# The Use of Liquid Isopropyl Alcohol and Hydrogen Peroxide Gas Plasma to Biologically Decontaminate Spacecraft Electronics

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#### Abstract

Legitimate concern exists regarding sending spacecraft and their associated hardware to solar system bodies where they could possibly contaminate the body's surface with terrestrial microorganisms. The NASA approved guidelines for sterilization as set forth in NPG 8020.12C, which is consistent with the biological contamination control objectives of the Committee on Space Research (COSPAR), recommends subjecting the spacecraft and its associated hardware to dry heat—a dry heat regimen that could potentially employ a temperature of 110°C for up to 200 hours. Such a temperature exposure could prove detrimental to the spacecraft electronics. The stimulated growth of intermetallic compounds (IMCs) in metallic interconnects and/or thermal degradation of organic materials composing much of the hardware could take place over a prolonged temperature regimen. Such detrimental phenomena would almost certainly compromise the integrity and reliability of the electronics. Investigation of sterilization procedures in the medical field suggests that hydrogen peroxide  $(H_2O_2)$  gas plasma (HPGP) technology can effectively function as an alternative to heat sterilization, especially for heat-sensitive items. Treatment with isopropyl alcohol (IPA) in liquid form prior to exposure of the hardware to HPGP should also prove beneficial. Although IPA is not a sterilant, it is frequently used as a disinfectant because of its bactericidal properties. The use of IPA in electronics cleaning is widely recognized and has been utilized for many years with no adverse affects reported. In addition, IPA is the principal ingredient of the test fluid used in ionic contamination testers to assess the amount of ionic contamination found on the surfaces of printed wiring assemblies. This paper will set forth experimental data confirming the feasibility of the IPA/H<sub>2</sub>O<sub>2</sub> approach to reach acceptable microbial reduction (MR) levels of spacecraft electronic hardware. In addition, a proposed process flow in which both IPA liquid and HPGP are utilized will be presented in Section 7.0 Future Work. A list of acronyms and chemical symbols used throughout this paper is given in Section 9.0 Acronyms and Chemical Symbols.

#### Background

Legitimate concern exists regarding sending spacecraft and its associated hardware to a planet where the spacecraft and/or its associated hardware could possibly contaminate the planetary surface with terrestrial microorganisms, thus vitiating the search for extraterrestrial life forms. This has resulted in increasing attention to ensure an acceptable level of microbial reduction (MR) of the entire spacecraft and its attendant hardware prior to leaving Earth to ensure that they do not inadvertently contaminate any other planetary surface. All future missions to promising cosmic destinations, such as Mars and Europa, will need an acceptable level of microbial reduction (MR) of the entire spacecraft, including the electronics, prior to leaving Earth.

The NASA approved guidelines for sterilization as set forth in NPG 8020.12C [1], which is consistent with the biological contamination control objectives of the Committee on Space Research (COSPAR), recommends subjecting the spacecraft and its associated hardware to dry heat. To achieve bulk spacecraft Dry Heat Microbial Reduction (DHMR), the dry heat process contemplated for sterilizing a spacecraft and its associated hardware could employ a temperature of 110°C for up to 200 hours. Such a temperature regimen is likely to prove detrimental to the spacecraft electronics. If such a bake-out is performed, it is anticipated that the growth of intermetallic compounds (IMCs) and/or thermal degradation of organic materials composing much of the hardware will take place. Potential interactions could occur from a dry heat treatment because of the complex material combinations of each electronic component, including the printed wiring board, board plating, solder or adhesive, and the metallization finish of the components. A 1967 preliminary study investigated the effect of thermal sterilization on microelectronics for the Voyager program. Subassemblies consisted of magnesium and aluminum substrates with epoxy glass circuit boards and polyurethane conformal coating, ceramic substrates in metallic packages, and potted metal-framed assemblies. All of these had components such as resistors, polystyrene dielectric and wet tantalum electrolytic capacitors, and transistors. The assemblies were subjected to six cycles of 92 hours at 135°C. The results proved

that failures and material degradation can occur, e.g. failure of the polystyrene dielectric capacitors occurred because of the dielectric film breakdown, failure of the wet tantalum electrolytic capacitors due to the conditions which exceeded their 85°C temperature rating, cracking of potting material, and polyurethane and filleting material discoloration [2]. Electronics today are significantly different. Today the main thrust is on surface mount devices (SMDs), especially active SMDs with high-count inputs/outputs (I/Os) and very small passive chip SMDs. In addition, high density chip-on-board (COB) with bare die, and new packaging materials, e.g., lead-free solder alloys, are coming into prominence. Potential reactions occurring during microbial reduction, leading to material degradation and failures, will prove unacceptable to the integrity of modern electronics.

