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HARDWARE CLEANLINESS METHODOLOGY AND CERTIFICATION

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ABSTRACT

Inadequacy of mass loss cleanliness criteria for selection of materials for contamination sensitive uses, and processing of flight hardware for contamination sensitive instruments is discussed. Materials selection for flight hardware is usually based on mass loss (ASTM E-595). However, flight hardware cleanliness (MIL 1246A) is a surface cleanliness assessement. It is possible for materials (e.g. Sil-Pad 2000) to pass ASTM E-595 and fail MIL 1246A class A by orders of magnitude. Conversely, it is possible for small amounts of nonconforming material (Huma-Seal conformal coating) to not present significant cleanliness problems to an optical flight instrument. Effective cleaning (precleaning, precision cleaning, and ultra cleaning) and cleanliness verification are essential for contamination sensitive flight instruments. Polish cleaning of hardware, e.g. vacuum baking for vacuum applications, and storage of clean hardware, e.g. laser optics, is discussed. Silicone materials present special concerns for use in space because of the rapid conversion of the outgassed residues to glass by solar ultraviolet radiation and/or atomic oxygen. Non ozone depleting solvent cleaning and institutional support for cleaning and certification are also discussed.

INTRODUCTION

Hardware cleanliness for space flight applications is usually driven by performance requirements for optics, adhesion of potting and staking compounds and coatings, and electrical continuity or insulation of conformal coatings. The rapid, large temperature fluctuations often experienced by hardware in low earth orbit and resulting from entering and exiting the Earth's shadow, and the acceleration of outgassing in vacuum play a significant role in optical component contamination. The long- term bulk outgassing and chemical reversion (depolymerization) of silicones is a special problem (ref. 1). The polymerization of organic films and the conversion of silicone films to glass by solar ultraviolet radiation and/or atomic oxygen also play significant roles. The vacuum ultraviolet and mid-infrared spectral regions are especially sensitive regions to certain contaminations because of strong molecular absorbtions in these regions. Molecular contamination can also be a concern to the electronic, pharmaceutical, and food industries.

A contamination sensitive flight instrument such as the Halogen Occulation Experiment (HALOE) instrument (ref. 2) should include the following six major cleanliness activities: 1. Selection of Materials; 2. Cleaning of parts, i.e. precleaning, precision cleaning, and ultracleaning; 3. Rough vacuum bake of parts or systems; 4. High-vacuum bake or functional test of instrument; 5. Purge or vacuum storage of instrument or contamination sensitive parts; and 6. Cleanliness verification or certification by wipes, washes, witness plates, mass spectroscopy, etc..

However, in practice, cleanliness of many instruments at Langley Research Center (LaRC) is based primarily on 1. selection of "low outgassing materials" from RP-1124 (ref. 3) and integration and testing of the instruments in clean rooms. The clean rooms do an excellent job of minimizing particle contamination, but generally do not address molecular contamination. The main pitfalls from this expediency are that some materials such as silicones and urethanes that are approved, or are suitable for very limited use in non-critical areas are used in significant amounts and in critical areas, and that handling, environments, and aging effects are overlooked. For example, clean rooms designed to greatly reduce particle contamination often use heavy outgassing materials (such as vinyl) in downflow curtains. Another common practice is to use a solvent such as alcohol and new cleanroom wipes and gloves to wipe particles and stains from work surfaces and instrumentation. The solvent extracts plasticizers (e.g. akyl phthalates and other esters) from the gloves and wipes and deposits this material as a thin film on the "cleaned" surface. Although nitrile and polyethylene gloves are relatively low in molecular residue, Teflon is the only really clean glove material for solvent cleaning. Unfortunately, Teflon gloves are expensive, and tear easily. Fortunately, only a small amount (~5%) of clean room work is solvent cleaning. However, much current precision cleaning is with trichlorotrifluoroethane (Freon 113), and gloves and plastic wear can easily contaminate this cleaning solvent.

