19950025384

N95- 31805

CLEANING VERIFICATION BY AIR/WATER IMPINGEMENT

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ABSTRACT

This paper will discuss how the Kennedy Space Center intends to perform precision cleaning verification by Air/Water Impingement in lieu of chlorofluorocarbon-113 gravimetric nonvolatile residue analysis (NVR). Test results will be given that demonstrate the effectiveness of the Air/Water system. A brief discussion of the Total Carbon method via the use of a high temperature combustion analyzer will also be given. The necessary equipment for impingement will be shown along with other possible applications of this technology.

INTRODUCTION

Recent links between chlorofluorocarbon 113 (CFC-113) and upper atmospheric ozone depletion have caused the John F. Kennedy Space Center (KSC) to plan the phase out of all CFC's by 1995. CFC-113 is currently in use at the Kennedy Space Center as a precision cleaning and verification solvent. A CFC-113 rinse is routinely used to verify that small fittings, valves and regulators, large valves, pipes, flex hoses, and tubing meet non-volatile residue (NVR) requirement of less than 11.1 milligram (mg) per square meter (m^2) (1 mg/ft^2) of surface area.

Small parts NVR verification has successfully been met by the use of deionized water and ultrasonic baths (Allen, pp 37-48). Currently, CFC-113 is being phased out and water/ultrasonics is being phased in for small parts only. However, a technique for the verification of large components needed to be identified. Based on the success of small parts with water, and for environmental reasons, it was desirable to attempt large component verification with water.

Presently, KSC processes close to 250,000 piece parts through the component cleaning facility per year. Only 1000 of these parts fall under the heading of large components. These are components too large for the cleaning and verification processes conducted in the cleanroom. Consequently, these parts are cleaned and verified in an area known as Field Cleaning. Current CFC-113 cleaning and verification techniques accounted for the purchase of about 60,000 pounds of solvent during the 1993 calendar year. While 1000 is not a large number of components, the quantity of CFC-113 used for verification is quite large due to their size and configuration.

Items found under the heading of large components are fluid system components: valves, regulators, and relief valves. KSC has a large number of oxidizer systems. These systems, both cryogenic and hypergolic, require a cleanliness level of 11.1 mg/m^2 (1 mg/ft^2) to eliminate any possible fuel to support combustion in a highly oxidizing environment. The variety of large components entering Field Cleaning eliminates the possibility of an automated system due to a lack of similarity among parts. A manual system of cleaning verification by a properly trained technician is required. The system needs to be as insensitive to the variation in the technician-related procedure as possible.

Large components are routinely received in the Field Cleaning Facility that may be contaminated with several families of substances. These families can be summarized as hydrocarbons, silicones, fluorosilicones, and fluorocarbons. Therefore, any test to evaluate a new cleaning and/or verification method must address these contaminants.

IMPINGEMENT VERIFICATION SYSTEM

KSC's answer to this problem is cleaning verification by air/water impingement. Over the last several years development has been progressing on this new verification technique (Dearing, pp 66-77; Melton, pp 642-650; Melton, pp 97-107). The evolution of the design and development process has produced the air/water system shown in Figure 1. The system consists of a regulated gas supply, a pressurized water tank, a water metering and injection device, a flex hose, a nozzle assembly, a catchpan, and associated valves, fittings, and hardware.

The gas supply pressure is used to pressurize the water tank via the water injector. The water injector utilizes two orifices to control the gas and water flowrate. The first orifice provides a pressure differential between the gas stream upstream and downstream of the orifice. This, in turn, pressurizes the water tank. The pressurized deionized water is then injected through a liquid metering orifice just downstream of the gas metering orifice at the point of lowest pressure. The flex hose allows the nozzle to be manipulated freely for work on various components or surfaces. The nozzle assembly consists of one or more supersonic, converging-diverging nozzles.

