
A Preliminary Study on the Toxic Combustion Products Testing of Polymers Used in High-Pressure Oxygen Systems

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ABSTRACT

One likely cause of polymer ignition in a high-pressure oxygen system is adiabatic-compression heating of polymers caused by pneumatic impact. Oxidative pyrolysis or combustion of polymers in a high-pressure oxygen system could generate toxic gases. This paper reports the preliminary results of toxic combustion product testing of selected polymers in a pneumatic-impact test system.

Five polymers commonly used in high-pressure oxygen systems, Nylon 6/6, polychlorotrifluoroethylene (CTFE), polytetrafluoroethylene (PTFE), fluoroelastomer (Viton® A), and nitrile rubber (Buna N), were tested in a pneumatic-impact test system at 2500- or 3500-psia oxygen pressure. The polymers were ignited and burned, then combustion products were collected in a stainless-steel sample bottle and analyzed by GC/MS/IRD, GC/FID, and GC/Methanizer/FID.

The results of adiabatic-compression tests show that combustion of hydrocarbon polymers, nitrogen-containing polymers, and halogenated polymers in high-pressure oxygen systems are relatively complete. Toxicity of the combustion product gas is presumably much lower than the combustion product gas generated from ambient-pressure oxygen (or air) environments.

The NASA-Lewis equilibrium code was used to determine the composition of combustion product gas generated from a simulated, adiabatic-compression test of nine polymers. The results are presented and discussed.

INTRODUCTION

Organic polymers have been widely used in oxygen systems. Applications include O-rings, seals, diaphragms, manifold gaskets, flexible hoses, valve seats, and endotracheal tubes. Some organic polymers are frequently used in high-pressure oxygen environments.

It is well known that polymers are more flammable in oxygen-enriched environments than in air, especially in high-pressure oxygen environments. Ignition of organic polymers in a high-pressure oxygen system may be due to mechanical impact, rapid compression, friction, high-speed particle impact, electrical arcs, or high operating

temperatures.¹ When the temperature exceeds the ignition temperature of a polymer, a fire may occur.

It has been realized that toxic gases could be generated by oxidative pyrolysis or combustion of polymers in a high-pressure oxygen system. If toxic combustion gases are generated in oxygen equipment used in medicine, direct inhalation of toxic gases could seriously injure the patient. The ISO document entitled *Anaesthetic and Respiratory Equipment – Compatibility with Oxygen* (ISO/DIS 15001.3)² recommends test methods for the combustion and the quantitative analysis of the combustion products of non-metallic materials. In addition, the ASTM G04 Plastics Toxicity Task Force has started to look into: (1) the development and validation of a repeatable methodology for the presence and quantity of combustion products produced by plastics and elastomers used in oxygen systems; and (2) the extension of the methodology developed in (1) to the measurement of combustion products produced by medical cylinder valves, regulators, and flexible tubing.

Because the most likely mechanism for the ignition of organic polymers in a high-pressure oxygen system is adiabatic compression, ISO/DIS 15001.3² recommends an adiabatic-compression test for toxic combustion products. To date, no experimental data have been published for the toxic combustion products testing via an adiabatic-compression mechanism. NASA White Sands Test Facility (WSTF) conducted some tests in an oxygen bomb calorimeter with a test pressure of 450 psia and a thin hot-wire ignition source.³ The main purposes of the current study are to evaluate the feasibility of using the NASA pneumatic-impact test system to perform the toxic combustion product testing, and to determine the combustion products generated from a simulated, adiabatic-compression testing of polymers by the NASA-Lewis equilibrium code.⁴

MATERIALS AND TEST METHOD

Materials

Materials tested in adiabatic-compression tests were Zytel^{®1} 42 (Nylon 6/6), Buna N (nitrile rubber), Viton^{®*} A (copolymer of vinylidene fluoride and hexafluoropropylene), Teflon^{®*} (polytetrafluoroethylene), and Neoflon^{®2} (polychlorotrifluoroethylene).

