

## The Benefits of Viton Outgassing

**Summary:** Significant reduction, from a few times  $10^{-7}$  torr-liters  $\text{sec}^{-1} \text{cm}^{-2}$  to a few times  $10^{-10}$  torr-liters  $\text{sec}^{-1} \text{cm}^{-2}$ , in the outgassing rate of Viton "O" rings can be accomplished by vacuum bakeout at 150-200°C for 4 hours. Most of the evolved gas is water vapor. Re-exposure of the material to ambient air for extended periods results in significant, but not complete, reversal of the outgassing. Storage in a dry atmosphere retains most of the benefits of outgassing.

### **Discussion:**

Out of a couple of dozen references about outgassing of materials and specifically Viton, only two were of much use:

1. "Degassing Characteristics of Some 'O' Ring Materials", D. J. Crawley and L. de Csernatony, VACUUM, Volume 14, pp. 7-9.

See Figure 5, Mass spectra obtained during the heating of Viton fluoroelastomer, attached.

"Typical results for Viton are shown for the black loaded, air baked sample of Viton A. figure 5 shows successive mass spectra obtained during the heating of the sample. At 65°C (Fig. 5a) the water vapor peaks have increased markedly, and there is a very slight increase in the carbon monoxide and carbon dioxide peaks. At 100°C, (Fig. 5b) the water vapor peaks are very large, but the carbon oxide peaks have increased only slightly. Upon further heating the water vapor peaks decreased, but at 210°C (Fig. 5c) the carbon oxide peaks, especially carbon dioxide, began to increase significantly. As the temperature continued to increase above this point, the water vapor began to increase again, and the carbon oxide peaks became quite large (Fig. 5d). No other peak heights increased until a temperature close to 290°C was reached. Then a large number of other peaks appeared, some of which have been identified as hydrogen fluoride, vinylidene fluoride monomer, and a number of fluorocarbon fragments (Fig. 5e). At 310°C (Fig. 5f) these additional peaks have become very prominent, and the water vapor and carbon oxide peaks have increased substantially. Upon continued heating to 300°C for 1 ½ hours, all of these peaks have decreased substantially except the hydrogen fluoride peak (Fig. 5g) which has increased. After cooling at room temperature, all peaks returned to the background level. At no time was there any sign of significant hydrocarbon evolution.

When this same sample was reheated without having been removed from the vacuum, the water vapor no longer reached a maximum at 100°C, but continued to increase slowly, along with the carbon oxide peaks, as the temperature increased. The peak heights remained much smaller than that seen during the first heating, indicating that the water vapor and carbon oxide effectively can be baked out of the Viton."