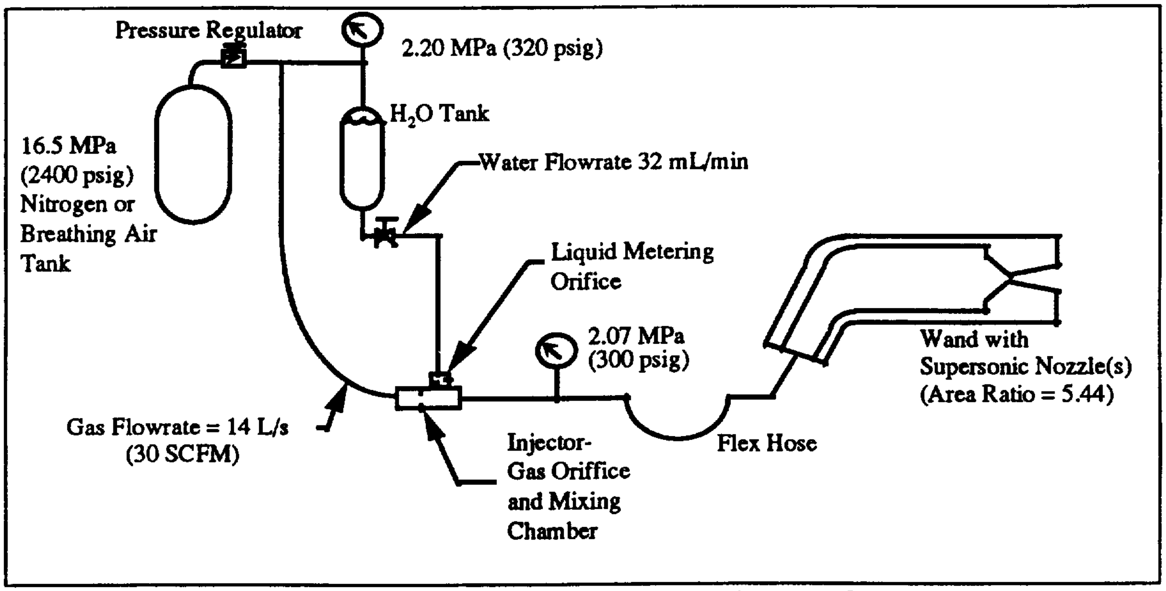

Figure 1 Schematic of Gas Liquid Supersonic Nozzle System

Theory

The nozzles were designed for two-phase flow using the assumption that the nozzle would expand the mixture isentropically. The area ratio was optimized to create the highest velocity with the shortest nozzle geometry, having a ratio of the throat area to the exit area of 5.44. It was found that, if the diverging section was too long, friction would cause a normal shock to form inside the nozzle, reducing the nozzle effectiveness. The exit mach number of the nozzle as designed is 3.2. The water flowrate and gas pressure required were determined empirically. The quantity of water used is small compared with other impingement methods; thus, the concentration of contaminant in the water is high and relatively easy to evaluate.

The solubility of most contaminants is very low in room temperature water. Because a homogeneous suspension is required for Total Carbon (TC) analysis (the technique used to verify NVR level), a technique capable of putting the contaminant into suspension is required. In the small parts verification process, ultrasonic energy is used to place the contaminant into suspension. In this impingement process, the velocity of the water droplets, which are accelerated by the air, provides the energy required to remove the contaminants and place them into a water emulsion. After impingement, the collected water is subjected to TC analysis, which determines the parts per million (ppm) of carbon in the sample.

The Dohrmann DC-190 High Temperature (880 °C) Combustion TC Analyzer was used. In the TC analysis technique, a sample of water/contaminant rinse is introduced into the combustion chamber, converted to carbon dioxide, and measured by a nondispersive infrared detector. The concentration of carbon is measured in parts per million (ppm). This is a simple technique and easily adaptable to a production environment. The major disadvantage is low response to silicone class compounds, where carbon content is dependent on functional groups.. With TC analysis, the concentrations of inorganic and organic carbon can be determined. It is known that the majority of contribution from contaminants entails organic carbon. Therefore, TC is a good representation of the amount of contaminant in a sample.