Materials used in the simulated calculation were: PPO (polypropylene oxide), PEEK (polyetheretherketone), Zytel 42, Buna N, Vespel^{®*} SP-21 (polyimide), Teflon, Neoflon, Viton A, and Tefzel^{®*} (copolymer of tetrafluoroethylene and ethylene).

The above materials are the commonly used polymers for the softgoods in regulators and valves. The sample weight ranged from 0.029 to 0.060 g, which is the typical weight range for softgoods. The use of trade names in this paper does not

¹ Zytel,® Viton,® Teflon,® Vespel,® and Tefzel® are registered trademarks of the DuPont deNemours Company, Wilmington, Delaware.

² Neoflon® is a registered trademark of Daikin Kogyo Kabushiki Kaisha dba Daikin Kogyo Company, Osaka, Japan.

constitute an endorsement of these materials but is used only to simplify their description and identification.

Method

Adiabatic-compression (Pneumatic-impact) Test

The adiabatic-compression (pneumatic-impact) tests were conducted according to NASA-STD-6001 Test 14.⁵ This test method is similar to that of ASTM G74, *Standard Test Method for Ignition Sensitivity of Materials to Gaseous Fluid Impact*.⁶ Figure 1 shows the schematic diagram of the pneumatic-impact system. A sample (4.8-mm diameter) was placed in the test chamber, and an upstream accumulator (1.5 L) was pressurized with the desired pressure of oxygen. The test chamber was then purged with oxygen, and the sample was impacted with high-pressure oxygen by opening a high-speed valve between the accumulator and test chamber. Test pressure was 2500 psia for Zytel 42 and 3500 psia for other materials. The test chamber was pressurized from ambient pressure to the desired test pressure in 50 ms. In this study the sample was impacted only one time, instead of a sequence of five times as specified by NASA and ASTM test standards. An ignition (reaction) was evidenced by a sudden rise of test chamber temperature. If an ignition (reaction) occurred, the combustion product gas was collected using a 320-ml stainless steel sample bottle and analyzed by GC/MS/IRD, GC/FID, and GC/Methanizer/FID.

Determining the Composition of Combustion Product Gas Using the NASA-Lewis Equilibrium Code

In this study, we used the NASA-Lewis Equilibrium Code to determine the composition of the combustion product gas generated from the simulated, adiabatic-compression test of polymers. The sample weight for Zytel 42, Buna N, Teflon, Neoflon, and Viton A was the same as that of the polymers tested in the adiabatic-compression test, and the sample weight used for PPO, PEEK, Vespel SP-21, and Tefzel was 0.05 g. The simulated calculation was conducted at 2000, 2500, 3000, and 3500 psia. Since the space between the accumulator and test chamber measured 12 ml in volume, the amounts of oxygen reacted with polymers were 0.067, 0.084, 0.100, and 0.117 moles for the test pressures of 2000, 2500, 3000, and 3500 psia, respectively. The equilibrium temperatures were set at the maximum temperatures of the test chamber, 100 and 200 °C.

RESULTS AND DISCUSSION

1. Adiabatic-compression (Pneumatic-impact) Test

One of the main purposes of this study is to evaluate the feasibility to use the NASA pneumatic-impact test system to perform toxic combustion products testing. The accumulator (1.5 L), test chamber, and sample bottle (320 ml) in the test system were used to simulate a medical cylinder, compartment of the softgood in a regulator, and human lung, respectively. For adults, the tidal volume of the human lung is about 0.5 L,⁷

but the lungs can hold about ten times this volume if they are filled to full capacity. We have done extensive studies using the NASA pneumatic-impact test system to simulate oxygen equipment used for medical purposes. The results have demonstrated that polymers can be ignited successfully in the NASA pneumatic-impact test system under certain conditions, and the high-pressure combustion product gas (up to 3500 psia) can be safely transferred to the sample bottle for chemical analysis. In this study, we used GC/MS/IRD, GC/FID, and GC/Methanizer/FID to analyze the combustion product gas. Because of the lack of some calibration standards, combustion products such as NO, CF₄, COF₂, HCl, and COCl₂ (phosgene) can be identified but not quantified. We are now in a process of refining and improving our analytical techniques. Table 1 lists the major gaseous combustion products from pneumatic-impact tests of five different polymers.