To avoid such a scenario, an alternate sterilization process to DHMR was sought. The chief focus of this paper lies in methods of protecting the electronic hardware from the adverse degradation anticipated with extended dry heat. Therefore, the principal discussion centers on an alternative method of achieving an acceptable level of sterilization while still preserving the integrity of the electronic hardware.

### Introduction

Investigation of microbial reduction or a sterilization treatment in the medical field suggests that hydrogen peroxide  $(H_2O_2)$  in moderate to high concentrations can effectively function as an alternative to DHMR, especially for items that are heat sensitive. The use of hydrogen peroxide  $(H_2O_2)$  is cited as a microbial reduction method in the *Planetary Protection Design Guidelines* (JPL D-18635) [3]. Within [4], the terms "microbial reduction" and "sterilization" are used interchangeably. Recent investigation also suggests that the hydrogen peroxide process is a viable option [5], and work is underway to validate its use [6]. This report builds on this research that HPGP technology would prove to be an effective microbial reducing agent for the electronic hardware in a spacecraft.

Hydrogen peroxide, in the form of HPGP, is an oxidizing agent that affects sterilization by oxidation of key cellular components (e.g., membrane lipids, DNA, and other essential constituents) [7, 8]. These chemical interactions at multiple biologically important reaction sites are believed to be responsible for inactivating the microorganisms. Gas plasmas are highly ionized gases composed of ions, electrons and neutral particles. The plasma breaks down the peroxide into a "cloud" of highly energized species that recombine, ultimately turning the hydrogen peroxide into water and oxygen. The maximum exposure temperature in a hydrogen peroxide sterilization unit is less than 60°C and actual exposure time to HPGP is less than one hour. This temperature/time combination is much more benign to the electronics than the temperature/time regimen of the dry heat process.

In addition to HPGP, treatment with isopropyl alcohol (IPA) in liquid form followed by IPA vapor prior to exposure of the hardware to HPGP is postulated to add an additional microbial reduction benefit. IPA is frequently used as a disinfectant because of its bactericidal properties. The use of IPA in electronics cleaning is widely recognized and has been utilized for many years with no adverse affects reported. In addition, IPA is the principal ingredient of the test fluid used in ionic contamination testers to assess the amount of ionic contamination found on the surfaces of printed wiring assemblies (PWAs) [9].

The treatment of electronic hardware with isopropyl alcohol (IPA) as an initial step prior to full sterilization by HPGP would take place in a specially constructed totally enclosed machine utilizing the latest in vacuum technology, known as "zeroemission" technology. This is to prevent IPA solvent losses to the atmosphere. This type of system has received approval by the South Coast Air Quality Management District (SCAQMD) for use in Southern California. The use of a vacuum to reduce the boiling point of the IPA is also considered beneficial. Although at standard atmospheric pressure IPA boils at 82°C, under vacuum the boiling of the liquid will be around 60°C-65°C. The entire process is not projected to be any longer than 15-20 minutes. Again, this process results in a benign temperature/time exposure.

Hence, the following two processes are suggested to achieve effective MR of electronic hardware: (1) a "zero-emission" machine using IPA as a cleaning/disinfecting agent followed by (2) HPGP sterilization performed in a suitable system. HPGP could affect the functionality of the electronic components if applied before or after conformal coating, which covers the electronic components. The primary failure types for HPGP treatment would be driven by the nature of reaction since HPGP is known as an oxidizing agent and decomposes into water and oxygen. These failures might include the potential oxidation of components and/or corrosion of metals and polymer degradation. Local pinholes in the conformal coating might also allow a pathway for vapor penetration and allow for oxidation, corrosion, and degradation.