Procedure

The objective of testing was to obtain both water impingement samples and CFC-113 samples in parallel tests from each of four similarly sized valve bodies. This was accomplished by subjecting each valve body to an initial cleaning process, followed by either an impingement cleanliness verification or a CFC-113 gravimetric NVR cleanliness verification. In both set-ups, the valve body was suspended over a catchpan, which caught the effluent from the process and directed it into a beaker located beneath the pan.

Before testing began, both catchpans were thoroughly cleaned for approximately 15 minutes using the impingement nozzle. Water and CFC-113 blanks were then captured in order to determine a baseline cleanliness of each pan. All valve bodies were immersed in a 60°C (140°F) bath of Brulin 815GD for 30 minutes and then rinsed with 82°C (180°F) water. The valves were then immersed in an ultrasonic bath rinse tank for 15 minutes with a submerged water jet, cooled with an ambient water rinse, and dried with air. Impingement samples were taken after the initial cleaning of each valve to establish a baseline cleanliness level.

Each valve body was evenly contaminated with one of four contaminants at one of three contaminant levels. The four contaminants used were:

Amoco-Rykon II (petroleum grease)
Chevron Molykote (molybdenum disulfide grease)
Dow-Corning DC-55M (silicone grease)
Dupont Krytox 240 AC (fluorinated polyether grease)

Each contaminant was tested at three contamination levels: 11.1, 22.2, 111 mg/m^2 (1, 2, and 10 mg/ft^2). After contamination, the next step was impingement for two minutes followed by a CFC-113 rinse, which removed any contaminant that may have remained on the body. In the case of the equivalent CFC-113 test, the valve body was rinsed with approximately 100 ml of CFC-113 after the contamination occurred.

In summary, the valve bodies were processed through a complete cleaning and each of the two cleanliness verification cycles. One process involved cleaning, contamination, and verification by impingement; the other involved cleaning, contamination, and verification by CFC-113 rinsing. This procedure allowed for direct comparison of the results.

Analysis

After each series of tests, TC analysis and two NVR analyses were performed (one each for the CFC-113 rinse following impingement and the CFC-113 rinse verification tests). In the TC analysis, a 200 microliter (µL) sample was injected into the combustion chamber for processing. A TC reading, in ppm, was obtained. Typically, an average of three to five injection samples was required to obtain a consistent value. The remaining effluent volume was then measured for use in the equivalent NVR (ENVR) calculation. ENVR is the value of a NVR analysis that would have been obtained using conventional CFC-113 rinse methods. The equation was as follows:

$$\mathrm{ENVR} = \frac{\mathrm{Va} * (\mathrm{TC_S} - \mathrm{TC_B})}{\mathrm{Vav} * \mathrm{S} * \mathrm{A}} \qquad \text{(Equation 1)}$$

where

Va = volume, actual collected (mL)
$\mathrm{TC_S}$ = total carbon, sample (ppm)
$\mathrm{TC_B}$ = total carbon, blank (ppm)
Vav = average volume of effluent collected, 45 mL (based on impingement duration, nozzle flowrate)
S = sensitivity, (ppm/mg)
A = area of impinged surface (m^2)

A gravimetric NVR was performed on the CFC-113 rinse that followed impingement to determine if any contaminant remained after the process. This was another check on the removal efficiency of the impingement method.

Results

The sensitivity factor is a measure of the level of responsiveness of the process. Sensitivity values were determined from the following equation:

$$\mathrm{S} = \frac{\mathrm{Va} * (\mathrm{TC_S} - \mathrm{TC_B})}{\mathrm{Vav} * \mathrm{C_{act}} * \mathrm{A}} \qquad \text{(Equation 2)}$$

where: $\mathrm{C_{act}}$ = actual contamination level of valve body (mg/m^2)

Table 1 contains the calculated sensitivities for the contaminants at each of the three contamination levels. From these data, an overall sensitivity (used in Equation 1) was determined. An average value of sensitivity for each contaminant at each level was first calculated and weighed, based on the likelihood of finding it on the actual component. Since the area of primary concern was in the 11.1 mg/m^2 (1 mg/ft^2) range, the overall sensitivity factor for the process was chosen at the 11.1 mg/m^2 (1 mg/ft^2) level. While this will give lower ENVR's for higher contamination levels, it should fail any component having an initial contamination greater that 11.1 mg/m^2 (1 mg/ft^2).