For nitrogen-containing polymers, Zytel 42 (Nylon 6/6) and Buna N (nitrile rubber), the combustion product gas contains a significant amount of CO₂ (at 15,000-ppm level) and a trace amount of CO (at 10-ppm level). This result indicates that the burning of Zytel 42 and Buna N in the pneumatic-impact test was very complete. When testing Zytel 42, NO, NO₂, N₂O, HCN, and NH₃ were not detected in the combustion product gas. When testing Buna N, trace amounts of NO were detected; however, H₂S, SO₂, HCN, NO₂, N₂O, and NH₃ were not detected.

It was reported that the burning of fluorinated polymers generates CO, CO₂, CF₄, and COF₂.⁸ If the fluorinated polymer contains hydrogen or there is some moisture around the burning environment, significant amounts of HF can be generated.

Table 1 shows that the burning of Teflon produced a significant amount of CO₂ (about 6000 ppm), a small amount of CO (about 60 ppm), and some CF₄. COF₂ was not detected in the combustion product gas. It is suspected that COF₂ may have been decomposed to form CO₂ and CF₄ during the thermal equilibrium of the combustion product gas.

The burning of Viton A produced a significant amount of CO₂ (13,000 ppm), trace amounts of CO (about 10 ppm), and some COF₂ and CF₄. Surprisingly, HF was not detected in the combustion product gas. This is probably because some HF was lost during sampling and some reacted with the silica in a GC column to form SiF₄.

Table 1 also indicates that the burning of Neoflon (polychlorotrifluoroethylene) produced a significant amount of CO₂ (about 4000 ppm), a small amount of CO (about 70 ppm), some CF₄, HCl, ClO₂ and COCl₂ (phosgene). Phosgene is a very toxic compound; its toxic potency (LC₅₀ = 5 ppmv) is about 28 times that of HCN (LC₅₀ = 140 ppmv).² Clearly, this compound should be the major concern for the burning of Neoflon.

2. Determining the Composition of Combustion Product Gas Using the NASA-Lewis Equilibrium Code

It has been realized that the composition of combustion product gas is strongly dependent on the polymer composition. The nine polymers evaluated include two hydrocarbon polymers (PPO and PEEK contain only carbon and hydrogen), three nitrogen-containing polymers (Zytel 42, Buna N, and Vespel SP-21), and four halogenated polymers (Teflon, Neoflon, Viton A, and Tefzel). Tables 2 through 10 list the calculated combustion products of these polymers at 2000 psia. The concentration of combustion products in Tables 2 through 10 is reported as mole fraction. If we view

those product gases as ideal gases, "mole fraction" can be used to indicate the concentration (ppmv) of individual combustion products. For example, 0.04916 mole fraction is equivalent to 49,160 ppmv. The CO₂ concentration in the adiabatic-compression test of five different polymers is in the range of 4240 to 18,200 ppm (see Table 1). The calculated CO₂ concentration shown in Tables 2 through 10 is within the same order of magnitude of those from the adiabatic-compression tests (see Table 1). Hence, the accuracy of the calculated composition of combustion product gas should be reasonable.

2.1 Burning of Hydrocarbon and Nitrogen-containing Polymers

Tables 2 and 3 indicate that the combustion of hydrocarbon polymers (PPO and PEEK) in a high-pressure oxygen system is very complete. The combustion products are CO₂, H₂O and O₂ the equilibrium temperature (up to 200 °C) has no effect on the composition of combustion products. Since the CO₂ is not considered a toxicant, the combustion product gas generated from the burning of PPO and PEEK is essentially nontoxic.

The calculated combustion products of three nitrogen-containing polymers (Zytel 42, Buna N, and Vespel SP-21) are shown in Tables 4, 5, and 6 respectively. The results suggest that the combustion of nitrogen-containing polymers in a high-pressure oxygen system is also very complete. The nitrogen atoms in polymers are totally converted to nitrogen gas; HCN, NO, NO₂, and N₂O are not found in the combustion product gas. This result is slightly different from the adiabatic-compression test of Buna N. In that test, some NO was detected in the combustion product gas. It is also found that the equilibrium temperature (up to 200 °C) has no effect on the composition of combustion products.