The hardware that is precision cleaned at LaRC varies greatly in size, shape,and use. A general guideline used at LaRC is that if hardware is visibly dirty, or is suspected to have more than 10 mg of molecular contamination, it is precleaned with soap and water, or wiped with alcohol before being run through the ultrasonic solvent parts cleaner. The ultrasonic parts cleaner does a good job of removing loose particles and cleans the parts to about 0.5 mg per square foot of molecular films. A rough vacuum bake (with an LN_2 trap) will generally reduce this by a factor of two by offgassing the lighter molecular weight (MW<300) constituents. This rule-of-thumb was obtained from several before-and-after vacuum bake surface-cleanliness-wipes of hardware.

MASS LOSS AND SURFACE CLEANLINESS

Materials selection for flight hardware is often based on mass loss (ASTM E-595, ref. 3). This selection process is generally a quick and convenient procedure to address hardware cleanliness. It has been effective for a large number of applications and has become standard practice. It does not address the identification of the outgassing products and is sometimes used to imply surface cleanliness, which is a Shuttle requirement for space flight. That is , there are two types of cleanliness: 1. material outgassing, and 2. surface cleanliness. Bulk material outgassing is indicative of potential to contaminate nearby hardware with molecular films. Surface cleanliness (the molecular films) affects adhesion, electrical and optical properties. Currently, many cleanliness evaluations are based on outgassing (ASTM E-595) alone. This is an expedient practice, but allows hardware with slow outgassing material, or that has acquired significant surface films, or that has deteriorated, to be used in contamination sensitive applications.

Heavy silicone and akyl phthalate fluids (synthetic oils) outgas relatively slowly, but significantly, during long exposure to vacuum. Hardware processed in facilities or equipment with silicone or plastic material will accumulate films of silicones and synthetic oils until each type of outgassing product reaches equilibrium between adsorbtion and evaporation. The equilibrium time is days or weeks for light and volatile compounds such as alcohols and ethers (MW<200) and years for heavy silicones and phthalates (MW>300). The equilibrium time is much quicker if the hardware is in a small, confined space (e.g., an optic stored in a plastic shipping box). Some materials deteriorate with age and some silicones undergo chemical reversion (depolymerization) when exposed to chemicals such as ammonia. These processes can lead to great disparity between cleanliness inferred from ASTM E- 595, and measured surface cleanliness (MIL 1246A).

Surface cleanliness can be measured by washing or wiping the surface with suitable solvents and by electron microscope techniques. Table 1 lists several examples of hardware measured at LaRC which demonstrate this disparity. The Sil-Pad-2000 is a silicone heat transfer pad used with the Measurement of Air Polution from Satellites (MAPS) instrument, and the high voltage cable is used in a high energy laser instrument (Laser Atmospheric Sensing Experiment (LASE)). The expediency of treating cleanliness primarily by reliance on ASTM E-595) is difficult to question when non-cleanliness-related performance and schedule requirements drive most projects.

The environmental testing and development group at LaRC has initiated customized cleaning of hardware at little or no monetary or schedule costs to projects when hardware such as that listed in Table 1 is brought into the test facilities. That is , the facility personnel assume ownership of hardware as well as facility cleanliness concerns. The greater continuity in cleanliness resources and hands-on expertise in the facilities compared to the projects is part of the rationale for this practice.

VACUUM OUTGASSING

Low vacuum ($P \sim 10^{-1}$ Torr) can be used to polish-clean and measure cleanliness of hardware for vacuum applications. The outgassing products from the hardware can be collected on a cold surface, i.e. a scavenger plate, inside the chamber during the test, and then removed, weighed, and analyzed after the test. Infrared analyses of the thin films of the outgassed residue can conveniently be performed to determine chemical composition. Table 2 and figure 1 list common outgassing products collected during vacuum cleaning and testing at LaRC. Generally the hardware is heated to accelerate the outgassing or to simulate the space environment. Dynamic pumping and LN_2 foreline traps are used to maintain chamber cleanliness under vacuum.

Mass spectrometry can be used to measure cleanliness of hardware in high vavuum ($P \sim 10^{-6}$ Torr). A mass spectrometer gives real-time outgassing history as well as chemical identification of the outgassing products. Table 3 and figure 2 list typical outgassing species and show their mass spectra. Infrared analyses of residues collected on a scavenger plate in the vacuum chamber provide complimentary chemical analyses. A polish cleaning of hardware or instruments intended for space or vacuum application can be performed. This cleaning process is designed to benignly remove molecular contamination which could migrate to contamination sensitive surfaces in vacuum and is amenable to integrated hardware and systems. A 24 hour bake at 125° C is a typical vacuum bake process. This corresponds to roughly one year under vacuum at room temperature. Clean hardware should be maintained under vacuum, or protected from recontamination by sealing in appropriate bagging material, preferably with an ultrapure gas purge such as LN_2 boiloff.