Table -1 Sensitivity vs. Contaminant and Contamination Level

Contaminant	Level (mg/m^2)	Sensitivity (ppm/mg)
Amoco Rykon II	111	0.93
	22.2	1.22
	11.1	2.97
Chevron Molykote	111	1.11
	22.2	1.75
	11.1	3.03
Dow Corning DC-55M	111	1.16
	22.2	2.13
	11.1	4.20
Krytox 240 AC	111	0.12
	22.2	1.18
	11.1	1.21

Table 2 contains a comparison of average ENVR's calculated from the impingement results and NVR values obtained directly from the CFC-113 testing. The ENVR for each contaminant at the three contamination levels was plotted and may be seen in Figures 2 - 6. Three of the four contaminants were readily detected; Krytox 240 AC, which has very low levels of carbon, was not detectable by TC analysis. However, the NVR analyses of the CFC-113 rinse that followed the valve impingement showed that the impingement method effectively removed Krytox 240 AC from the valve body surface.

Table 2 - Average ENVR vs. NVR (mg/0.09m^2)

Contaminant\Level (mg/0.09m^2)	1.0		2.0		10.0	
	ENVR	NVR	ENVR	NVR	ENVR	NVR
Amoco Rykon	0.9	0.7	0.7	1.7	2.7	7.1
Chevron Molykote	0.9	0.7	1.0	1.3	3.3	6.7
DC-55M	1.2	1.0	1.3	1.4	3.4	6.4
Krytox 240 AC	0.4	0.7	0.7	1.4	0.4	7.9

On viewing these data, some scatter will be seen. This is due to the sensitivity of the system to the operator's procedure and environmental conditions. The data presented in this paper were gathered in less than ideal environmental conditions, but are felt to generally be representative of the capabilities of the system. Presently, operations have been moved to a clean room facility where testing is being conducted using the same baths and rinses as the production cleaning operations are using.