2.2 Burning of Halogenated Polymers

Tables 7, 8, 9, and 10 show the calculated combustion products of Teflon, Neoflon, Viton A, and Tefzel, respectively. It can be seen that CF₄ and COF₂ are the common combustion products for the burning of Teflon, Neoflon, and Viton A.

COF₂ is a medium-toxic compound; its toxicity (LC₅₀ = 360 ppmv) is about ten times that of CO (LC₅₀ = 3760 ppmv).² Figure 2 shows the effect of equilibrium temperature on the COF₂ concentration of four fluorinated polymers. The COF₂ concentration is increased as the equilibrium temperature increases, and is in the range of 0 to 300 ppmv. It appears that the COF₂ concentration is material dependent and that a halogenated polymer having more fluorine in its monomer produces more COF₂. Hence, Teflon produces more COF₂ than Neoflon, and Neoflon produces more COF₂ than Viton A. The COF₂ concentration for Tefzel is below 1 ppm.

CF₄ is generally not considered a toxicant; its toxicity (LD₅₀) is not yet established. However, inhalation of high concentrations of vapor may cause heart irregularities and unconsciousness.⁹ Figure 3 shows the effect of equilibrium temperature on the CF₄ concentration of four halogenated polymers. The CF₄ concentration decreases slightly as the equilibrium temperature increases. The CF₄ concentration is in the range of 0 to 8890 ppm. Figure 3 also indicates that the CF₄ concentration is material dependent. Teflon produces more CF₄ than Neoflon, and Neoflon produces more CF₄ than Viton A. The CF₄ concentration for Tefzel is below 1 ppm.

HF is an acid gas; its LD₅₀ is 966 ppmv.² The burning of halogenated polymers having hydrogen and fluorine produces HF. For Teflon and Neoflon, if there is some moisture around the burning environment, HF will be present in the combustion product gas. It is reported that HF concentration decays very quickly in the presence of absorptive surfaces and water droplets at temperatures below 100 °C.¹⁰ Figure 4 shows the effect of equilibrium temperature on the HF concentration of Viton A and Tefzel, and the concentration remains about the same in the temperature range of 66 to 200 °C. The HF concentration of Viton A and Tefzel is about 13,500 ppmv and 27,800 ppmv, respectively. Table 1 shows that the HF concentration in the combustion product gas of Viton A is not detected. Obviously, the HF concentration decays very quickly in the combustion product gas produced from a real adiabatic-compression test.

Neoflon (polychlorotrifluoroethylene) is a halogenated polymer that contains not only fluorine, but also chlorine. Table 8 shows that a significant amount of Cl₂ is produced for the burning of Neoflon in a high-pressure oxygen system. Figure 5 presents the effects of equilibrium temperature and oxygen pressure on the Cl₂ concentration. It can be seen that the equilibrium temperature has no effect on the Cl₂ concentration and the Cl₂ concentration is decreased as the oxygen pressure increases. The combustion product gas of Neoflon in a real adiabatic-compression test contains HCl, ClO₂, and phosgene (see Table 1). This result is quite different from that of the calculated data. Apparently, the burning of Neoflon is much more complicated in the real adiabatic-compression test.

3. Some Thoughts on Toxicity Assessment of Combustion Product Gas

Tables 2 through 10 indicate that oxygen is the major component in the combustion product gas of different polymers. Combustion product gases consist of 93 to 98% of oxygen. Since the toxicity of combustion product gas (LD₅₀) is determined primarily for the "air-based" gas mixtures, the use of the reported LD₅₀ to assess the toxicity of the "oxygen-enriched" combustion product gas may not be appropriate. The high oxygen concentration may have a "smoothing" effect on the toxicity.

