PROPELLANT ANALYSIS AND DISTILLATION UNIT DESIGN

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ABSTRACT

The NASA White Sands Test Facility (WSTF) routinely operates hypergolic propulsion systems. Some of the onsite activities include performing long duration studies on the operational life of these systems. A few of them have been in use for over twenty years. During this span of time contamination has built up in the propellant and some of the distribution infrastructure. This study investigated the nature of this contamination, the pathology of its generation, and developed a process for removal of the contamination that was cost efficient with minimal waste generation.

1. INTRODUCTION

Since the early 1960s, NASA WSTF has served as the site for the Johnson Space Center (JSC) Propulsion Systems Development Facility for which hypergolic test systems have supported testing and research.

During this span of time, contamination has built up in the propellant and some of the distribution infrastructure.

This contamination has led to increased propellant procurement, additional time needed to prepare test systems and additional test costs to run repetitive propellant analysis.

A survey was performed on all hypergolic propellants at WSTF to determine which were contaminated and what quantities of contamination were present. Monomethylhydrazine (MMH) was the material determined to have the greatest quantity of contamination. A nonvolatile residue (NVR) level greater than 10 mg/L is unacceptable for use in hypergolic systems per SE-S-0073, *NASA Specification Fluid Procurement and Use Control*.[1] Quantities of MMH were analyzed and found to have NVR levels as high as 30 mg/L.

In this survey, it was found WSTF currently has more than 3,000 gallons (Fig. 1) of MMH with an NVR concentration in excess of the specification limit. The MMH has been collected in tanks for several years for future disposition. Rather than dispose of unusable MMH, the feasibility of decontaminating the propellant was proposed as a cost-effective and environmentally friendly alternative.

Figure 1. 300 Area Fuel Dump Tank

2. NON-VOLATILE RESIDUE (NVR)

NVR in MMH based fuels is composed of organic derivatives of MMH. Analysis by Fourier Transform Infrared Spectroscopy (FTIR), shown in Fig. 2, indicates that fuel NVR is an organic compound with nitrogen-hydrogen and carbon-hydrogen bonds.

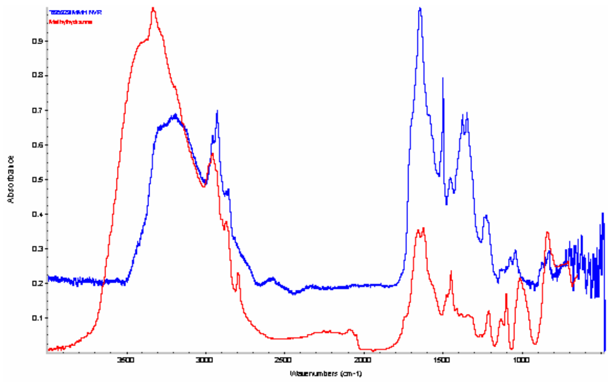

Figure 2. Monomethylhydrazine NVR FTIR

WSTF scientists and technicians hypothesize that fuel contaminants result from interaction between hydrazine based fuels and atmospheric components during handling and transferring of the propellants. Long term stored propellants in undisturbed containers do not show the decomposition that is shown onsite. [2]

In an experiment performed by the WSTF Chemistry Laboratory,[3] the artificial aging of MMH was conducted by injecting air and carbon dioxide into a test container filled with MMH, causing what may be NVR precursors. When comparing NVR contamination, MMH forms NVR more readily than oxidizer, causing more MMH to be rejected for use per NASA specifications.

Table 1 is a list of possible precursors to impurities found in NVR during this investigation in aged and non-aged MMH.

Table 1: MMH NVR Precursors

Name	Formula
Nitrogen	N_2
Dimethyldiazine	$CH_3N{=}NCH_3$
Ethylene oxide	$C_2 H_4O$
Ammonia	NH_3
Methylamine	CH_3NH_2
uns-Dimethylhydrazine	$(CH_3)_2NNH_2$
sym-Dimethylhydrazine	$CH_3NHNHCH_3$
Formaldehyde methylhydrazone	$CH_3HNN{=}CH_2$
Methoxyethane	$CH_3OCH_2CH_3$

These potential precursors of NVR can be readily synthesized with common reactions in the conditions found in the experiment.

3. METHODS OF DECONTAMINATION

To ensure the development of an appropriate and safe process, the chemical characteristics of the propellant were considered during a separation technique assessment[2]. MMH is corrosive and toxic; it is also highly flammable, toxic by contact or inhalation, and causes severe burns. This propellant requires personal protection equipment and limited human exposure. In addition, there are limited materials that can be exposed to MMH.

Research on separation techniques was conducted to find the best method for contamination removal based on information known about NVR and propellant. The theories of filtration, adsorption, and distillation were evaluated for the feasibility of removing NVR from propellant. The separation techniques were chosen because they were simplistic and would work with all types of propellants.

Filtration uses a membrane to prevent the flow of certain sized molecules through the pores of the filter. The ability of filtration to remove NVR is limited by the particle size of the substances because the filter pore sizes range from two to two thousand nanometers, only trapping larger particles of NVR. Monomethylhydrazine measures smaller than 500 pm (0.0005 micrometers) and derivatives of MMH form the contamination; therefore most contamination can pass through the filters. Filtration allows for the continuous flow of liquid, making it time efficient but the size limits and the decreasing efficiency cause filtration to be ineffective. As the filtrate is flowed through the filters, the pores become blocked and efficiency decreases and the filters must be regenerated or replaced.