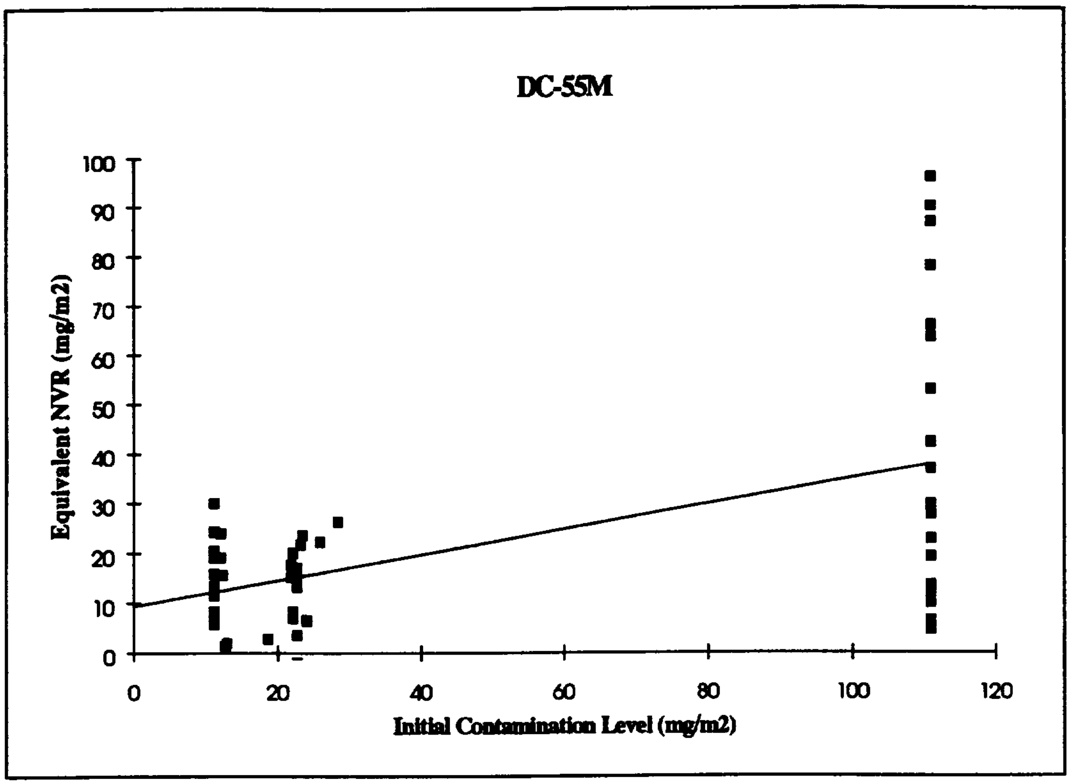

Figure 2 - Initial Contamination Level vs. Equivalent NVR for DC-55M

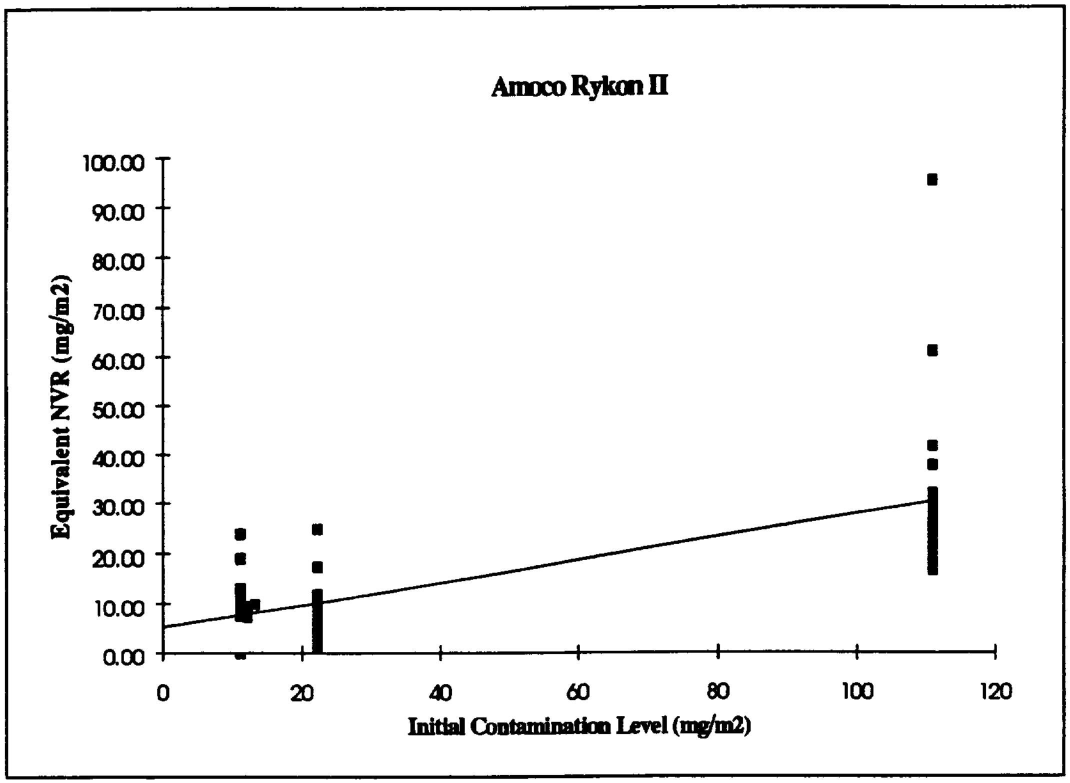

Figure 3 - Initial Contamination Level vs. Equivalent NVR for Amoco Rykon II

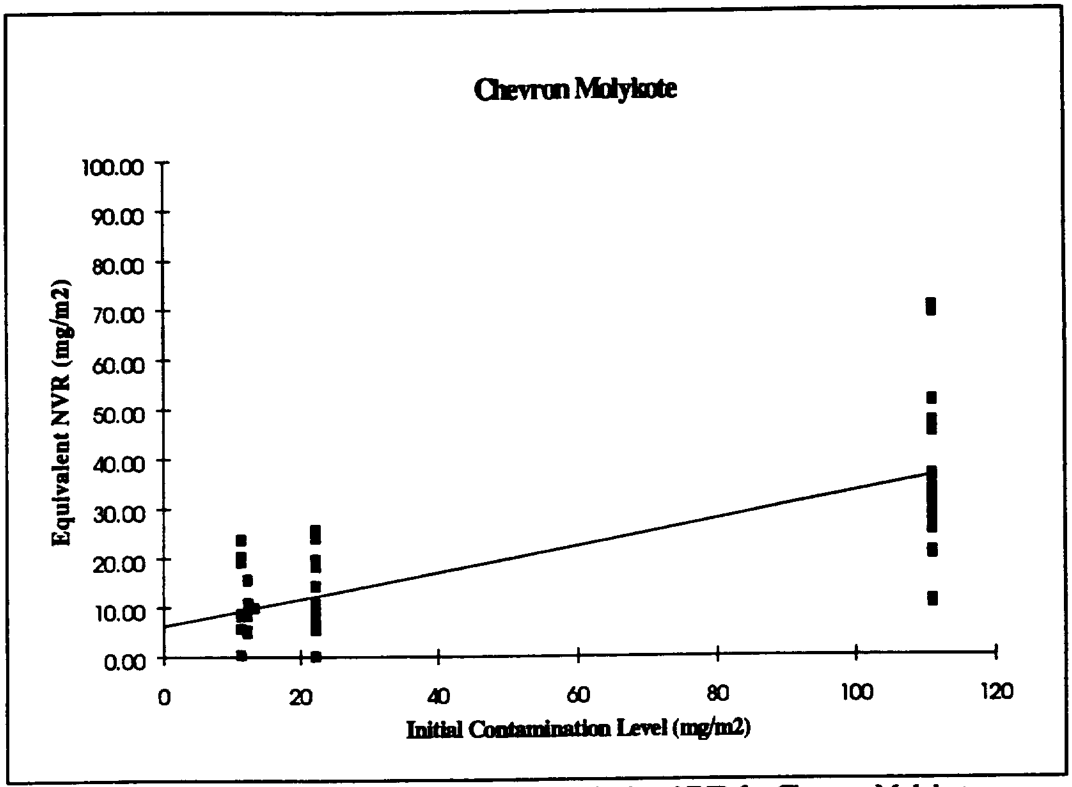

Figure 4 - Initial Contamination Level vs. Equivalent NVR for Chevron Molykote

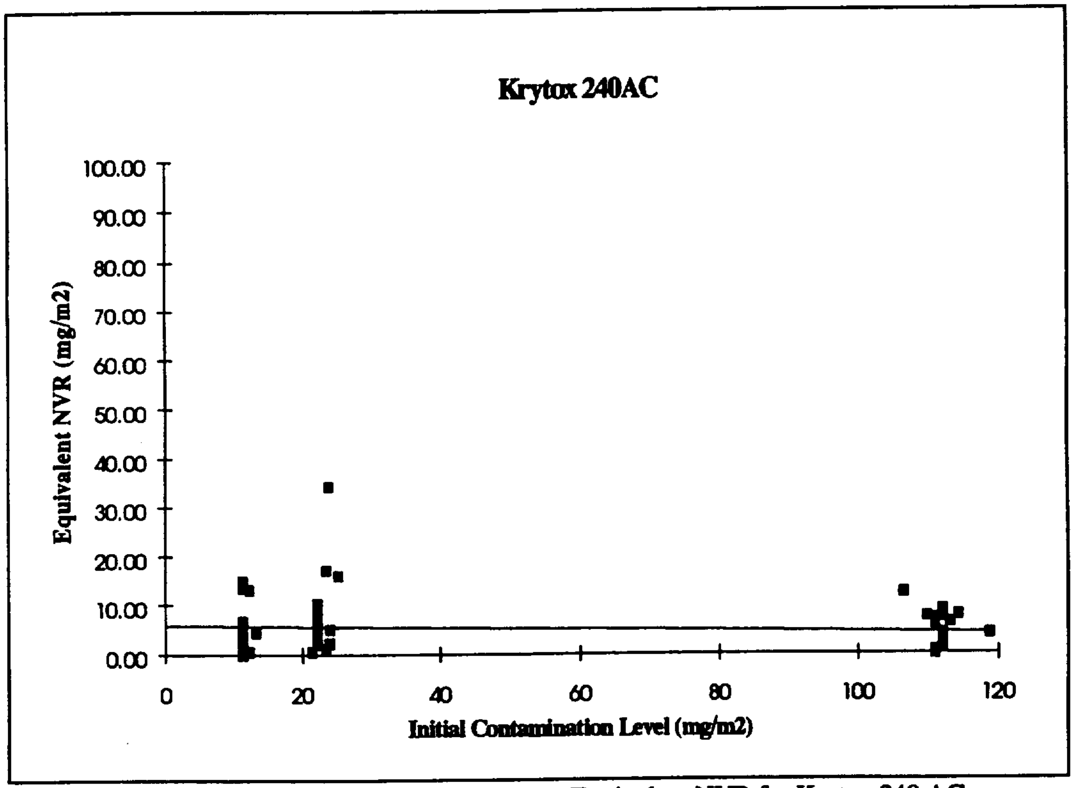

Figure 5 - Initial Contamination Level vs. Equivalent NVR for Krytox 240 AC

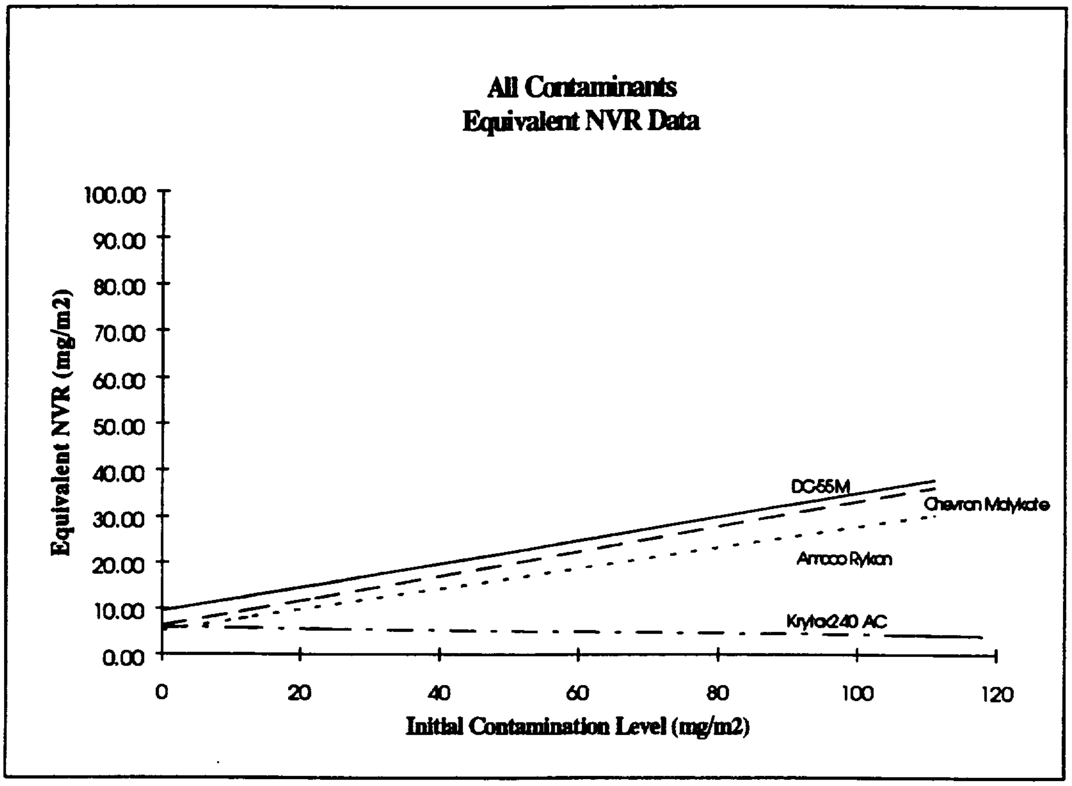

Figure 6

The curves in Figure 6 indicate that, if extrapolated to an initial contamination level of zero, all of the curves intersect the ENVR axis at values above zero. Since it is not possible to completely clean a component to the zero level, and the exact level and content of initial contaminant is unknown, there will be a baseline "noise" level of ENVR.