# Objectives

The main objectives of this feasibility investigation are to determine the following:

- Bioburden of the control test vehicle
- Effect of the IPA on bioburden reduction
- Effect of HPGP on sterilization efficacy
- Effect of HPGP on the functionality and material compatibility of the electronic components

# Methodology

# Description of test vehicles (TVs) and testing performed

The following test vehicles (TVs) were used in this investigation:

- Populated electronic boards (PWAs) with Parylene C® conformal coating
- Bare printed wiring boards (PWBs) without conformal coating and with immersion silver plating over copper
- Unpackaged, silicon (Si) die with strain gauge and gold (Au) backside metallization
- Populated printed wiring boards, a.k.a. printed wiring assemblies (PWAs), were utilized after having been thermal cycled from -120°C to 85°C in another investigation

Table 1 shows the description of the test vehicles (TVs) and the testing performed in this investigation.

Table 1 - Description of Test Venteres (1 V s) and Testing Terror med				
TV Description	Testing and number of TVs per test			
Populated boards (PWAs) with	Bioburden estimation (4 TVs)			
Parylene C conformal coating	Bioburden estimation after IPA cleaning (2 TVs)			
	Sterilization efficacy and material compatibility in HPGP (2 TVs)			
Bare (without conformal coating)	Bioburden estimation (4 TVs)			
PWBs with immersion silver plating	Bioburden estimation after IPA cleaning (2 TVs)			
	Sterilization efficacy in HPGP (2 TVs)			
Silicon (Si) die with strain gauge and	Bioburden estimation (2 TVs)			
Au backside metallization (without	Bioburden estimation after IPA cleaning (1 TV)			
conformal coating)	Sterilization efficacy (2 TVs)			
Aluminum boat (board holder)	Bioburden estimation (1 TV)			

### Table 1 - Description of Test Vehicles (TVs) and Testing Performed

# **Bioburden estimation**

The term "bioburden" is commonly used to describe the population of viable microorganisms present on a material or product. It is not possible to determine the exact bioburden; therefore, in practice a viable count is determined using a defined technique [10]. An appropriate swabbing method was chosen for the electronics to facilitate removal of microorganisms from the irregularly shaped areas. The entire surface of each test vehicle was wiped with the swab moistened with United States Pharmacopeia (USP) Fluid D [11]. The swab head was then transferred to a tube containing 25 milliliter (mL) of Fluid D and vortexed for three minutes. The resulting Fluid D was filtered through a 0.45 micrometer ( $\mu$ m) membrane filter and rinsed with 100 mL of Fluid D. The filters were subsequently placed on tryptic soy agar (TSA) plates. The plates were incubated at 30-35°C for three days and at 20-25°C for five additional days. To determine the recovery efficiency of this method, a repetitive recovery method was performed as shown in Figure 1. The correction factor was calculated for each test vehicle type using the data from repetitive recovery method to compensate for the incomplete removal of microorganisms from the test vehicles. Gram stain was performed to determine the predominant colony types.



Figure 1 - Recovery Method Evaluation for Bioburden Estimation

### **Bioburden estimation after IPA cleaning**

During this experiment, a manual IPA cleaning process was performed. The electronic test vehicles were immersed in 100% IPA and shaken intermittently for a total of ten minutes. The test vehicles were then removed from IPA and air-dried. The swabbing method, described above in 4.2, was performed, and the correction factor was applied for the bioburden estimation.

# Sterilization efficacy evaluation in HPGP

*Geobacillus stearothermophilus* (ATCC 7953) spores have been determined to be the most resistant microorganism to sterilization by HPGP [12] and are used as the biological indicator (BI). BI disks containing at least  $10^6$  spores of *G. stearothermophilus* were used to evaluate the sterilization efficacy of the HPGP for electronics. Electronics with BI disks were packaged in Tyvek® pouches. They were then processed in the industrial HPGP sterilizer under the proposed half-cycle conditions (with two injections). After cycle completion, the BI disks were transferred to individual TSB tubes and incubated at 55-60°C for 14 days.