Adsorption is a selective interaction between the contaminant and adsorbent. The contaminant accumulates on the surface of the solid or liquid adsorbent and is removed from the propellant. Experiments have been conducted at WSTF with nitrogen tetroxide and various adsorbents. It resulted in a time consuming process that had column compression and channeling. Problems with adsorption for this project would be selecting an absorbent for fuel that was only selective to the various MMH derivatives and not MMH. The absorbent would also vary for each propellant, require replacement, and decrease in efficiency with use.

Distillation is based on the volatilities of substances being separated and consists of the addition and removal of heat. Any compound that can evaporate can be separated from nonvolatile substances using this technique and extra waste is not generated. The process materials are non-selective and can be used with any propellant as long as the boiling point range and autoignition temperature is known. The still can also be constructed with compatible materials, easily stored, cleaned, and operated. In addition, distillation is used at WSTF to determine the concentration of NVR in a one hundred milliliter sample by evaporating the propellant from the sample and collecting only the contamination. Other measures can be made with distillation to increase safety and efficiency.

Evaluation of these separation techniques resulted in the selection of distillation as the separation technique. Distilling the MMH will be done by mechanical methods (heat input and removal) and will not need the use of catalysts (chemical and/or biological). Waste generation will be minimal with less than 1 pound of waste per 3000 gallons of MMH.

This paper recognizes some impurities found in NVR will be distilled out with the MMH, due to their boiling points being lower than that of MMH. Some of these materials still may be reduced in concentration by using a condenser temperature that wouldn't condense them. Table 2 shows the boiling points for the possible NVR precursors.

Table 2: MMH NVR Precursors Boiling Points

Name	Formula	Boiling Points (°F)
MMH	NH_3CH_3	191
Nitrogen	N_2	-321
Dimethyldiazine	$CH_3N{=}NCH_3$	NA
Ethylene oxide	$C_2 H_4O$	52
Ammonia	NH_3	-28
Methylamine	CH_3NH_2	21
uns-Dimethylhydrazine	$(CH_3)_2NNH_2$	145
sym-Dimethylhydrazine	$CH_3NHNHCH_3$	NA
Formaldehyde methylhydrazone	$CH_3HNN{=}CH_2$	NA
Methoxyethane	$CH_3OCH_2CH_3$	46

4. NVR BENCH-TOP ANALYSIS

To determine the purity of propellants, WSTF performs monthly single pass NVR chemical purity analyses. The method described below is a double pass NVR chemical purity analysis[4].

Propellant sampling locations were assessed based on the level of contamination and volume of the container. Two propellant storage tanks were chosen for sampling, and two samples were taken from each tank. Samples were 1 L each, and were processed individually through the apparatus. The pilot plant was able to reduce contamination levels of 12 to 25 mg/L to a level of 0.1 to 0.2 mg/L.

Based on the same principles for text-book distillation, the WSTF Chemistry Laboratory performed a 'bench-top analysis' in which a table-top distillation, shown in Fig. 3, was performed under vacuum of the initial sample and the distillate was collected in a clean flask. The distillate was then distilled a second time. The NVR value is the difference between the gross and tare weight, normalized for the amount of non-volatile residue present in one liter of the fluid.

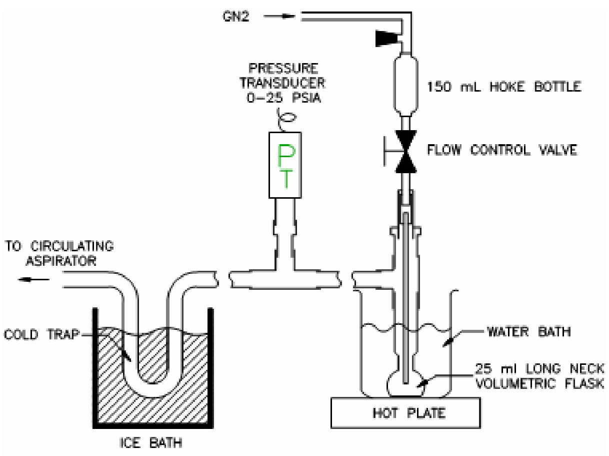

Figure 3. Table Top Analysis Assembly

Table 3 shows the results from the bench-top analysis performed on samples from the two sampling locations; WSTF Propulsion Test Area, the 300 Area Fuel Dump Tank, and the 400 Area Ready Storage Unit (RSU).

Table 3. Bench-Top Analysis Results

Source	Initial NVR	Final NVR
300 Fuel Dump Tank	13.4	5
	12.8	0.6
400 Fuel RSU	25.2	0.1
	14.3	0.2

This analysis validated distillation as an adequate method to reduce the NVR to acceptable levels (and below) per NASA SE-S-0073[1].

5. DISTILLATION UNIT PROCESSING REQUIREMENTS

It is the intent of this project to process contaminated MMH through a distillation unit where the distillate will be collected and stored for future use.

To process the 3,000+ gallons of MMH currently at WSTF a large-scale version of the bench-top assembly is required that is constructed from MMH compatible materials, easily stored, cleaned, and operated.

The unit will be fabricated per the latest revision of the American Society of Mechanical Engineers (ASME) B31.3 *Process Piping*, "Normal Fluid Service,"[5] to take advantage of industry experience and provide an appropriately robust processing unit. The unit will contain measures to increase safety, efficiency and produce minimal waste.