Table 3 containing ENVR values for the tested valve size has been generated from the data shown in Figure 6 and Equation 1. Using this table, a technician will be able to read an ENVR value based on the component size, TC reading, and actual volume of effluent collected. The technician will then compare the ENVR to the 11.1 mg/m^2 (1 mg/ft^2) pass/fail criterion. A series of tables for different component surface areas will be generated for field usage after future tests are completed.

Table 3 - Example ENVR Calculation Worksheet
Equivalent NVR (mg/0.09m^2)

S = 3.4　　A = 0.9 ft2

Vol (ml)\TC (ppm)	2.0	3.0	4.0	5.0	6.0	7.0	8.0	9.0	10.0	11.0
30	0.4	0.6	0.8	1.0	1.2	1.4	1.6	1.8	2.0	2.2
32	0.4	0.6	0.8	1.0	1.3	1.5	1.7	1.9	2.1	2.3
34	0.4	0.7	0.9	1.1	1.3	1.6	1.8	2.0	2.2	2.4
36	0.5	0.7	0.9	1.2	1.4	1.6	1.9	2.1	2.4	2.6
38	0.5	0.7	1.0	1.2	1.5	1.7	2.0	2.2	2.5	2.7
40	0.5	0.8	1.0	1.3	1.6	1.8	2.1	2.4	2.6	2.9
