X-322-71-165
PREPRINT

NASA TM X- 65571

EXPERIMENTS ON THE EFFECT OF ULTRAVIOLET ON CONTAMINATION IN VACUUM SYSTEMS

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APRIL 1971

GSFC — GODDARD SPACE FLIGHT CENTER —
GREENBELT, MARYLAND

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ABSTRACT

This report describes experiments carried out in an otherwise empty vacuum chamber. Data obtained with the quartz crystal microbalance show that, under ultraviolet irradiation, "clean" and "contaminated" mean different things than in situations that do not involve irradiation. The history of the chamber appears to be of paramount importance, not the pumping mechanism. UV irradiation check for contamination is recommended in critical experiments.

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INTRODUCTION

This report describes a series of experiments designed to discover the effect of irradiating a portion of a vacuum system with ultraviolet (UV) light.

The chamber used in the experiments was a stainless steel cylindrical jar 45 cm in diameter and 51 cm high, evacuated by a 25 cm oil diffusion pump using DC 704 pump oil; this pump was backed by a booster pump, and the train by a mechanical pump. Between the pump and the vacuum area is a commercial chevron baffle and a trap cooled with liquid nitrogen. The source of ultraviolet light was a pen ray lamp*; an ordinary glass slide acted as a collector; and a quartz crystal microbalance measured the deposition. All this equipment was put into a large pyrex beaker, and all items were duplicated outside the beaker except for the UV source (Figure 1). After each experiment, the investigators used both dark-field and light-field optical microscope techniques to examine the slides for deposited material.

DISCUSSION

The fact that the reflectance of a mirrored surface varies so widely after exposure to UV** led the authors to examine the proposition that the variation resulted from a surface deposit of some contaminant in the chamber, rather than from a change in the surface itself. Examination of this hypothesis proved to be easy. An experiment was devised which consisted of placing a quartz crystal microbalance, slides, and UV source inside a pyrex beaker and duplicate items, less the UV source outside the beaker. The entire arrangement was contained in the vacuum chamber and, since the beaker is relatively opaque to UV, UV exposed and unexposed slides could then be studied at the same time.

Anything in the ambient environment of the chamber that would polymerize or deposit on the collector slide under UV irradiation, should then do so only inside the beaker. This assumption proved correct; the microbalance inside the beaker indicated a deposit while the outside microbalance did not.

Inspection of the microscope glass slide proved inconclusive; the presence or absence of a deposit could not be positively established.

*Manufactured by Ultra Violet Products, San Gabriel, Calif.; major spectral content at 2537A
**General Electric Corporation report. Behavior of Materials in Vacuum. Contract NAS5-11057

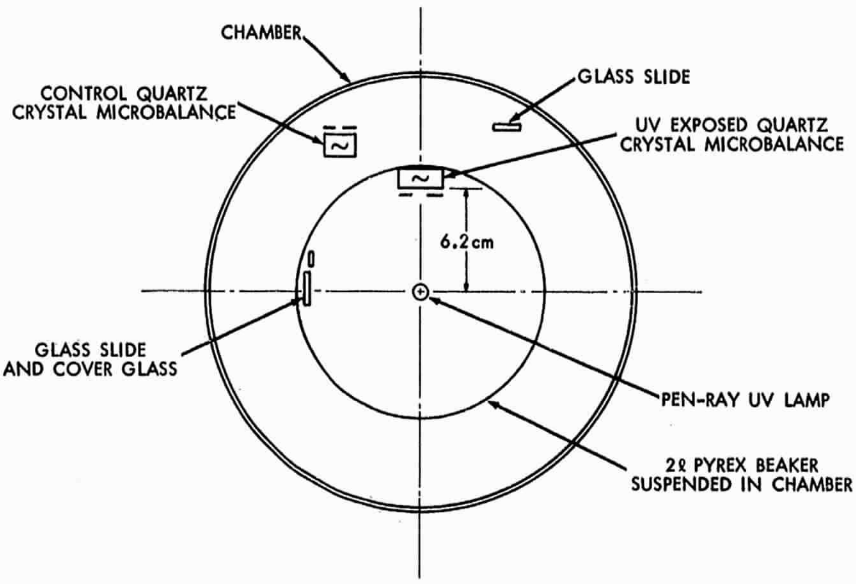

Figure 1. Experiment Layout as Seen From Above

The beaker, which had been accumulating deposit since the beginning of the experiments, was refluxed with methylene chloride, the solution was evaporated to a few milliliters, and infrared analysis was attempted. Although no pattern appeared strongly enough to be identified positively, the siloxane absorption bands characteristic of DC 704 diffusion-pump oil at 12.4μm and 14.2μm were absent.

Acknowledging the possibility that backstreaming from the diffusion pump might be providing the deposit material, the experimenters transferred the UV irradiation portion of the experiment to an ion-pumped system never previously used. This experiment was done two ways. In one way the pen-ray source was inside the vacuum system; in the other, irradiation was through a quartz window from outside the chamber. The microbalance showed absolutely nothing being deposited.

Disassembly of the diffusion pump vacuum system, scrubbing the components with soap and water, and rinsing them with alcohol produced a sharp drop in the rate of deposit. Failure to prevent deposition entirely was probably due to the two labyrinth wells previously used for another purpose, which made thorough cleaning most difficult.

RESULTS

Data produced by these experiments are in terms of crystal beat frequencies (Hz), this beat representing the difference in oscillating frequency between an

Table 1

Differences in Oscillating Frequency Between Exposed and Enclosed Crystals (Hz)

As-Used Vacuum System

Date	Hour	UV - Exposed ΔF	Control Δ
11/14/69	0800	0	0
	1600	35	4
11/17/69	0800	275	20
11/18/69	0800	351	24
11/19/69	0800	423	26
11/20/69	0800	492	27
11/21/69	0800	571	28
11/24/69	0800	726	31

After Cleaning

2/10/70	0800	0	
	1600	9	
2/11/70	0800	13	0
2/12/70	0800	34	2
2/13/70	1400	42	4

Ion-Pumped System

1/22/70	0900	0	
1/23/70	0900	4	
1/26/70	1600	0	
1/27/70	1600	3	

enclosed reference crystal and an exposed active crystal. This beat may be related to mass by calibration, which yields about 1 Hz/10^{-3} μg in the range of operation of this experiment.

The microbalance indicated for the accretion period a weight gain equivalent to 624 Hz; as the active area of the crystal was 0.2 cm^2, the surface density of the deposit was

$$\frac{(624\,\text{Hz})\;(10^{-3}\;\mu\text{g/Hz})}{0.2\,\text{cm}^2} = 3.1\,\mu\text{g/cm}^2$$

Fluctuations in the order of 2 to 3 Hz per day are probably attributable to temperature variations or electronic instability.

Data presented here were recorded with the microbalances stabilized and the UV source on; the readings were at nearly regular intervals over the period of time noted. Table 1 shows the cumulative change in frequency for the time shown in column 1 for both the UV-exposed crystal and the control crystal. The table shows all three experimental situations; the ion pumped system used no control crystal, and the cumulative total change is essentially zero. The figures show some drift due to temperature changes from day to night, and from day to day.

CONCLUSIONS

These experiments show that a chamber which is "clean" for some experiments can be very "dirty" for other experiments. They also strongly suggest that the history of the chamber's use can be much more important to its cleanliness than is the method of pumping used. The experiments offer a relatively simple and very sensitive way to check the cleanliness of a chamber when UV irradiation may be involved; the only special requirements are a pen ray source of UV and a quartz crystal microbalance, both of which are readily obtainable.