6. DISTILLATION UNIT FABRICATION REQUIREMENTS

A set of requirements were developed for the procurement of a distillation unit and are included in a statement of work to process contaminated MMH into re-usable MMH below NASA Shuttle specification (SE-S-0073)[1] standards.

Introducing heat to MMH needs to be under very specific and well controlled parameters. Below are some general requirements included in the statement of work for the purchase of an MMH distillation unit.

6.1. MMH Fire, Explosion, and Safety Hazards

Since MMH is a strong reducing agent, very hygroscopic, reacts readily with a variety of organic and inorganic compounds and is thermodynamically unstable, WSTF will require the vendor to make assessments in which fire hazards, deflagration, exothermic reactions, and detonation are considered during the design process.

All potential ignition sources located within the NFPA 497[6] designated hazardous area(s) must be rated for Class I Division 1 Group C.

6.2. Material Compatibility

MMH can react with various materials; therefore it is imperative that all components in contact with it are compatible. The following materials have been proven compatible with MMH and are allowable for constant contact at 120 °F or less per WSTF standard instructions.

Use of other materials not listed in the WSTF standard instruction at elevated temperatures must be reviewed and approved by the WSTF Hypergol Materials Approval Committee (HMAC). These materials include:

6.3. MMH Handling

MMH is highly reactive with oxygen in air, oxidizing agents, organic matter, and oxides of metals such as iron, copper, lead, manganese, and molybdenum. Because MMH is so reactive, storage conditions must be chosen carefully.

Although it is not susceptible to decomposition through normal impact or friction (hydrodynamic shear), MMH reacts with oxygen and carbon dioxide in the air; therefore, the unit shall be designed so that MMH is stored and operated under a nitrogen blanket.

Guidelines for designing safe storage and processing for hydrazine fuels include:

a. Use a minimum of mechanical fittings (welded systems are preferred)
b. Consider maximum system pressure
c. Avoid low-lying liquid traps
d. Provide for inert gas purges
e. Electrically ground the holding container and protect it from electrical sparks, open flames and heat sources

6.4. Energy Efficient Products

In compliance with Executive Order 13123,[7] the distillation unit will incorporate energy and water conservation efficient components and operating methodology. The design will incorporate Energy Star®–qualified and Federal Energy Management Program (FEMP)-designated products.

6.5. Explosion Proofing

All electrical and electronic equipment that are part of an electrical installation will be enclosed in explosion-proof apparatuses per Class 1, Division 1, Article 500.5 of the National Electric Code (NEC)[8]. All wiring will be explosion-proof per Article 501 NEC 2005. An explosion-proof apparatus is defined as an ‘apparatus enclosed in a case that is capable of withstanding an explosion of a specified gas or vapor that may occur within it and of preventing the ignition of a specified gas or vapor surrounding the enclosure by sparks, flashes, or explosion of the gas or vapor within and that operates at such an external temperature that a surrounding flammable atmosphere will not be ignited thereby.”

6.6. Alarming

The unit will come equipped with a system of alarms that will inform personnel when process conditions are exceeded above or below the required input.

This includes:

- Leak detection
- Safety Interlocks
- Pressure monitoring and relief systems
- Temperature monitoring (including cut off switches)
- Remote Shutdown
- Emergency Shutdown

All pertinent alarms will be wired to the control room console to inform the blockhouse monitor in the event a parameter is out of tolerance.

6.7. Cleanliness

All unit components which will be in contact with the process fluid must be cleaned per the latest revision of JPG 5322.1, Contamination Control Requirements Manual to level 200[9].

6.8. Failure Modes and Effects Analysis (FMEA)

An FMEA will be performed at the vendor location during the design process. An FMEA is a formal analysis identifying all single component failure modes, the effects of those failures on the operation and safety of the system, and a recommendation for resolution of the failure by design change (if possible), or procedural control. Each component of the unit shall be analyzed in this manner.

7. CONCLUSION

Reclaiming contaminated MMH currently at WSTF for re-use was an attractive alternative to disposal for two main reasons; cost savings and environmental protection.

Three feasible methods of NVR removal were considered: filtration, adsorption, and distillation from which distillation was determined to be the simplest and most efficient method to remove NVR from MMH.

A statement of work detailing requirements for the purchase of a distillation unit was written to meet ASME B31.3[5] code requirements and all WSTF safety regulations, of which some are included here.
This distillation unit will be fabricated so as to meet all code and safety requirements while removing high concentrations of NVR from MMH for re-use.

8. FUTURE WORK

This study developed the foundation for designing propellant systems that minimize the potential for developing NVR. It will be incorporated into future WSTF designs.

Additionally, with successful validation of this unit, it is anticipated that other NASA Agency locations with propellant contamination issues can develop remediation processes of a similar nature.

