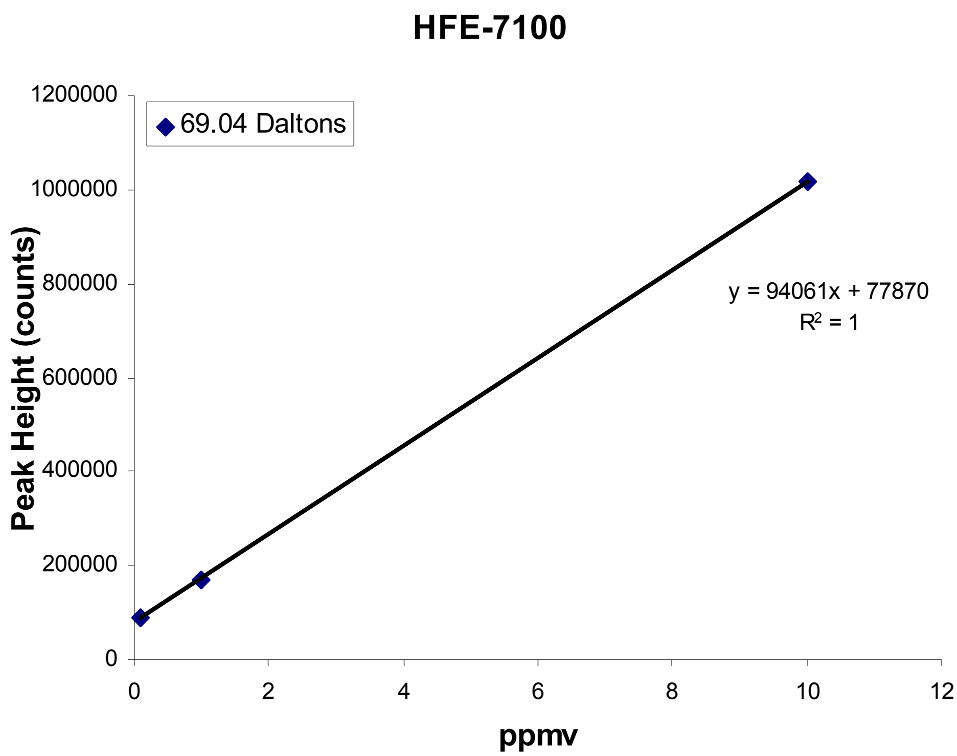

Figure 9: HFE-7100 Calibration Curve for 0.1, 1, and 10 ppmv Standards

Table 7: Data Used to Generate HFE-7100 Calibration Curve for 0.1, 1, and 10 ppmv Standards