Vacuum chamber cleanliness measurement can be used to determine the contamination potential of non-approved material in hardware. Huma-Seal conformal coating (not an approved material) was applied to electronic boxes for a high energy laser instrument. Extracted cleanroom wipes were placed at the vents of the cover of these boxes and collected about 0.3 mg of akyl phthalates during a high vacuum test. The small amount of akyl phthalates on these wipes, on witness plates, and on external instrument surfaces, showed that the Huma-Seal did not pose a risk of contamination to the laser instrument or to nearby hardware.

NON-CFC SOLVENT CLEANING

The standard practice in the aerospace industry is to precision clean parts in an ultrasonic cleaner using Freon 113 as the cleaning solvent. This solvent is a non-polar chloroflurocarbon solvent which readily dissolves many organic fluids such as hydrocarbons and esters, and silicones. However, two of its most redeeming properties for cleaning purposes are that it is nonflammable and that it evaporates quickly (expediting quick cleaning). Freon 113 is one of several chemicals listed as ozone depleting substances by the Montreal Protocol and scheduled for restricted use or phaseout in the present decade.

Cleaning efficiencies of several aqueous systems were measured and compared to the present in-house cleaning. New polyester cleanroom wipes were used as the test coupons. The results are listed in Table 3. A negative cleaning efficiency means that more material is added to the test wipe than is removed by the cleaning process. The residue on the test wipes was measured by the standard practice of soaking a 225 cm^2 test wipe in 40 ml of analytical grade isopropyl alcohol for 30 minutes, and weighing the residue after evaporation of the alcohol. The infrared spectra show significant differences in the composition of the residue left on the wipes after cleaning. Therefore, the cleaning efficiences of column 3 in Table 4 represent a loss of some types of residue and a gain of other types of residue. A negative efficiency means that more residue is added than is removed by the cleaning process.

Four of the six aqueous cleaning procedures tested added more residue than they removed. This is probably because these aqueous cleaners are added in much larger amounts than the original residues to be cleaned, so it requires much higher dilutions in the rinse stage to remove almost all of the added cleaner.

Water is strongly polar and not a good solvent for most of the nonpolar residues, e.g. akyl phthalates, on LaRC flight hardware to be precision cleaned. Freon 113 is a good nonpolar solvent and leaves little residue because the Freon 113 has low suface tension and drains off. Small amounts of Freon will be absorbed, and will wick along wires. This Freon is readily detected by a mass spectrometer if hardware cleaned in a Freon cleaner is placed in a high vaccum chamber within a few weeks of being cleaned (Fig. 2). This is seldom a problem since Freon is relatively inert and will eventually offgas from the hardware. These properties: nonpolarity, low surface tension, and rapid evaporation, make Freon 113 a good solvent for nonpolar residues. The low boiling temperature and small heat capacity of Freon 113 make it easy to purify or reclaim by distillation.

Since water is not a good solvent for most organic residues, a detergent and/or surfactant is needed to remove and suspend organic residue. These cleaning additives are used in many times the amount of residue to be cleaned from the hardware in order to be effective, and must be removed by a subsequent cleaning process (generally deionized water rinses). A lot of ultrapure water is needed to remove almost all of the surfactants and detergents. It was concluded from the data in Table 1 that aqueous cleaning of LaRC flight hardware could not meet the existing cleanliness requirements with the available equipment and facility resources.

Several nonpolar solvents such as hexane, benzene, toluene, acetone, and light alcohols are as good organic solvents as Freon 113 and also have low surface tension and evaporate quickly. However these solvents are flammable and some pose health concerns. LaRC has experience in ultracleaning using Soxhlet extractors and isopropyl alcohol (IPA) as the working solvent (ref. 4). Much higher cleaning efficiencies (Table 4) are attainable with a Soxhlet extractor and IPA than with Freon in an ultrasonic cleaner because the working temperature of the IPA is higher (180^0 F) in the Soxhlet extractor, and generally the wash time is much longer. The Soxhlet extractor uses a closed loop repetitive distillation and flush process and is generally constructed of glass. The high cleaning efficiencies obtained with Soxhlet extractors led to modification of an existing ultrasonic cleaner in order to use IPA as the working solvent. The modifications included use of covered tanks (telescoping lids) for the ultrasonification-wash tank, use of ultrapure deionized water for rinsing, and use of an organic vapor detector in the cleaning station.