9. REFERENCES

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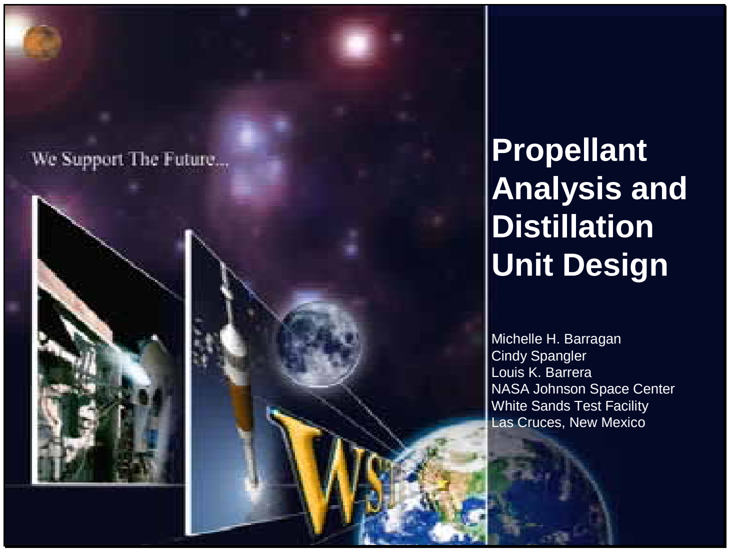

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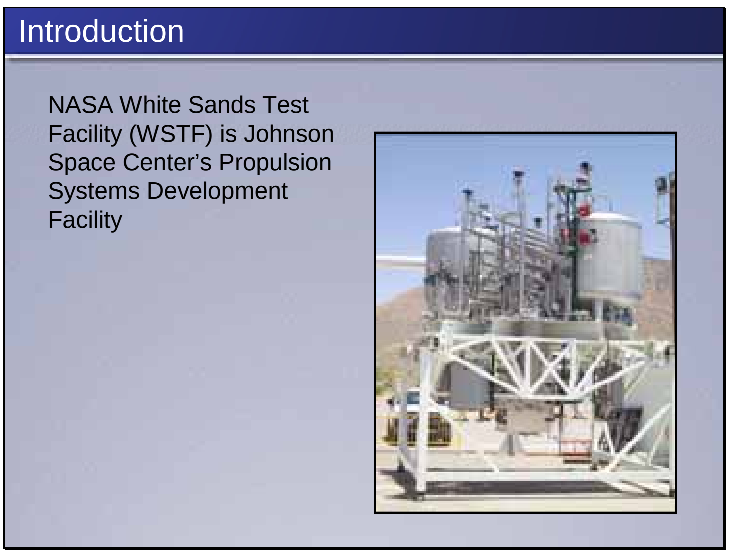

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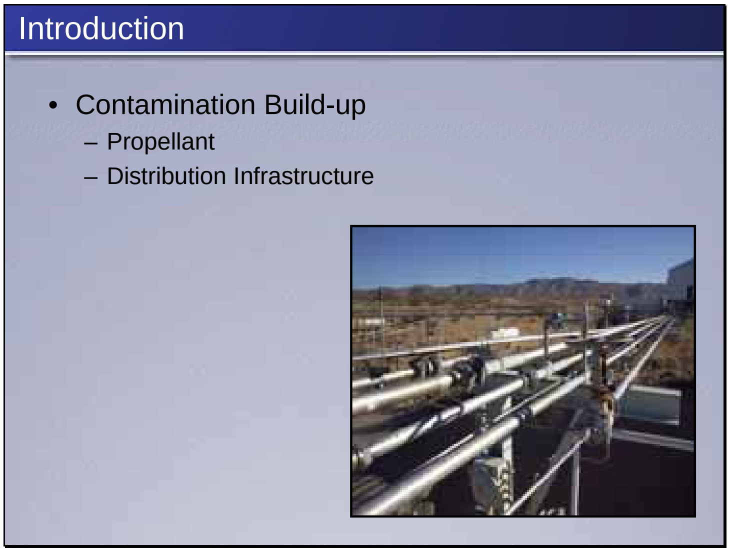

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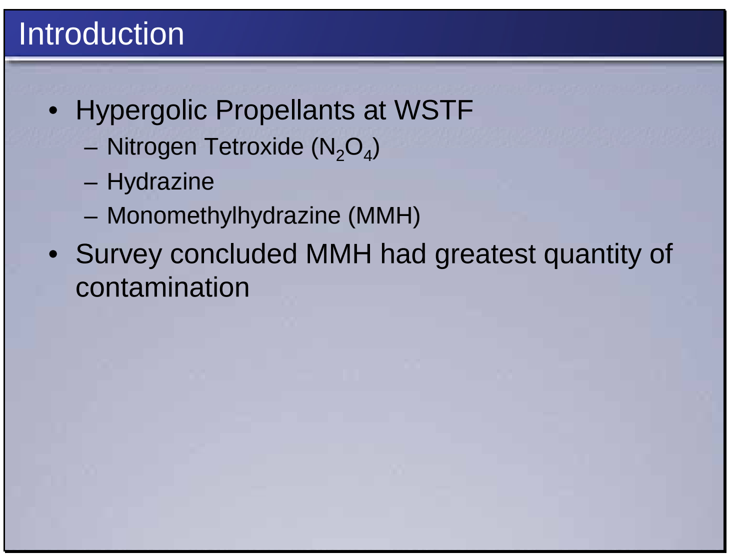