Concentration (ppmv)	69.04 ± 0.4 Daltons (counts)
0.1	88982
1	170054
10	1018646

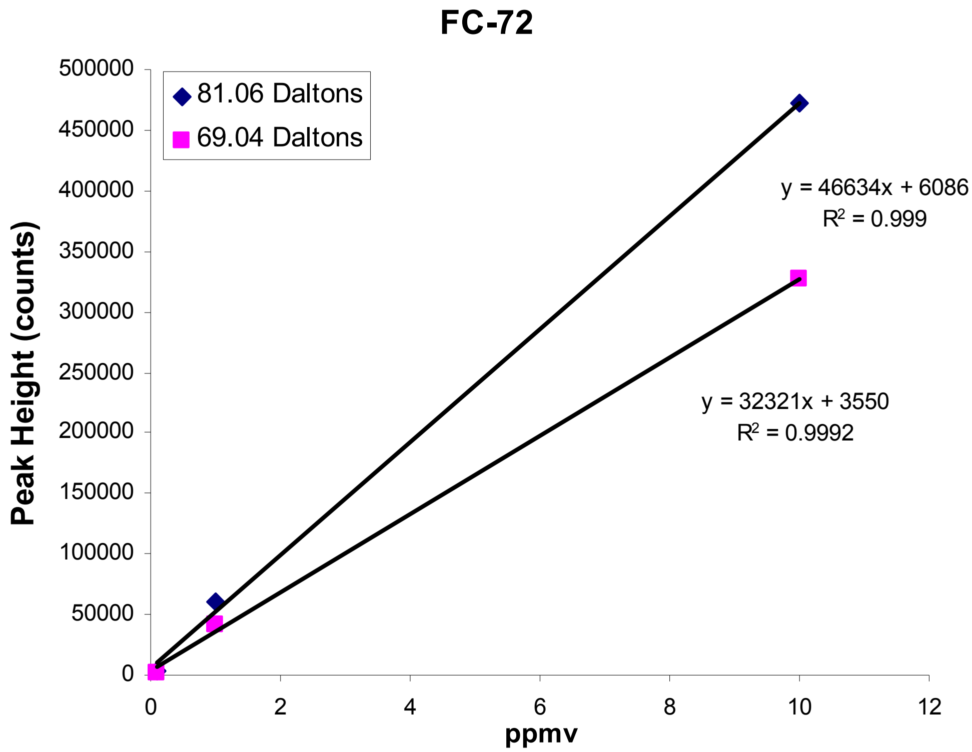

Figure 10: FC-72 Calibration Curve for the 0.1, 1, and 10 ppmv Standards

Table 8: Data Used to Generate FC-72 Calibration Curve for 0.1, 1, and 10 ppmv Standards

Concentration (ppmv)	69.04 ± 0.4 Daltons (counts)	89.06 ± 0.4 Daltons (counts)
0.1	1990	2918
1	41142	61334
10	326278	471638

The accuracy of this manifold design is demonstrated through the coefficient of determination (R^2) values. Both sets of standards analyzed by the Jeol GC Mate II produced one or more linear regression lines with an R^2 value of greater then 0.99. This indicates a strong linear relationship between ppmv and peak intensity.

The design of this manifold provides several advantages. First, the manifold design allows for the removal of the room air present in the headspace above each liquid. Each standard only contains HFE-7100, FC-72, and the gas used to backfill. Second, the manifold design enables all standards in each set to be made by the opening and closing valves in a specified order. No gas samples bottles are exchanged during the process thereby decreasing the likelihood of a leak and contamination becoming introduced. Third, this manifold allows for wide concentration range of standard concentrations to be generated by simply using the appropriate Baratron gauge. Although not part of this report, lower concentration standards could be generated using a 1 torr Baratron gauge or by performing more dilution processes.

GENERAL REFERENCES

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Swagelok [homepage on the internet]. Solon, Ohio; Swagelok Company; c2007. Available from: **www.swagelok.com**

APPENDIX

PARTS UTILIZED

Gauges

1	MKS Instruments, Baratron, 10 torr, model 690A11TRC
1	MKS Instruments, Baratron, 1000 torr, model 690A13TRC
1	Hastings, dual sensor vacuum gauge, model HPM-2002-OBE

Glass Components

1*	461010 461011 461023	MDC Instruments, glass to metal adapter, flange 2 ¾ in.
1*	461000 – 461007	MDC Instruments, glass to metal adapter, flange 1 1/3 in.

*Note: Only one glass to metal adapter is required from each group. Models differ in their glass diameters and/or wall thickness. Flange size remains constant within each grouping.

Flanges and Associated Fittings

1	404002	MDC Instruments, tee, 2 ¾ in, Ref # 150-3
1	414007	MDC Instruments, male VCR to Del-Seal CF, male VCR nom size ¼ in., flange NOM 2 ¾, ref # VCR25-275
1	414009	MDC Instruments, male VCR to Del-Seal CF, male VCR

Valves

3	SS-4BG-V51	SS bellows-sealed valve, ¼ in female Swagelok VCR face seal fitting
1	SS-4BG-TW	SS bellows-sealed valve, gasket, spherical stem tip, ¼ in. TSW and 3/8 in TBW
1	312029	MDC Instruments, angle valve – 1 ½ in port. model ref: AV-150M
1	300001	MDC Instruments, circular gate valve – 1 ½ in. gate valve, model ref: GV-1500V

VCR Metal Gasket Fittings

5	SS-4-VCR-9	316 SS VCR face seal fitting, ¼ in union elbow body
6	SS-4-WVCR-6-DF	316 SS welded VCR face seal fitting, ¼ in. rotating female union

3	SS-4-VCR-T	316 SS VCR face seal fitting, ¼ in. union tee
5	SS-4-VCR-6-DM	316 SS VCR face seal fitting, ¼ in. double male union body
2	SS-4-VCR-7-8VCRF	316 SS VCR face seal fitting, reducing adapter body, ¼ in. VCR x ½ in. female VCR
1	SS-4-VCR-CG	316 SS VCR face seal fitting, ¼ in. female VCR coupling body
6	SS-4-VCR-1	316 SS VCR face seal fitting, 1/4 in. female nut
1	SS-8-VCR-4	316 SS VCR face seal fitting, 1/2 in. male nut