The rinsing operation is often necessary because the wash solvent usually becomes contaminated soon after hardware is placed in it, and this will result in localized deposits of residue on the hardware when the alcohol evaporates. The rinse operation washes the slightly contaminated IPA off the hardware. The use of ultrapure water as a rinse fluid usually requires blow drying because of the low levels of colloidal silica in most ultrapure water, which is converted to silica during drying.

CERTIFICATION

The cleanliness requirement for hardware and instruments to be flown in the orbiter payload bay is <10 mg/m^2 of molecular residue. The certification of this requirement is usually in the form of gravimetric analysis of residue washed from an accessible surface of the hardware. This measurement is usually included in a test report or as part of a quality assurance report. These measurements are sometimes performed for historical purposes only, and have long processing and distribution times.

The LaRC cleanliness measurements laboratory has adopted the practice of writing and distributing cleanliness test reports for every major vacuum test of flight hardware and for periodic cleanliness measurements of the hardware. These reports are distributed within five working days of the test, or return of the witness specimens to the lab. A handwritten report is usually given to the test conductor within 24 hours, and sometimes during the same shift, so that the results can be used in project evaluations and decision making. Chemical analyses of the residues, by infrared spectroscopy and, or mass spectroscopy are usually included in the reports. These reports are used by project personnel, facility personnel and by quality assurance personnel for functional assessments, facility assessments, and compliance with regulations.

Cleanliness activities within NASA have been institutionally supported by projects which either assigned a design engineer or test engineer from an engineering organization the responsibilities of contamination control. These contamination control engineers are sometimes left on their own to find or develop laboratory support for cleanliness measurements. Their access to hardware and responsibility usually decreases as the project hardware is integrated onto a spacecraft, or moves to a non-project owned facility. The support for tracking of cleanliness effects and for formal reporting usually follows a similar decline and has resulted in a small data base for hardware flown on the the Shuttle and exposed to the induced space environment. There is a current need for more dedicated laboratory support for hardware cleanliness measurements. These hardware and instrument cleanliness measurements are a legitimate area of responsibility of Quality Assurance.

CONCLUSIONS

There is a general overreliance on mass loss criteria (ASTM E-595) for hardware and instrument cleanliness. This is partly a result of a general lack of inhouse cleanliness measurement capability. More recognition is needed for continuity of cleanliness assessments through mission completion, for measurement and analyses capability and for reporting. There are many measurement techniques that can be used to measure cleanliness at very low or no cost.

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TABLE 1

CLEANING OF MATERIALS

Material	As received	After cleaning
Sil pad 2000	125 mg/ft^2	6 mg/ft^2
Silicone gasket	10 mg/ft^2	l mg/ft^2
HV cable	400 mg/ft	l mg/ft^2
SEDS cable	52 mg/ft	ND
RG58 cable	.1 mg/ft	ND

TABLE 2

IR ABSORBTIONS OF COMMON CONTAMINANTS

Class	Absorbtions, cm^{-1}
Phthalates mw ~ 400	1730 (C= 0), 1600 and 1575 (C_6H_4)
Urethanes	1640 (NH_2), 1615 (NH), 1540 (NH)
Silicones mw ~ 250 DC704 = 484	1265 ($SiCH_3$), 1025-1100 (SiO), 1120 (Siϕ)
Hydrocarbons C_{18} mw ~ 250	1460 (CH_2, CH_3), 1375 (CH_3)

TABLE 3

MASS FRAGMENTS OF COMMON CONTAMINANTS

Class	Mass, amu
Phthalates	149, 167
Freons	$\Delta M = 2$; $Cl_{35}/Cl_{37} = 3:1$
Silicones	73[$Si(CH_3)_3$], 147 [$Si_2O(CH_3)_5$]
Hydrocarbons	$\Delta M = 14[CH_2]$ picket fence