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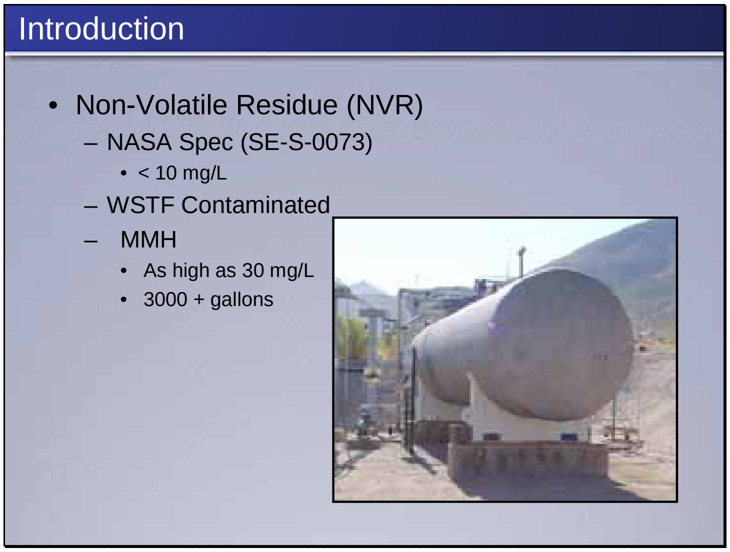

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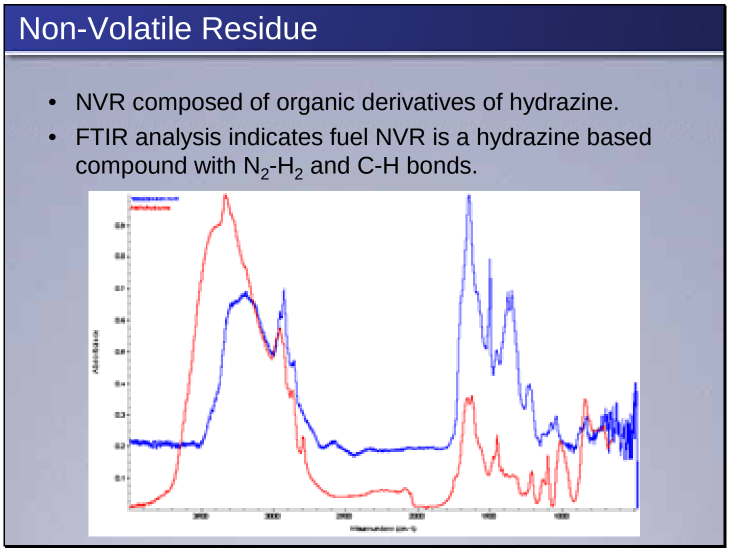

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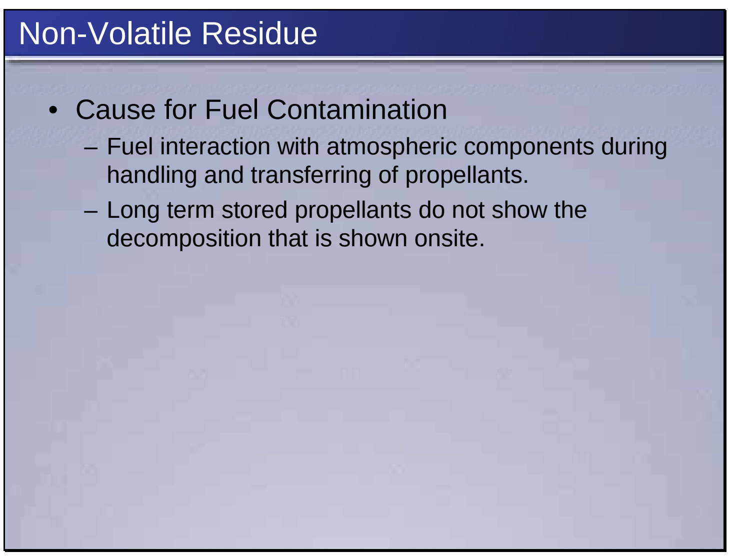

WSTF scientists and technicians hypothesize that fuel contaminants result from interaction between hydrazine based fuels and atmospheric components during handling and transferring of the propellants. Long term stored propellants do not show the decomposition that is shown onsite

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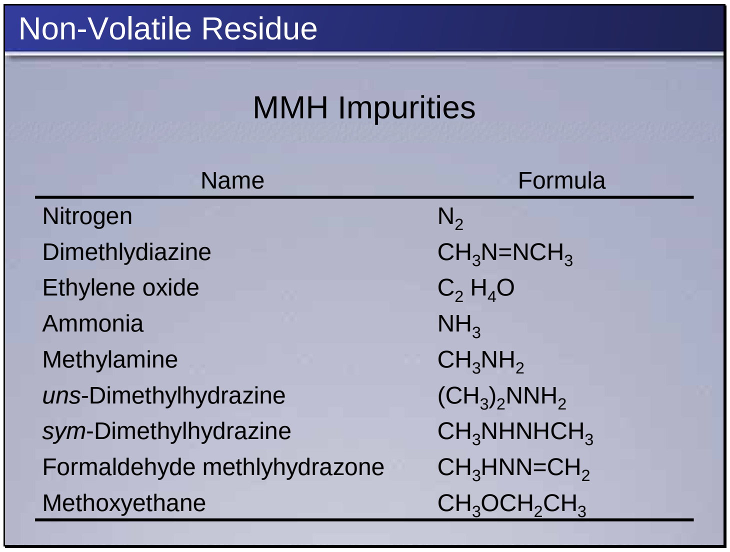