Weld Fittings

7	6LV-4-VCR-3S-4TB3	316L VAR VCR fitting, short tube butt weld gland, 1/4 in. VCR x 1/4 x 0.035 in. TBW, 0.38 in. (9.6 mm) extension
2	316L-4-ATW-3	316L stainless steel automatic tube butt weld union tee, 1/4 in. OD
1	316L-4-ATW-4	316L stainless steel automatic tube butt weld union cross, 1/4 in. OD
1	6LV-8-VCR-3-4TB7	316L VAR VCR fitting, long tube butt weld gland, 1/2 in. VCR x 1/4 x 0.035 in. TBW, 0.75 in. (19.1 mm) extension

INSTRUCTIONS

Section 1: Headspace Purification

Directions:

1. Close valves 2, 7 (HFE-7100), and 8 (FC-72). Valve 1 to the vacuum pump should remain open.
2. Using liquid nitrogen, slowly freeze either the HFE-7100 or FC-72.
3. While the solvent is frozen, open the corresponding valve slowly to allow the headspace above the frozen liquid to be evacuated. Wait 10 seconds. IMPORTANT: To prevent damaging the vacuum pump, the solvent must remain frozen while the valve is open.
4. Close the valve and wait for the fluid to thaw.
5. Repeat steps 2 through 4 twice for the same fluid.
6. Repeat steps 1-4 with the second fluid.

Section 2: Mixing the 1%, 3%, and 10% HFE-7100 and FC-72 Standards

Pressure Gauge Used: 1000 torr Baratron

Directions:

1. Evacuate the manifold by opening valves 1, 2, 6, and the valve to all sample bottles awaiting filling.
2. Close valve 1 and wait 1 minute. The Baratron and Hastings gauges should remain at zero torr. This step ensures there is no leak in the manifold.

3. Close valve 2 and the valves to any bottles you do not want to fill immediately.
4. Open valve 7 (HFE-7100) allowing the HFE-7100 vapor to fill the first portion of the manifold. Wait 1 minute.
5. Close valve 7.
6. Open valve 2 slowly. Watch the Baratron gauge and fill the second portion of the manifold to the desired pressure: 9.9 torr, 1%; 29.7 torr, 3%; 99 torr, 10%. Close valve 2 when desired pressure is reached.
7. Open valve 1 to evacuate the first portion of the manifold.
8. Close valve 1.
9. Open valve 8 (FC-72) allowing the FC-72 vapor to fill the first portion of the manifold. Wait 1 minute.
10. Close valve 8.
11. Open valve 2 slowly. Watch the Baratron gauge the fill the second portion of the manifold to the desired pressure: 19.8 torr, 1%, 59.4 torr, 3%, 198 torr, 10 %. Close valve 2 when desired pressure is reached.
12. Open valve 3 slowly allowing the manifold to fill with the backfill gas (typically N_2 or Ar). Close valve 3 when the pressure reaches 990 torr.
13. Close the valve leading to the filled standard bottle.
14. Open valve 1 and 2 to evacuate the entire manifold.

This process then repeated until all three standards are made. All steps are the same except the pressure described in step 6.

15. Close valves 2 and 6.
16. Remove 1%, 3%, and 10% HFE-7100/FC-72 standards from the manifold.

When filling 1% and 3% standards, the order of gas introduction to the second section manifold is not important. However, when generating the 10% standard, the HFE-7100 must be introduced prior to the FC-72. This is because the vapor pressure of FC-72 (232 mmHg at 20°C) is higher than that of HFE-7100 (202 mmHg at 25°C). This order makes it easier to achieve a combined pressure of 198 mmHg.

Section 3: Mixing the 1% HFE-7100 and FC-72 Standard for Further Dilution

Pressure Gauges Used: 1000 torr Hastings, 10 torr Baratron

Directions:
1. Evacuate the manifold by opening valves 1, 2, 4, and 6.
2. Close valve 1 and wait 1 minute. The Baratron and Hastings gauges should remain at zero torr. This step ensures there is no leak in the manifold.
3. Close all valves.
4. Open valve 7 allowing the vapor of FC-72 to fill the first portion of the manifold.
5. Close valve 7.
6. Open valves 2 and 4. The vapor pressure of the first liquid should register on the Hastings gauge.
7. Open valve 1 slowly allowing some of the vapor to escape into the vacuum. Close valve 1 when the Hastings gauge displays a pressure just less then 10 torr.