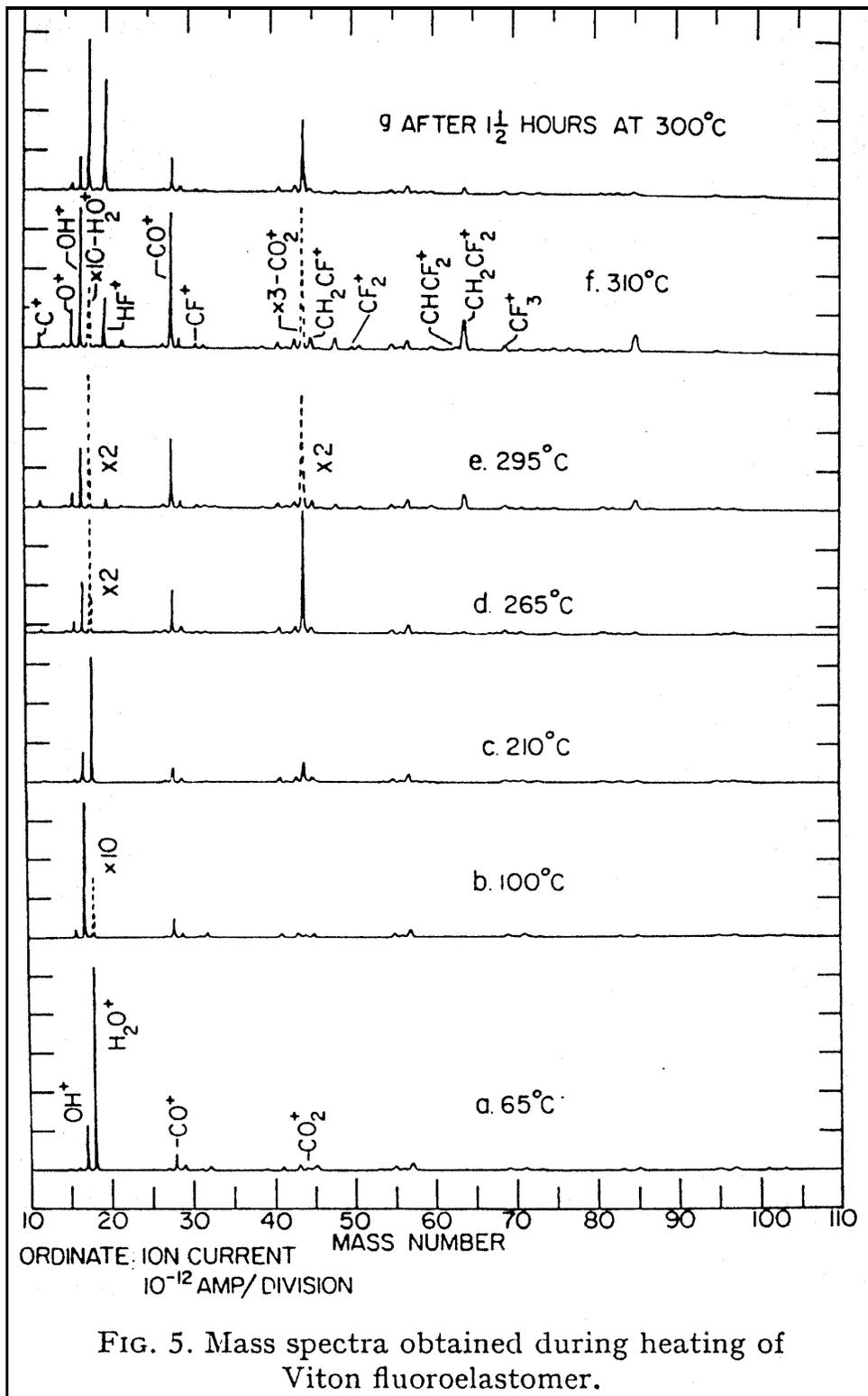


FIG. 5. Mass spectra obtained during heating of Viton fluoroelastomer.

2. "Evaluation of a New Fluoroelastomer as a Gasketing Material for High Vacuum Systems", R. R. Addiss, Jr., L. Pensak and Nancy J. Scott, Transactions of the Seventh National Symposium on Vacuum Technology, 1960, pp. 39-44.

See Figure 2. Comparative degassing rates for nitrile and Viton A "O" rings, and Table I, Summary of Sample Histories, attached.

"Curves of degassing rates against time are presented in Fig.2 for all cases, and the history of the samples is summarized in Table 1" (column 3 in Table 1 shows exposure time to atmosphere, i.e. water vapor. "The curves were taken in numerical order, and the same samples remained in the apparatus from one run to the next.

Figure 2 is mainly self explanatory, but a few salient points are discussed here.

Degassing rates given by the first pumpdown for Viton (curve 1) and nitrile (curve 8) are for new "O" rings straight from the manufacturers, which were merely wiped in tissue paper before test. These runs can be regarded mainly as a method for bringing the samples to a standard condition.

Comparison of degassing rates given by the second pumpdown (curves 2 and 9) for unbaked Viton and nitrile respectively, shows that the Viton is better by about 2:1, an improvement which is not in general sufficient to lead to much lower pressures in unbaked systems, unless comparatively large areas of gasket are exposed.

However, as soon as mild baking is employed, the situation becomes rather different. Curves 3 and 11 show that after baking for 4 hours at 100°C, Viton is better than nitrile by a factor of 50-60 times. In fact curve 4 for baked Viton (16 hour at 100°C) is as good as, or better than, many unbaked metal samples which have been tested. A typical figure for unbaked copper for example, has been found to be  $4 \times 10^{-10}$  torr-liter sec<sup>-1</sup>cm<sup>-2</sup> after 20 hours of pumping, although this rate drops by two or more decades if subject to the same baking treatment.

Further curves for Viton, each after ½ hour atmospheric exposure, indicate how far baking needs to be carried for thorough degassing. Curve 5 (4 hours at 150°C) is better than curve 4 (16 hours at 100°C) by a factor of about two. However, baking Viton for 16 hours at 200°C (curve 6) actually worsens the degassing rate. It is believed that at about 150°C degassing of water vapor becomes virtually complete, and that at higher temperatures more volatile constituents of the material are driven off so rapidly as to contaminate the test chamber before they can be pumped away (brown films characteristic of hydrocarbon contamination were also observed in the ion gauge after this run)."