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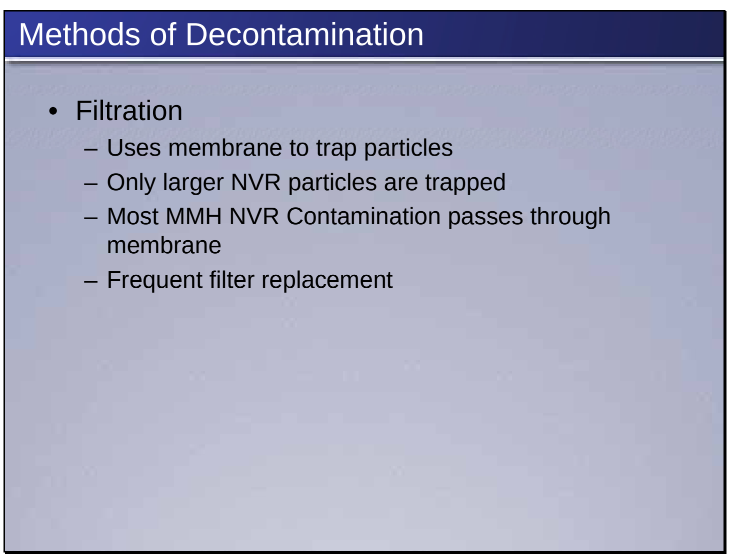

Filtration uses a membrane to prevent the flow of certain sized molecules through the pores of the filter. The ability of filtration to remove NVR is limited by the particle size of the substances because the filter pore sizes range from two to two thousand nanometers, only trapping larger particles of NVR. Monomethylhydrazine measures smaller than 500 pm (0.0005 micrometers) and derivatives of MMH form the contamination; therefore most contamination can pass through the filters. Filtration allows for the continuous flow of liquid, making it time efficient but the size limits and the decreasing efficiency cause filtration to be ineffective. As the filtrate is flowed through the filters, the pores become blocked and efficiency decreases and the filters must be regenerated or replaced.

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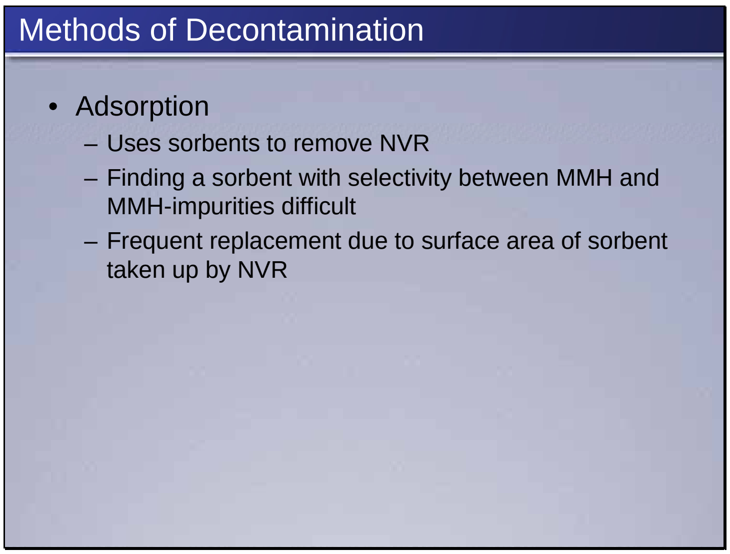

Adsorption is a selective interaction between the contaminant and adsorbent. The contaminant accumulates on the surface of the solid or liquid adsorbent and is removed from the propellant. Experiments have been conducted at WSTF with nitrogen tetroxide and various adsorbents. It resulted in a time consuming process that had column compression and channeling. Problems with adsorption for this project would be selecting an absorbent for fuel that was only selective to the various MMH derivatives and not MMH. The absorbent would also vary for each propellant, require replacement, and decrease in efficiency with use.

Solute from the fluid selectively attaches itself to the solid. The solid being the sorbent.

At ordinary temperatures, adsorption is usually caused by intermolecular forces rather than by formation of new chemical bonds. At higher temperatures above 400 °F, the activation energy is available to make or break chemical bonds, and if such a mechanism prevails, the adsorption is called chemisorption or activated adsorption.

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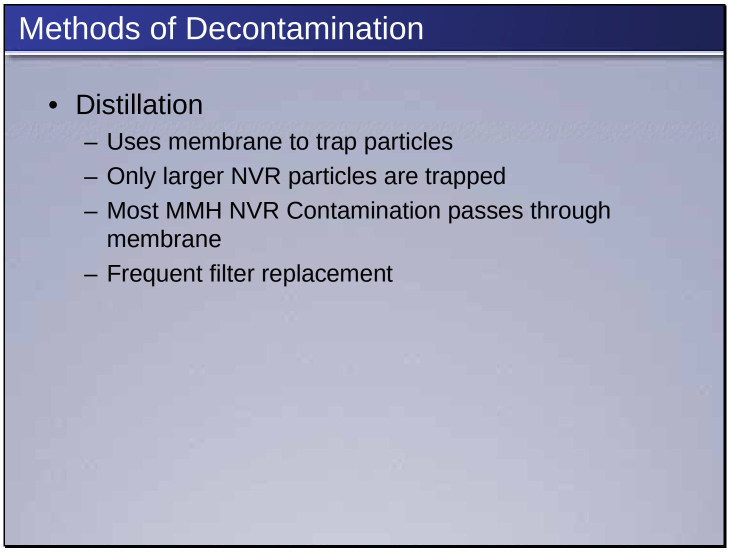

Distillation is based on the volatilities of substances being separated and consists of the addition and removal of heat. Any compound that can evaporate can be separated from nonvolatile substances using this technique and extra waste is not generated. The process materials are non-selective and can be used with any propellant as long as the boiling point range and autoignition temperature is known. The still can also be constructed with compatible materials, easily stored, cleaned, and operated. In addition, distillation is used at WSTF to determine the concentration of NVR in a one hundred milliliter sample by evaporating the propellant from the sample and collecting only the contamination. Other measures can be made with distillation to increase safety and efficiency.