8. Open valve 6 slowly to ensure the Baratron gauge is not pressurized to greater then 10 torr.
9. Open valve 1 slowly allowing some of the vapor to escape into the vacuum. Close valve 1 when the Baratron gauge displays a pressure of 5 torr.
10. Close valve 2.
11. Open valve 1. This will evacuate only the first half of the manifold.
12. Close valve 1.
13. Open valve 8 allowing the vapor of HFE-7100 to fill the first portion of the manifold.
14. Close valve 8.
15. Open valve 2 slowly allowing some of the vapor to fill the second half of the manifold. Close Valve 2 when the Baratron gauge reads 10 torr.
16. Open valve 1 to evacuate only the first half of the manifold.
17. Close valve 6 to protect the Baratron gauge.
18. Open valve 3 slowly allowing the backfill gas (typically N_2 or Ar) to fill the manifold. Close valve 3 when the Hastings gauge reads 500 torr.
19. Close valve 4 to close the newly formed 1% HFE-7100/FC-72 standard.
20. Open valves 1 and 2 to evacuate the full manifold. When the pressure reads less then 10 torr on the Hastings gauge, open valve 6 to evacuate the space before the Baratron.

Section 4: Mixing the 1, 10, 100 ppmv HFE-7100 and FC-72 Standards

The 1, 10, and 100 ppmv standards are generated by further diluting the 1% mixture made in Section 2 or 3.

Pressure Gauges Used: 1000 torr Hastings, 10 torr Baratron

Directions:

1. Evacuate the manifold by opening valves 1, 2, 6, and the valve to any sample bottle awaiting filling.
2. Close valve 1 and wait 1 minute. The Baratron and Hastings gauges should remain at zero torr. This step ensures there is no leak in the manifold.
3. Close all valves.
4. Open valve 4 allowing the 1% HFE-7100/FC-72 mixture to fill the manifold. The increased pressure should register on the Hastings gauge.
5. Wait 10 seconds and close valve 4.
6. Open valve 5 to allow the 1% mixture to enter the sample bottle to be filled immediately.
7. Open valve 1. Open valve 2 slowly allowing some of the vapor to escape into the vacuum. Close valve 2 when the Hastings gauge displays a pressure just under 10 torr.
8. Open valve 6 slowly ensuring the Baratron is not pressurized to greater then 10 torr.
9. Open valve 2 slowly to allow more of the vapor to escape into the vacuum. Close valve 2 when the Baratron displays the desired pressure: 100 ppmv, 7.6 torr; 10 ppmv, 0.76 torr; 1 ppmv, 0.076 torr.
10. Close valve 6 to protect the Baratron gauge.

11. Open valve 3 slowly to allow the backfill gas (typically N_2 or Ar) to fill the manifold. Close valve 3 when the Hastings reads 760 torr.
12. Close valve 5.
13. Open valve 2 to evacuate the full manifold. When the pressure on the Hastings gauge reads less then 10 torr, open valve 6 to evacuate the space before the Baratron.

This process is then repeated until all three standards are made. All steps are the same except the pressure described in step 9.

14. After all three standards have been generated. Close valves 2 and 6.
15. Remove 1, 10, and 100 ppmv HFE-7100/FC-72 standards from the manifold.

Section 5: Mixing the 0.1, 1, and 10 ppmv HFE-7100 and FC-72 Standards

This section describes the method for making the 0.1, 1, and 10 ppmv HFE-7100/FC-72 standards using a 10 torr Baratron gauge. These standards are also produced through a multiple dilution process. The 10 ppmv standards were generated by diluting a 1% standard, while the 0.1 and 1 ppmv standards were generated by diluting a 100 ppmv standard. These instructions assume that a 1% standard is already attached to the manifold at position 4 (see Section 3).