# Functionality and material compatibility in HPGP

The functionality and material compatibility of the populated PWAs were performed with the test vehicles processed through additional two full cycles after being used for the sterilization efficacy evaluation. The proposed full cycle for both PWAs had four injections in the industrial HPGP sterilizer. Thus, these test vehicles received a total of ten injections or 80 minutes of exposure to the HPGP at 53°C. Continuity or resistance values (measured in ohms) were measured on the PWAs with Parylene C conformal coating using a Fluke 87 III True RMS Multimeter. The values after HPGP treatment were compared to those before HPGP treatment. The 20 mil aluminum (Al) wire bond with Parylene C coating area on the PWA was cross-sectioned, potted using an Allied High Tech Epoxy set resin, and analyzed using a LEO Zeiss Supra 50 VP scanning electron microscope (SEM) with an IXRF Systems Energy Dispersive Spectrometer (EDS) elemental X-ray Analyzer. Elemental X-ray data were plotted using Sigma Plot version 9.0 [13].

# **Results and Discussion**

#### Bioburden of the control test vehicles

The numbers of colonies from bioburden estimation of the unprocessed electronics are tabulated in Table 2. The overall bioburden levels of the electronics were lower than expected. A suitable explanation could be that the electronics had been previously employed in a thermal cycling experiment, namely, from  $-120^{\circ}$ C to  $+85^{\circ}$ C. It is interesting to note that one of the bare electronic boards and aluminum boat had higher counts compared to the rest of the test vehicles, probably resulting from extensive handling of them, thus increasing the bioburden levels. Gram stain results indicated that the predominant bioburden isolates were gram-positive cocci (GPC) and gram-positive rods (GPR). All GPR were spore-forming *Bacillus* species as the spores were visible during the phase contrast microscopy. Skin is considered to be a primary natural habitat of common GPC. It is generally accepted that the primary habitat of the majority of *Bacillus* species is the soil. From soil, aerobic spore formers can contaminate everything by dust or other means [14]. The percent recovery was calculated for the recovery efficiency after the first swabbing procedure, recommended for the routine bioburden estimation. The correction factor was calculated using the lowest percent recovery observed for each TV type to take the conservative approach for the bioburden estimation. The calculated correction factors are summarized in Table 3.

TV torse	Replicate	Rec Colony Fo	overed coun orming Units	Total	% Recovery	
т у туре	TV #	After 1st recovery	After 2nd recovery	After 3rd recovery	(CFUs)	(after 1st recovery)
Dopulated boards (DWAs)	1	8	2	5	15	53
with Derulena C conformal	2	8	4	0	12	67
with Farylene C conformation	3	7	10	2	19	37
coating	4	3	2	0	5	60
Bare (without conformal coating) PWBs with	1	6	3	0	9	67
	2	9	6	4	19	47
	3	7	3	0	10	70
minersion silver plating	4	77	6	0	83	93
Silicon (Si) die with strain	1	0	0	0	0	NA
gauge and Au backside metallization (without conformal coating)	2	0	0	0	0	NA
Aluminum boat (board holder)	1	121	6	1	128	95

# Table 2 - Bioburden Data of the Control Test Vehicles (TVs)

# Table 3 - Calculation of Correction Factor

TV type	Average % recovery	Range of % recovery	Correction factor
Populated boards (PWAs) with Parylene C conformal coating	54%	37% - 67%	2.7 (=100/37)
Bare (without conformal coating) PWBs with immersion silver plating	69%	47% - 93%	2.1 (=100/47)
Silicon (Si) die with strain gauge and Au backside metallization (without conformal coating)	NA	NA	NA
Aluminum boat (board holder)	NA	NA	1.3 (=100/95)

# **Bioburden estimation after IPA cleaning**

The data in Table 4 show a significant reduction on the bioburden level after IPA cleaning. As expected—only GPR and mold without any GPC—were recovered after IPA cleaning. It is well known that IPA possesses general antimicrobial properties but without sporicidal effect. The extent of the bioburden reduction of three types of electronics after IPA cleaning is shown in Figure 2.