It has been stated in the Materials and Test Method section, that the amount of oxygen reacted with polymers was 0.067 to 0.117 moles. These amounts of gas represent 1.64 to 2.86 L of gas at 14.7-psia pressure. The gas flow rate in a medical cylinder is usually about 2 to 15 L/min, depending on the doctor's prescription. Therefore, in the worse case (i.e., 2.86 L gas and 2 L/min flow rate), the exposure time for the patient is only about 1.43 min. It is suggested to give more consideration to the "short exposure time" when one attempts to assess the toxicity of combustion product gas produced from the burning of polymers in high-pressure oxygen systems.

SUMMARY

This study investigates the toxic combustion product testing of polymers used in high-pressure oxygen systems. The preliminary results can be summarized as follows:

1. This study has demonstrated that the NASA pneumatic-impact test system can be used to perform toxic combustion products testing of polymers.

2. Combustion of hydrocarbon- and nitrogen-containing polymers in high-pressure oxygen systems is relatively complete. Toxicity of the combustion product gas is presumably much lower than that of combustion product gas produced from ambient-pressure oxygen (or air) environments.
3. Halogenated polymers, such as Teflon, and Viton A produce COF₂, and Neoflon produces COF₂ and COCl₂. COF₂ is a medium-toxicity compound and COCl₂ is an extreme-toxicity compound. Both of them should be major concerns for the combustion toxicity assessment.
4. For the combustion toxicity assessment of the polymers used in high-pressure oxygen systems the following factors should be considered: the amount of polymer to be consumed; the high oxygen concentration in the combustion product gas, and the short exposure time.

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Table 1. Combustion products of the adiabatic-compression tests of Zytel 42, Buna N, Viton-A, Neoflon, and Teflon.

Combustion Products	Quantity (ppm)
Zytel 42	
CO	6
CO ₂	18,200
HCN	ND
NO	ND
NO ₂	ND
N ₂ O	ND
NH ₃	ND
Buna N	
CO	7
CO ₂	14,300
H ₂ S	ND
SO ₂	ND
HCN	ND
NO	Detected, unable to quantify
NO ₂	ND
N ₂ O	ND
NH ₃	ND
Viton-A	
CO	11
CO ₂	13000
COF ₂	Detected, unable to quantify
CF ₄	Detected, unable to quantify
HF	ND
H ₂ S	ND
SO ₂	1
Neoflon	
CO	68
CO ₂	4240
COF ₂	ND
CF ₄	Detected, unable to quantify
COClF	ND
Cl ₂	ND
HCl	Detected, unable to quantify
COCl ₂	Detected, unable to quantify
ClO ₂	Detected, unable to quantify
Cl ₂ O	ND
Teflon	
CO	57
CO ₂	6020
COF ₂	ND
CF ₄	Detected, unable to quantify
HF	ND

ND = Non Detect

Note: the adiabatic-compression test of Zytel 42 was performed at 2500-psia oxygen, and that of other polymers was performed at 3500-psia oxygen.

Table 2. Calculated combustion products of PPO (sample weight 0.050 g) at different equilibrium temperatures. Simulated adiabatic-compression test was performed at 2000-psia oxygen.

Combustion Products	Mole Fractions		
	86 °C	100 °C	200 °C
CO ₂	0.04916	0.04916	0.04916
H ₂ O	0.02458	0.02458	0.02458
O ₂	0.92627	0.92627	0.92627

Table 3. Calculated combustion products of PEEK (sample weight 0.050 g) at different equilibrium temperatures. Simulated adiabatic-compression test was performed at 2000-psia oxygen.

Combustion Products	Mole Fractions		
	86 °C	100 °C	200 °C
CO ₂	0.04883	0.04883	0.04883
H ₂ O	0.01542	0.01542	0.01542
O ₂	0.93575	0.93575	0.93575

Table 4. Calculated combustion products of Zytel-42 (sample weight 0.037 g) at different equilibrium temperatures. Simulated adiabatic-compression test was performed at 2000-psia oxygen.