Distillation is the separation of the constituents of a liquid mixture via partial vaporization of the mixture and separate recovery of vapor and residue.

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Methods of Decontamination

Distillation

Name	Formula	Boiling Point (K)
MMH	NH3CH3	362
Nitrogen	N_2	77
Dimethlydiazine	$CH_3N=NCH_3$	NA
Ethylene oxide	$C_2 H_4O$	284
Ammonia	NH_3	240
Methylamine	CH_3NH_2	267
uns-Dimethylhydrazine	$(CH_3)_2NNH_2$	336
sym-Dimethylhydrazine	$CH_3NHNHCH_3$	NA
Formaldehyde methlyhydrazone	$CH_3HNN=CH_2$	NA
Methoxyethane	$CH_3OCH_2CH_3$	281

NA = Boiling point not available

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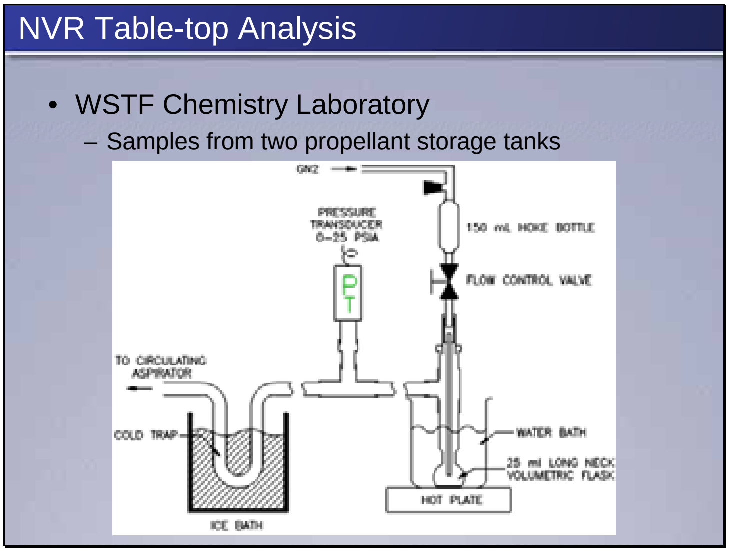

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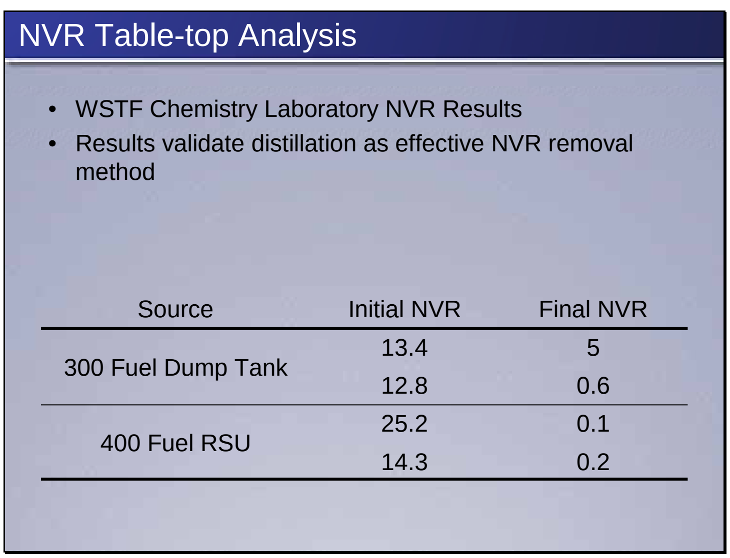

These were the results.

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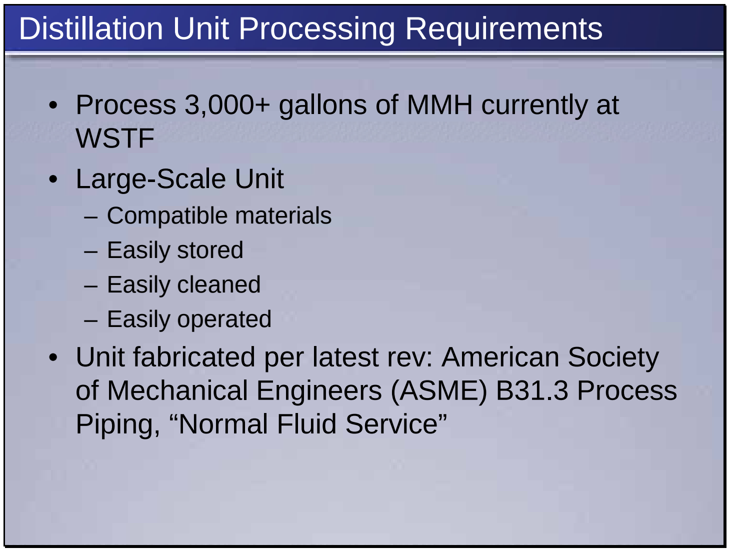