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<b>Т</b> V Туре	Replicate TV #	Bioburden count (CFUs)	Correction factor	Corrected bioburden count (CFUs)	Isolate types
Populated boards (PWAs) with	1	0	2.7	0	_
Parylene C conformal coating	2	0	2.7	0	-
Bare (without conformal	1	2	2.1	4	GPR
coating) PWBs with immersion silver plating	2	4	2.1	8	GPR, mold
Silicon (Si) die with strain gauge and Au backside metallization (without conformal coating)	1	0	NA	0	-

Table 4 -	Bioburden	Estimation	After	IPA	Cleaning
1 4010 1	Diobaiach	Louinteron	1 11001		Cicaning



Figure 2 - Effect of IPA Cleaning on Bioburden Reduction

# Sterilization Efficacy Evaluation in HPGP

All BI disks from the packaged electronics were sterile after the incubation period when processed through the proposed halfcycle conditions (with two injections, or 16 minutes of exposure time). The BI results from Table 5 indicate at least a six spore log reduction of *G. stearothermophilus* under the half-cycle conditions. Therefore, it is feasible to obtain the sterility assurance level (SAL) of  $10^{-6}$  for the packaged electronics under the full cycle conditions (with four injections or 32 minutes of exposure time) in the industrial HPGP Sterilizer. Note that the use of BI with at least  $10^6$  spores of *G. stearothermophilus* provides an ample safety margin for the process lethality considering the actual average bioburden levels were less than 100 CFUs/TV or even lower after IPA cleaning.

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TV type	BI results (# nonsterile/ # tested)			
Populated boards (PWAs) with Parylene C conformal coating	0/2			
Bare (without conformal coating) PWBs with immersion silver plating	0/2			
Silicon (Si) die with strain gauge and Au backside metallization (without conformal coating)	0/2			

Table 5 - BI results	After HPGP	Treatment
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# Impact of using Hydrogen Peroxide Gas Plasma (HPGP) on the material compatibility of the TVs

The functionality of selected electronic parts on the PWAs—see photo in Figure 3—was taken before and after HPGP treatment. The continuity measurements shown in Table 6 did not significantly change or result in a value greater than a 10% tolerance or indicate open circuits and failures. These initial results strongly suggest that HPGP is not detrimental to the functionality of the parts by causing electrical opens or shorts.



Figure 3 - PWA TV

<b>Fable 6 - Functiona</b>	lity Results of	<b>PWAs Before and</b>	After HPGP	Treatment
	•/			

TV	Time		Continuity	/ Resistance N	leasurements	(Ohms)	
S/N		Via	Resistor 1	<b>Resistor 2</b>	Resistor 3	<b>Resistor 4</b>	Connector
PWA with Parylene C	Before HPGP	0.3	1,000	100.4	100.1	100	1.3
#1	After HPGP	0.3	1,001	101.2	100.1	100	1.3
PWA with Parylene C	Before HPGP	0.4	1,001	100.1	100.1	99.9	1.2
#2	After HPGP	0.3	1,000	100.4	100.1	99.9	1.2

Parylene C, a common conformal coating used in electronic assembly, is a highly crystalline polymer material composed of carbon, hydrogen, and chlorine atoms. See Figure 4 depicting its molecular structure. Local HPGP penetration through the Parylene C coating on a 20 mil Al wire bond subjected to ten injections or 80 minutes of exposure to the HPGP at 53°C was compared between a control TV in Figure 5 below and a TV in Figure 6 below. The oxide trend through both test vehicles is similar except for the control's local oxide between the potting compound and the coating. The oxide found on the Parylene C coating to the Al wire bond. Local oxide on the Al wire is presumed to have been formed as a native aluminum oxide prior to the application of the conformal coating. This proves that the Parylene C coating in that area did not have any pinholes in this area to allow for HPGP to penetrate through it. It also appears that there is no degradation of the Parylene C or Al wire corrosion between the control and the TV submerged in HPGP.



Figure 4 - Parylene C Molecular Structure



Figure 5 - 20 Mil Aluminum Wire Bond with Parylene C Coating—Control at 5kx/5kev



Figure 6 - 20 Mil Aluminum Wire Bond with Parylene C Coating with HPGP Treatment at 5kX/5keV

# Conclusions

The following results of this feasibility investigation demonstrate that using IPA and HPGP can be a very advantageous, alternative planetary protection (PP) treatment. These results are:

• The average bioburden recovery of each type of TV was a function of handling (resulting in a higher bioburden) or environmental testing (resulting in a lower bioburden).