Combustion Products	Mole Fractions		
	80 °C	100 °C	200 °C
CO ₂	0.02889	0.02889	0.02889
H ₂ O	0.02648	0.02648	0.02648
N ₂	0.00241	0.00241	0.00241
O ₂	0.94222	0.94222	0.94222

Table 5. Calculated combustion products of Buna N (sample weight 0.029 g) at different equilibrium temperatures. Simulated adiabatic-compression test was performed at 2000-psia oxygen.

Combustion Products	Mole Fractions		
	86 °C	100 °C	200 °C
CO ₂	0.02591	0.02591	0.02591
H ₂ O	0.00700	0.00700	0.00700
N ₂	0.00078	0.00078	0.00078
O ₂	0.96631	0.96631	0.96631

Table 6. Calculated combustion products of Vespel SP-21 (sample weight 0.050 g) at different equilibrium temperatures. Simulated adiabatic-compression test was performed at 2000-psia oxygen.

Combustion Products	Mole Fractions		
	80 °C	100 °C	200 °C
CO ₂	0.04556	0.04556	0.04556
H ₂ O	0.00825	0.00825	0.00825
N ₂	0.00165	0.00165	0.00165
O ₂	0.94454	0.94454	0.94454

Table 7. Calculated combustion products of Teflon (sample weight 0.060 g) at different equilibrium temperatures. Simulated adiabatic-compression test was performed at 2000-psia oxygen.

Combustion Products	Mole Fractions		
	46 °C	100 °C	200 °C
CF ₄	0.00890	0.00887	0.00876
COF ₂	0.00002	0.00006	0.00030
CO ₂	0.00890	0.00887	0.00876
O ₂	0.98219	0.98219	0.98219

Table 8. Calculated combustion products of Neoflon (sample weight 0.059 g) at different equilibrium temperatures. Simulated adiabatic-compression test was performed at 2000-psia oxygen.

Combustion Products	Mole Fractions		
	46 °C	100 °C	200 °C
CF ₄	0.00563	0.00561	0.00552
COF ₂	0.00001	0.00005	0.00024
CO ₂	0.00939	0.00937	0.00927
Cl ₂	0.00376	0.00376	0.00376
O ₂	0.98121	0.98121	0.98121

Table 9. Calculated combustion products of Viton A (sample weight 0.051 g) at different equilibrium temperatures. Simulated adiabatic-compression test was performed at 2000-psia oxygen.

Combustion Products	Mole Fractions		
	66 °C	100 °C	200 °C
CF ₄	0.00168	0.00167	0.00159
COF ₂	0.00002	0.00004	0.00020
CO ₂	0.02147	0.02146	0.02138
HF	0.01350	0.01351	0.01352
O ₂	0.96333	0.96332	0.96331

Table 10. Calculated combustion products of Tefzel (sample weight 0.050 g) at different equilibrium temperatures. Simulated adiabatic-compression test was performed at 2000-psia oxygen.

Combustion Products	Mole Fractions		
	66 °C	100 °C	200 °C
CO ₂	0.02887	0.02887	0.02886
HF	0.02780	0.02284	0.02286
O ₂	0.95433	0.95430	0.95428