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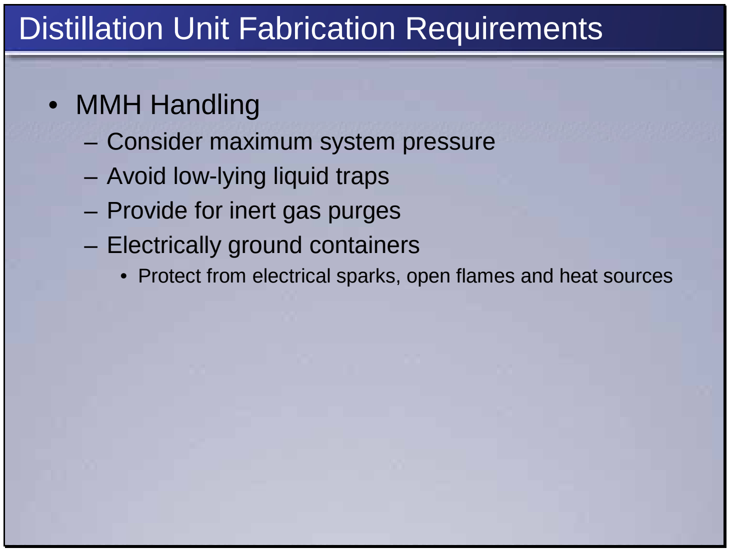

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- Electrically ground the holding container electrically and protect it from electrical sparks, open flames and heat sources

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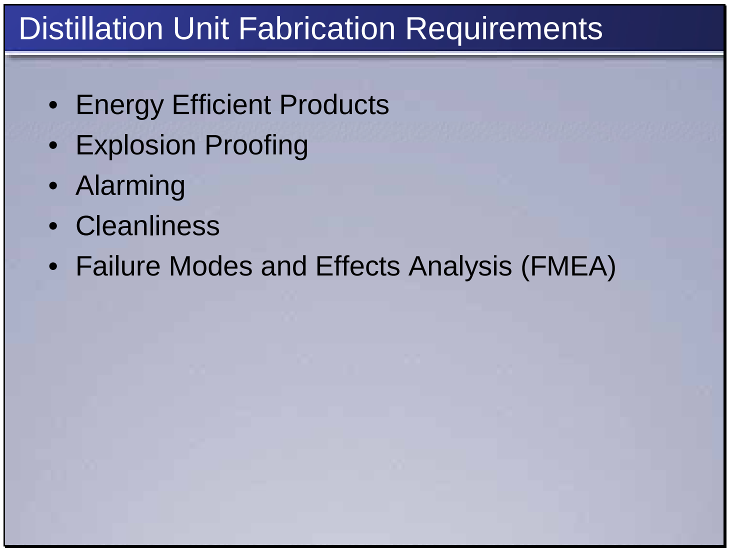

MMH is highly reactive with oxygen in air, oxidizing agents, organic matter, and oxides of metals such as iron, copper, lead, manganese, and molybdenum. Because MMH is so reactive, storage conditions must be chosen carefully.

Although it is not susceptible to decomposition through normal impact or friction (hydrodynamic shear), MMH reacts with oxygen and carbon dioxide in the air; therefore, the unit shall be designed so that MMH is stored and operated under a nitrogen blanket.

Guidelines for designing safe storage and processing for hydrazine fuels include:

- Use a minimum of mechanical joints
- Consider maximum system pressure
- Avoid low-lying liquid traps
- Provide for inert gas purges
- Electrically ground the holding container electrically and protect it from electrical sparks, open flames and heat sources

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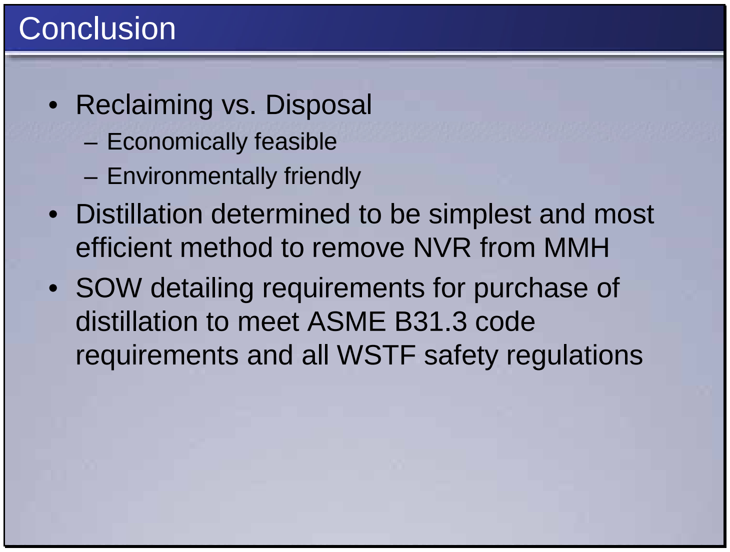

Reclaiming contaminated MMH currently at WSTF for re-use was an attractive alternative to disposal for two main reasons; cost savings and environmental.

Three feasible methods of NVR removal were considered: filtration, adsorption, and distillation from which distillation was determined to be the simplest and most efficient method to remove NVR from MMH.

A statement of work detailing requirements for the purchase of a distillation unit was written to meet ASME B31.3 code requirements and all WSTF safety regulations, of which some are included here.

It is anticipated that this distillation unit will be fabricated so as to meet all code and safety requirements while removing high concentrations of NVR from MMH for re-use.

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Future Work

Build compatible systems for the remediation of other contaminated propellants