TABLE I				
<i>Summary of Sample Histories</i>				
Sample material	Curve No.	Exposure (h)	Baking time (h)	Baking temp. (°C)
Viton A	1	—	—	—
"	2	$\frac{1}{2}$	—	—
"	3	$\frac{1}{2}$	4	100
"	4	$\frac{1}{2}$	16	100
"	5	$\frac{1}{2}$	4	150
"	6	$\frac{1}{2}$	16	200
"	7	64	—	—
Nitrile	8	—	—	—
"	9	$\frac{1}{2}$	—	—
"	10	$\frac{1}{2}$	4	50
"	11	$\frac{1}{2}$	4	100
"	12	64	—	—

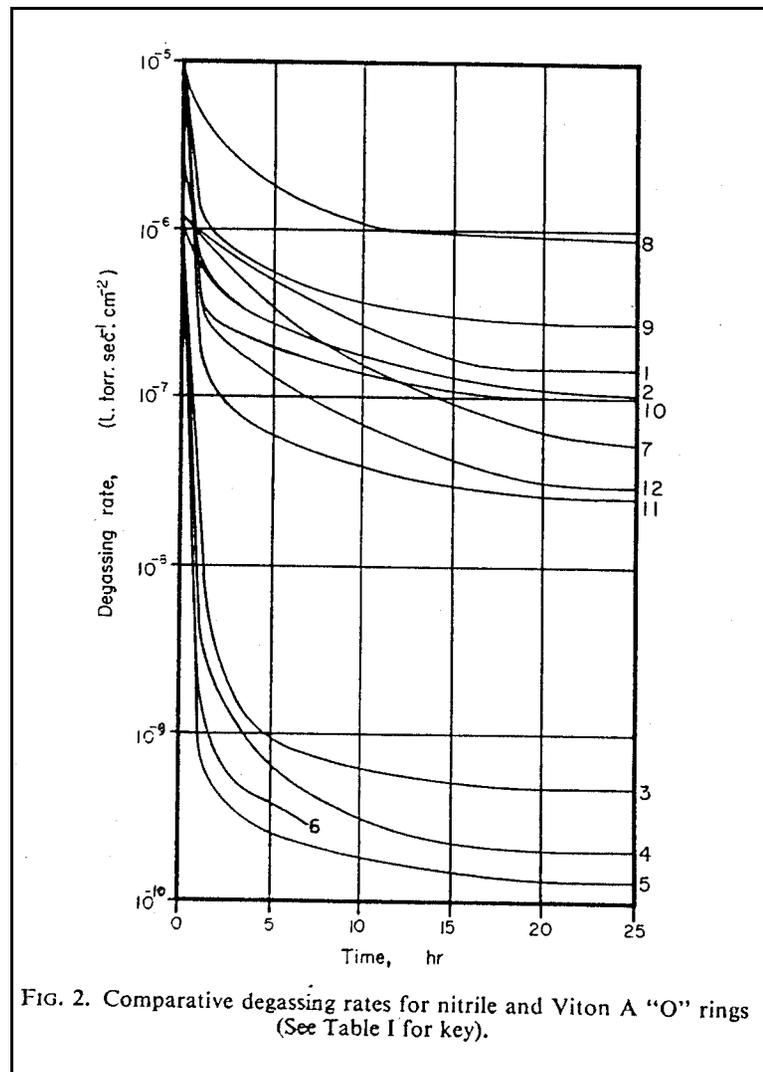


FIG. 2. Comparative degassing rates for nitrile and Viton A "O" rings (See Table I for key).

Other less important quotations, not backed up by experimental data, are shown below:

“Of the available plastics, the best is Viton-A (DuPont). This material is useful down to the middle of the  $1 \times 10^{-10}$  torr range; but if it is used in the as-received condition, it contributes a considerable amount of initial outgassing. This can be largely overcome by a preliminary bakeout at approximately 250° F in a vacuum chamber which removes the unreacted plasticizers, catalysts, and water vapor from the Viton-A, rendering it more useful in ultrahigh-vacuum systems.” (LaPelle: Practical Vacuum Systems)

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“Viton (fluorocarbon). This elastomer has aroused much interest in the vacuum world because of its high temperature resistance. Unlike silicone rubber, it has good mechanical and physical properties.

It has been investigated by Addis *et al.*<sup>(1)</sup> who obtained mass spectra at various temperatures as shown in Fig. 7.(This is reference 1 quoted above). Initially water vapour is evolved but near 210° C the carbon monoxide and carbon dioxide peaks appear and grow with increase in temperature while the water vapour decreases. At just over 300° C the pyrolytic decomposition products appear showing that this is too high a temperature to operate at.

The very low gas permeability...makes it even more attractive but a poor compression set resistance (87 per cent after 70 hours at 220° Centigrade) necessitates frequent replacement if high temperatures are continually used.” (Handbook of Vacuum Physics)

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