Pressure Gauges Used: 1000 torr Hastings, 10 torr Baratron

Directions:

1. Evacuate the manifold by opening valves 1, 2, 6, and the valve to any standard bottle awaiting filling.
2. Close valve 1 and wait 1 minute. The Baratron and Hastings gauges should remain at zero torr. This step ensures there is no leak in the manifold.
3. Close all valves.
4. Open valve 4 allowing the 1% HFE-7100 and FC-72 mixture to fill the second portion of the manifold. The increased pressure should register on the Hastings gauge.
5. Wait 10 seconds and close valve 4.
6. Open valve 5. The pressure in the manifold should drop slightly as the gas expands into the empty sample bottle.
7. Open valve 1. Open valve 2 allowing some of the vapor to escape into the vacuum. Close valve 2 when the Hastings gauge displays a pressure of just under 10 torr.
8. Open valve 6 slowly ensuring the Baratron gauge is not pressurized to greater then 10 torr.
9. Open valve 2 slowly to allow more vapor to escape into the vacuum. Close valve 2 when the Baratron gauge displays a pressure of 7.6 torr.
10. Close valve 6 to protect the Baratron gauge.
11. Open valve 3 slowly to allow the backfill gas (typically N_2 or Ar) to fill the manifold. Close valve 3 when the Hastings gauge reads 760 torr. This is a 100 ppmv standard that will be further diluted to make the 0.1 and 1 ppmv standards.
12. Close the valve leading to the 100 ppmv standard.

13. Open valve 2 to evacuate the full manifold. When the pressure reads less then 10 torr on the Hastings, open valve 6 to evacuate the space before the Baratron.

Step 14 begins the process of making the 0.1 and 1 ppmv standards:

14. Close valves 2 and 6.
15. Open the valve leading to the 100 ppmv standard to allow the standard to fill the second half of the manifold. The increased pressure should register on the Hastings gauge.
16. Wait 10 seconds and close the valve.
17. Open the valve leading to the sample bottle to be filled. The pressure in the manifold should drop slightly as the gas expands into the empty sample bottle.
18. Open valve 1. Open valve 2 allowing some of the vapor to escape into the vacuum. Close valve 2 when the Hastings gauge displays a pressure of just less then 10 torr.
19. Open valve 6 slowly to ensure the Baratron gauge is not pressurized to greater then 10 torr.
20. Open valve 2 slowly to allow more vapor to escape into the vacuum. Close valve 2 when the Baratron gauge displays the following pressure: 0.76 torr, 0.1 ppmv; 7.6 torr, 1 ppmv.
21. Close valve 6 to protect the Baratron gauge.
22. Open valve 3 slowly to allow the backfill gas (typically N_2 or Ar) to fill the manifold. Close valve 3 when the Hastings gauge reads 760 torr.
23. Close the valve leading to the newly formed standard.
24. Open valve 2 to evacuate the full manifold. When the pressure reads less then 10 torr on the Hastings gauge, open valve 6 to evacuate the space before the Baratron gauge.
25. Repeat steps 14 through 24 to form the second standard.

At this point a 0.1 and 1 ppmv HFE-7100/FC-72 should be prepared. Step 26 begins the process to make the 10 ppmv standard. All steps are the same except the pressure described in step 20.

26. Close valve 6.
27. Open the valve leading to the 100 ppmv standard. IMPORTANT: This will evacuate the 100 ppmv standard.
28. Close the valve leading to the new evacuated standard bottle.
29. Open valve 4 allowing the second half of the manifold to be filled with 1% HFE-7100/FC-72 standard. The increased pressure should register on the Hastings gauge.
30. Wait 10 seconds and close the valve.
31. Open the valve leading to the sample bottle to be filled. The pressure in the manifold should drop slightly as the gas expands into the empty sample bottle.
32. Open valve 1. Open valve 2 allowing some of the vapor to escape into the vacuum. Close valve 2 when the Hastings gauge displays a pressure of just under 10 torr.
33. Open valve 6 slowly to ensure the Baratron gauge is not pressurized to greater then 10 torr.
34. Open valve 2 slowly to allow more vapor to escape into the vacuum. Close valve 2 when the Baratron gauge displays a pressure of 0.76 torr.
35. Close valve 6 to protect the Baratron gauge.

36. Open valve 3 slowly to allow the backfill gas (typically N_2 or Ar) to fill the manifold. Close valve 3 when the Hastings gauge reads 760 torr.
37. Close the valve leading to the 10 ppmv standard.
38. Open valve 2 to evacuate the manifold. When the pressure reads less then 10 torr on the Hastings gauge, open valve 6 to evacuate the space before the Baratron gauge.
39. Close valve 2 and 6.
40. Remove 0.1, 1, and 10 ppmv HFE-7100/FC-72 standards.