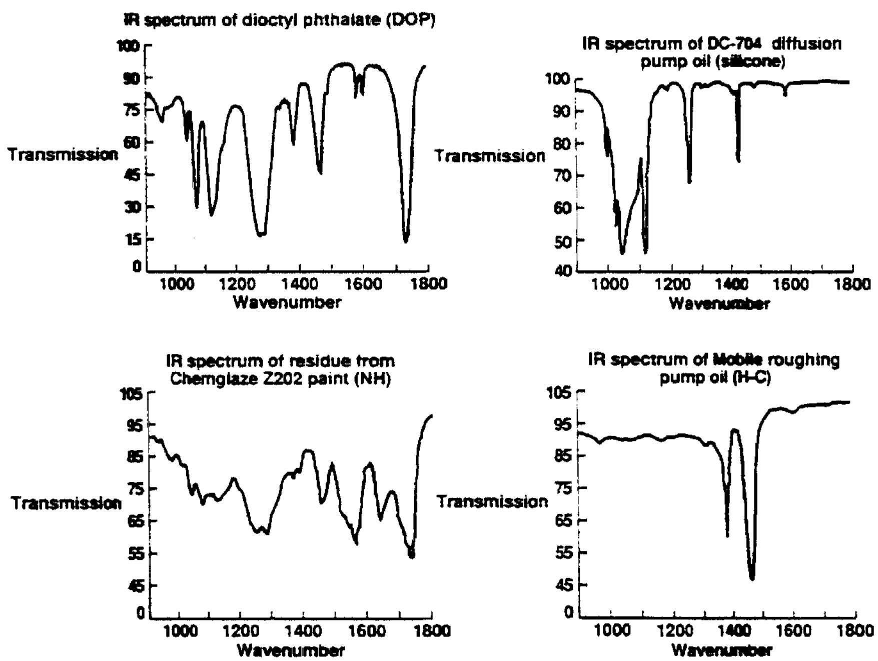


Figure 1.- IR spectra of common contaminants.