• IPA aids in the microbial reduction process of mechanically removing spores and killing nonspores on the PWAs, the bare PWBs with immersion silver plating, and silicon die with strain gauge and Au backside metallization.

• Hydrogen peroxide gas plasma (HPGP) successfully killed all spores using two sterilization injections or 16 minutes of vapor exposure at 50°C on all PWAs, bare PWBs with immersion silver plating, and silicon die with strain gauges and Au backside metallization. Therefore, it is feasible to obtain a SAL of  $10^{-6}$  for the packaged electronics under the full cycle conditions (with four injections or 32 minutes of exposure time) in the industrial HPGP Sterilizer.

• The PWAs subjected to 10 sterilization injections or 80 minutes (to include margin) of vapor exposure at 53°C for the material compatibility analysis did not reveal any change in resistance or failures after treatment.

• The SEM analysis revealed no change in oxidation thickness on the Al wire through the Parylene C coating, which was subjected to ten injections or 80 minutes of exposure to the HPGP at 53°C. There also does not appear to be any corrosion of the Al wire or degradation of the polymer conformal coating.

### **Future Work**

New missions may have electronics stored outside of a warm box or exposed to the ambient because of design and/or power restrictions/specifications. For example, rovers with environmentally exposed electronics sent to specifically selected Martian regions or having a perennial heat source may undergo IPA/HPGP planetary protection treatment and thermal cycling from -120°C to 85°C. Experiments investigating the survivability of the electronics through IPA/HPGP and low temperature fatigue conditions are also planned. Actual assembly conditions using IPA/HPGP treatment before and after conformal coating and functional measurements of the electronics will be made. Figure 7 below depicts a hypothetical process flow for future electronic assemblies undergoing IPA/HPGP planetary protection treatment since the work presented in this paper directly supports such a flow. These experiments are planned to ensure statistical confidence using a full factorial design and an adequate TV size. The material compatibility of additional active and passive devices, and PWAs having additional conformal coatings, such as polyurethane, will be subjected to IPA/HPGP and analyzed.

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#### Acronyms and Chemical Symbols

The following acronyms and chemical symbols are used throughout this paper.

Acronyms and chemical symbols	Meaning
Al	aluminum
Au	gold
ASP	Advanced Sterilization Products
BI	biological indicator
СОВ	chip-on-board
CFU	colony forming unit
COSPAR	Committee on Space Research
DHMR	dry heat microbial reduction
DNA	deoxyribonucleic acid
EDS	energy dispersive spectrometer
GPC	gram-positive cocci
GPR	gram-positive rods
HPGP	hydrogen peroxide gas plasma
H <sub>2</sub> O <sub>2</sub>	hydrogen peroxide
IMC	intermetallic compound
I/O	input/output
IPA	isopropyl alcohol
JPL	Jet Propulsion Laboratory
MR	microbial reduction
NASA	National Aeronautics and Space Administration
PP	planetary protection
PWA	printed wiring assembly
PWB	printed wiring board
SCAQMD	South Coast Air Quality Management District
SEM	scanning electron microscope
SMD	surface mount device
SAL	sterility assurance level
Si	silicon
TCRE	Thermal Cycle Resistance Electronics (a JPL project)
TSA	tryptic soy agar
TV	test vehicle
USP	United States Pharmacopeia





Symbols/terms used in Flow Process Chart	Meaning
0-	Indicates a particular operation performed on the workpiece being processed
T	Indicates that the workpiece is moved from one location to another for further processing
TS-	Indicates that the workpiece is temporarily stored until further processing
Ļ	Indicates that the workpiece moves from one process to the following process in the process flow
	Indicates a controlled access area where processing takes place
	Indicates a Class 100 Clean Room facility
w/	Means "with"
w/o	Means "without"
clean/disinfect	Means "treating the hardware with IPA"
sterilize	Means "treating the hardware with HPGP"

Figure 7 - Suggested Flow Process Chart for IPA/HPGP Treatment of Electronic PWAs

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