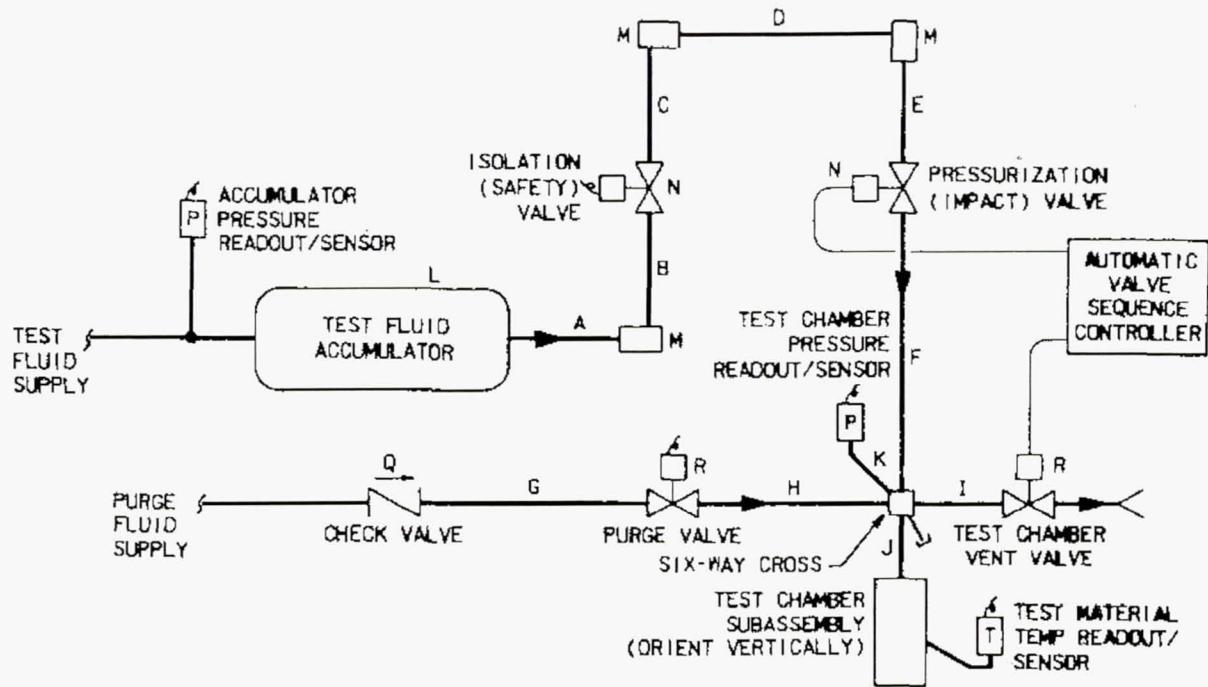


Figure 1. Schematic diagram of the adiabatic-compression (pneumatic-impact) test system.⁶

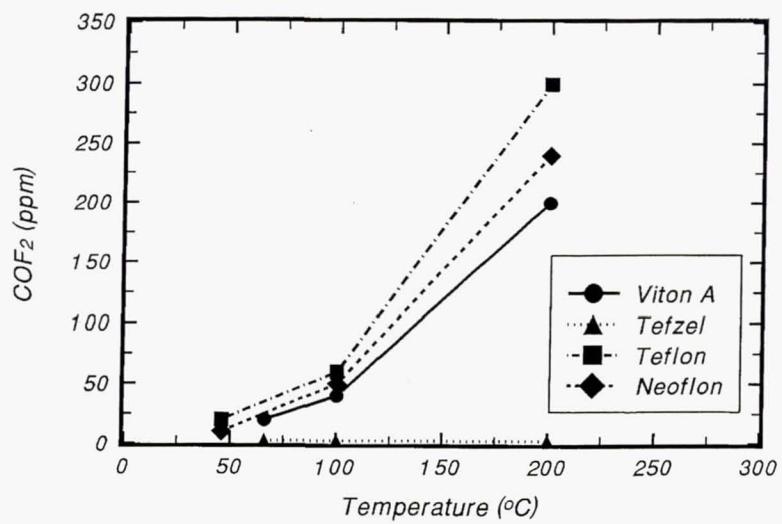


Figure 2. Calculated COF₂ concentrations at different equilibrium temperatures for Viton A, Tefzel, Teflon, and Neoflon in a simulated adiabatic-compression test (2000-psia oxygen).