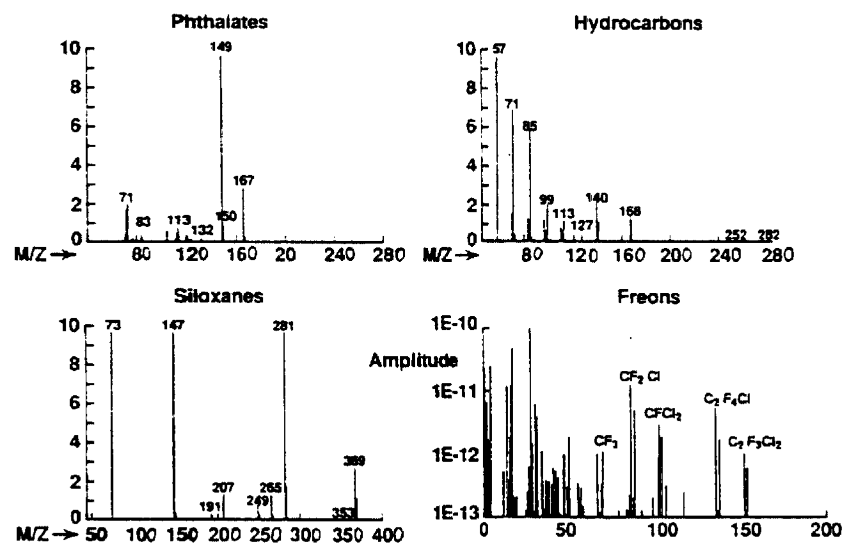
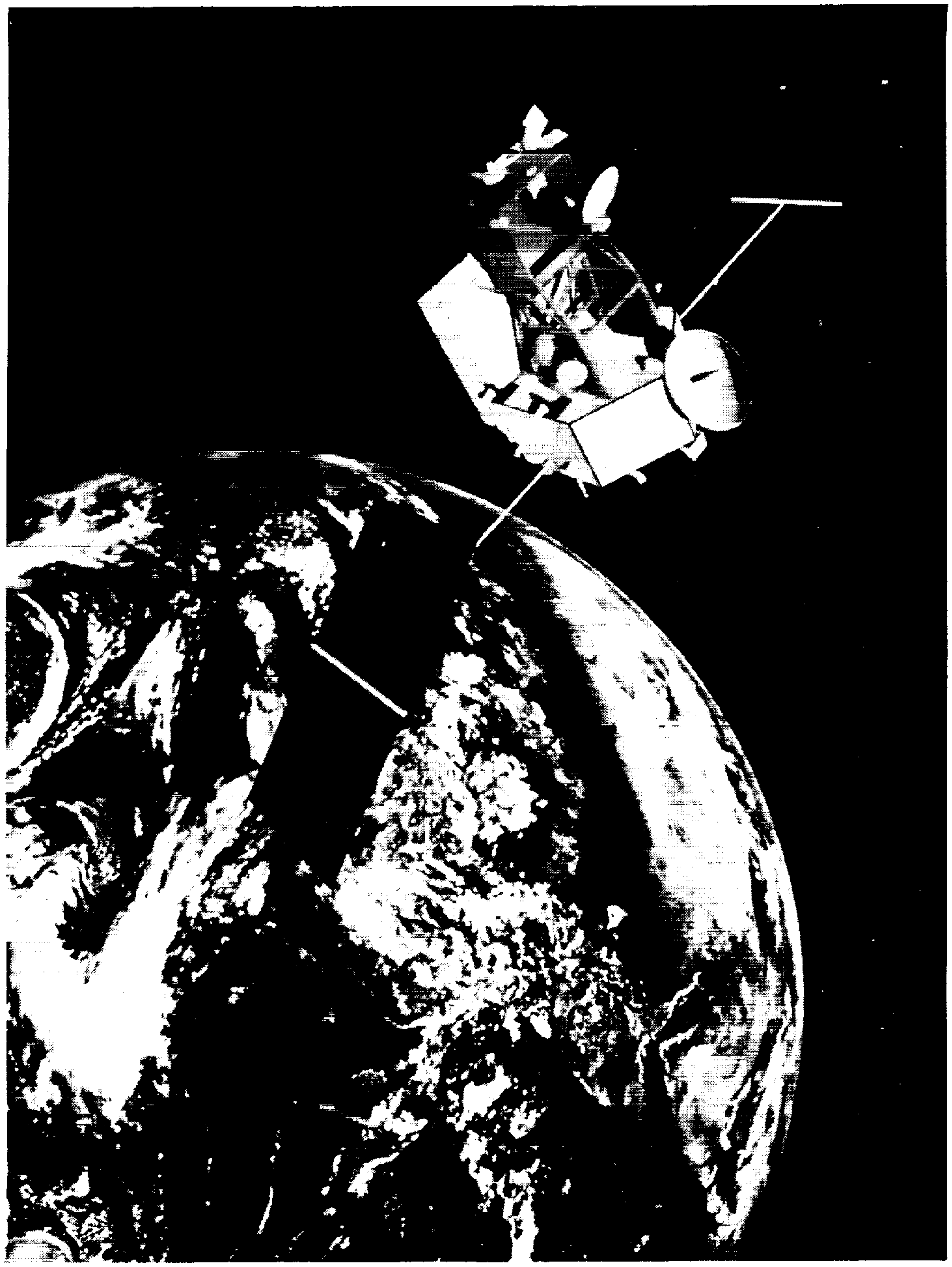


Figure 2.- Mass spectra of common contaminants.

TABLE 4.- CLEANING EFFICIENCIES

Cleaning	Recovered NVR (mg/ft^2)	NVR Cleaning Efficiency (%)
New Wipe-Coventry	2.22	N/A
DI H_2O	2.20	0.
Bronson Surfactant	2.44	-10
Trisodium Phosphate	2.59	-17
Hurri Clean	2.27	0
Triton X-100	3.02	-36
Alconox	.96	56
Zone Defense	8.12	-370
Freon 113	.49	78
IPA 5%	2.27	0
IPA 50%	2.48	-10
IPA 95%	.48, .75, 1.30	78, 66, 40
IPA 100%	.38, .37, .50	85, 83, 77
50% IPA/50% F113	1.05	50
Soxhlet IPA 2X	.04, .09, .12	98, 96, 95

FUTURE ACTIVITIES



Tropical Rainfall Measuring Mission (TRMM), artist's concept