•	•									•
•	•									•
50	0.7	1.0	1.3	1.6	2.0	2.3	2.6	2.9	3.3	3.6

Differing methods of applying this impingement technique are being developed. Rather than use a wand with a single nozzle, devices that automatically rotate and contain multiple nozzles are under development. These new tools should reduce the errors induced by differences in operators or procedures. Such new tools have the potential for greatly reducing the amount of time that is required to sample a component while greatly increasing the sensitivity to contamination on the component being verified.

CONCLUSIONS

The results of the testing performed to date have shown that the Impingement Verification System (IVS) will be a successful replacement for the traditional CFC-113 rinse method for cleanliness verification for large components. Three of the four contaminants tested were able to be detected using IVS and TC analysis. Krytox 240 AC was not detectable, however, it is oxidizer-compatible and could not support combustion, and, therefore, not of concern.

It must be emphasized that although this method is successful for the particular application at KSC for which it was developed, it must be appropriately tailored in order to be used in other applications. The system in which it would be applied, the types of contaminants, and the contamination level all play an important role in the determination of the sensitivity factor.

ACKNOWLEDGMENTS

The authors are sincerely grateful to the NASA Mechanical Engineering Division, NASA Materials Science Laboratory, I-NET Incorporated, and Wiltech Corporation for their invaluable contributions to the project. Permission by NASA, Kennedy Space Center, to publish the test data is gratefully acknowledged.

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