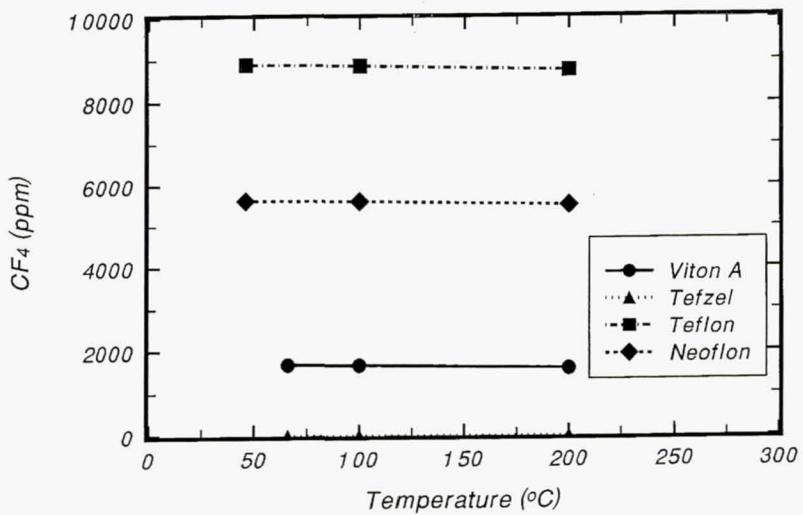


Figure 3. Calculated CF₄ concentrations at different equilibrium temperatures for Viton-A, Tefzel, Teflon, and Neoflon in a simulated adiabatic-compression test (2000-psia oxygen).

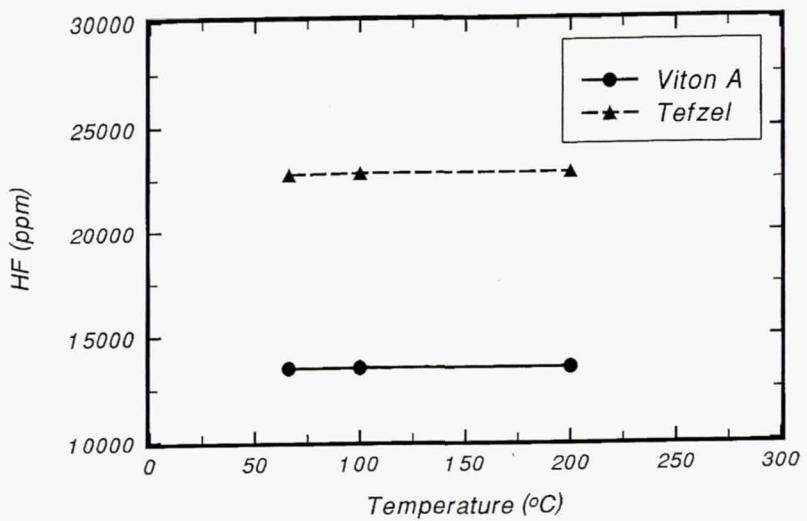


Figure 4. Calculated HF concentrations at different equilibrium temperatures for Viton A and Tefzel in a simulated adiabatic-compression test (2000-psia oxygen).

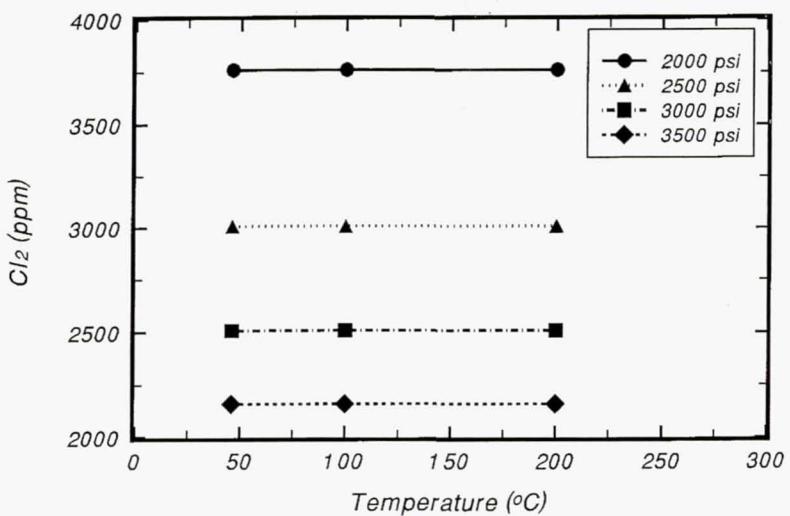


Figure 5. Calculated Cl₂ concentrations at different equilibrium temperatures for Neoflon in a simulated adiabatic-compression test (2000, 2500, 3000, and 3500-psia oxygen).