THEORY CALCULATIONS

1%, 3%, and 10% HFE-7100 and FC-72 Standards

$$\frac{\text{Partial Pressure of Each Analyte Gas}}{\text{Total Pressure}} = \frac{9.9 \text{ torr}}{990 \text{ torr}} = 0.01 \text{ or } 1\%$$

$$\frac{\text{Partial Pressure of Each Analyte Gas}}{\text{Total Pressure}} = \frac{29.7 \text{ torr}}{990 \text{ torr}} = 0.03 \text{ or } 3\%$$

$$\frac{\text{Partial Pressure of Each Analyte Gas}}{\text{Total Pressure}} = \frac{99 \text{ torr}}{990 \text{ torr}} = 0.1 \text{ or } 10\%$$

1% HFE-7100 and FC-72 Standard

$$\frac{\text{Partial Pressure of Each Analyte Gas}}{\text{Total Pressure}} = \frac{5 \text{ torr}}{500 \text{ torr}} = 0.01 \text{ or } 1\%$$

1, 10, 100 ppmv HFE-7100 and FC-72 Standards

Used for further dilution:

1% FC-72 or HFE-7100 = 10,000 ppmv

Final Standards:

1 ppmv:

$$\frac{1\% \text{ Standard (ppmv)}}{\text{Standard (ppmv)}} = \frac{10{,}000 \text{ ppmv}}{1 \text{ ppmv}} = 10{,}000 \text{ (dilution factor)}$$

$$\frac{\text{Total Pressure (torr)}}{\text{Dilution Factor}} = \frac{760 \text{ torr}}{10{,}000} = 0.076 \text{ torr of } 1\% \text{ standard}$$

10 ppmv:

$$\frac{1\% \text{ Standard (ppmv)}}{\text{Standard (ppmv)}} = \frac{10{,}000 \text{ ppmv}}{10 \text{ ppmv}} = 1{,}000 \text{ (dilution factor)}$$

$$\frac{\text{Total Pressure (torr)}}{\text{Dilution Factor}} = \frac{760 \text{ torr}}{1{,}000} = 0.76 \text{ torr of } 1\% \text{ standard}$$

100 ppmv:

$$\frac{\text{1\% Standard (ppmv)}}{\text{Standard (ppmv)}} = \frac{\text{10,000 ppmv}}{\text{100 ppmv}} = 100 \text{ (dilution factor)}$$

$$\frac{\text{Total Pressure (torr)}}{\text{Dilution Factor}} = \frac{\text{760 torr}}{100} = 7.6 \text{ torr of 1\% standard}$$

0.1, 1, and 10 ppmv HFE-7100 and FC-72 Standards

Used for further dilution:

1% FC-72 or HFE-7100 = 10,000 ppmv

100 ppmv:

$$\frac{\text{1\% Standard (ppmv)}}{\text{Standard (ppmv)}} = \frac{\text{10,000 ppmv}}{\text{100 ppmv}} = 100 \text{ (dilution factor)}$$

$$\frac{\text{Total Pressure (torr)}}{\text{Dilution Factor}} = \frac{\text{760 torr}}{100} = 7.6 \text{ torr of 1\% standard}$$

Final standards:

0.1 ppmv:

$$\frac{\text{100 ppmv Standard}}{\text{Standard (ppmv)}} = \frac{\text{100 ppmv}}{\text{0.1 ppmv}} = 1{,}000 \text{ (dilution factor)}$$

$$\frac{\text{Total Pressure (torr)}}{\text{Dilution Factor}} = \frac{\text{760 torr}}{1{,}000} = 0.76 \text{ torr of 100 ppmv standard}$$

1 ppmv:

$$\frac{\text{100 ppmv Standard}}{\text{Standard (ppmv)}} = \frac{\text{100 ppmv}}{\text{1 ppmv}} = 100 \text{ (dilution factor)}$$

$$\frac{\text{Total Pressure (torr)}}{\text{Dilution Factor}} = \frac{\text{760 torr}}{100} = 7.6 \text{ torr of 100 ppmv standard}$$

10 ppmv:

$$\frac{\text{1\% Standard (ppmv)}}{\text{Standard (ppmv)}} = \frac{\text{10,000 ppmv}}{\text{10 ppmv}} = 1{,}000 \text{ (dilution factor)}$$

$$\frac{\text{Total Pressure (torr)}}{\text{Dilution Factor}} = \frac{\text{760 torr}}{1{,}000} = 0.76 \text{ torr of 1\% standard}$$

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