Characterization of Monomethylhydrazine (MMH) Non-Volatile Residue

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ASTRACT

The Space Shuttle program has a unique propellant purity requirement for determination of non-volatile residue (NVR) in monomethylhydrazine (MMH). This requirement differs from the Military Specification procurement specification by requiring a NVR analysis with a limit of less than or equal to 10 milligrams per liter. In June 2008, a routine MMH replenishment delivery was transferred into a NASA KSC owned tanker for future delivery to the Space Shuttle pad MMH storage tank. Per Shuttle standard operating procedure, the receiving tanker was sampled and analyzed for purity and surprisingly it failed the Shuttle use NVR specification limit. Detailed examination of the NVR revealed that it was fundamentally different than the typical MMH NVR. This paper will examine various aspects of NVR determination in MMH and the analytical characterization processes used to identify the NVR.

INTRODUCTION

Monomethylhydrazine (MMH) is used as a fuel with dinitrogen tetroxide (N_2O_4) oxidizer in the NASA Space Shuttle orbiters' Orbital Maneuvering System (OMS) and Reaction Control Systems (RCS). Shuttle Program document, SE-S-0073, Fluid Procurement and Use Control Specification, for the various fluids and propellant typically mirror the Military procurement "PRF" specifications. However, SE-S-0073, Table 6.3-9 for (MMH) has a unique requirement to measure the non-volatile residue (NVR) in MMH instead of the particulate that is required by the MIL-PRF-27404C, Propellant Monomethylhydrazine. The rationale for this difference is that particulate can be mitigated with filters but NVR cannot. On a re-usable system such as the Shuttle OMS/RCS, NVR accumulation over years of service could cause decreased performance and potentially operational failures.

MMH Analysis	MIL-PRF-27404C Procurement	SE-S-0073, Table 6.3-9 Shuttle Use
Qualitative	Colorless, homogeneous liquid	None
Assay, % by wt.	98.3 (min.)	98.0 (min.)
Density @ 77° F (25° C)	None	Engineering Info Only
Water, % by wt.	1.5 (max.)	2.0 (max.) plus soluble impurities
Nonvolatile Residue, mg/L	None	10 (max.)
Particulate, mg/L	10 (max.)	None

Table 1: Specification Analysis Requirement Comparison.

MMH NVR DETERMINATION METHOD

Given there is no industry standard method for MMH NVR determination, the aerospace propellant community laboratories developed a method based on the NVR determination in hydrazine from MIL-PRF-26536, Propellant Hydrazine. The method summary is:

1. Low temperature (135 ºC) evaporation of MMH sample in platinum or Pyrex glass evaporation dishes (~ 3-4 hours) on an explosion-proof hot plate contained inside a fume hood. Sample size

is typically limited to 40 milliliters (mL) with 10 mL of water added to prevent auto ignition. Larger sample volumes are impractical due to the extremely slow evaporation rate needed to prevent auto ignition and the ability to manage a potential fire.

2. The evaporation dish is removed from the hot plate when 1-2 mL remains, then the dish is evaporated to dryness in a 105°C oven for thirty minutes to the final dryness. The dishes are stored inside desiccators to equilibrate to room temperature before weighing. Care must be taken to not "over-bake" the NVR dish.

3. NVR is calculated by weight difference of the dish after ambient temperature equilibration.

4. The total NVR is extrapolated to the mg/L value by multiplying the calculated NVR by 25. A maximum NVR weight of only 0.40 mg (from the 40 mL sample) is needed to fail the limit of 10 mg/L. Much of the reproducibility is due to this large dilution factor and by applied heating rate.

Even though this method is technique-sensitive; reproducible results have been obtained during 25+ years of use by NASA. Historically, MMH NVR is generally in the range of 3-5 mg/L.

A classic reason for either the NVR or Particulate failure is due to contamination with the fluorinated grease used to lubricate the system valves and fittings. This type of failure is typical in satellite propellant fueling operations, where the ratio of the MMH product to internal system volume is low. Grease is liberated from the components when opened to take the analysis sample.

LT-90 MMH NVR FAILURE

Liquid Tanker, LT-90, Figure 1, is one of four KSC-based tankers used for hypergolic fuel storage and delivery at KSC. On June 12, 2008, KSC received the vendor tanker VT-1994, containing approximately 18,000 pounds (~ 2460 gallons) of MMH conforming to MIL-PRF-27404C. The VT-1994 contents were transferred into LT-90 and sampled post-transfer per standard KSC operating procedure. The initial sample failed NVR results at 16 mg/L. The following week, on June 24, the tanker's fill/drain valve was sparged with helium and allowed to re-fill with MMH from within the tanker, and then a second sample was taken. This sample also failed the NVR level test at 18 mg/L, thus confirming the initial failure. Both LT-90 analyses were performed by the Aerospace Fuels Laboratory at Cape Canaveral Air Force Station (CCAFS - Cape Lab).

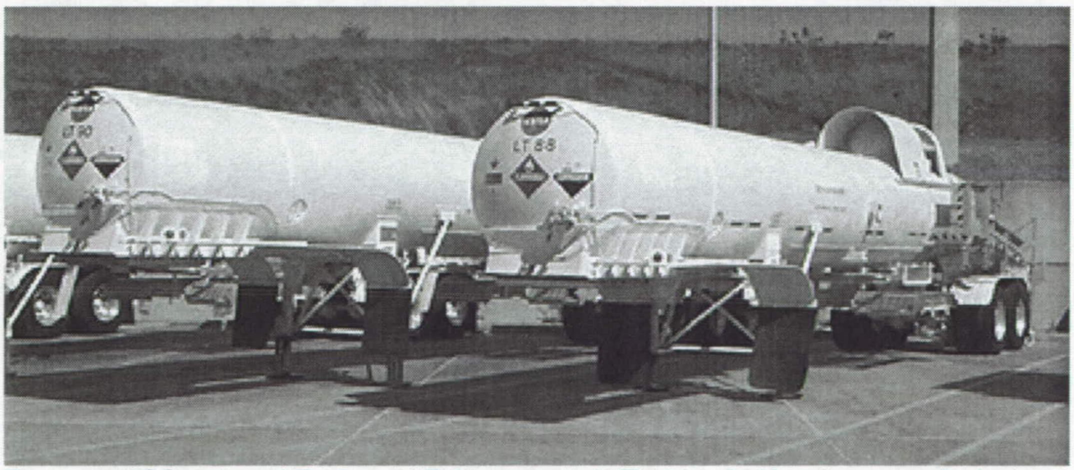

Figure 1: KSC tankers LT-88 and LT-90, typical of VT-1994, 2500-gallon capacity fuel tanker.

The tankers are of identical design and constructed of 304L stainless steel (SS). Each has been in continuous MMH-service with typical MMH sample test results since their last five-year recertification testing process. MMH is manufactured and stored by Arch Chemicals Corp., Lake Charles, LA. Arch uses railcars for storage vessels. The DODX-series railcars are constructed of 304L SS and entered service in 2007. The DAF-series railcars are constructed of 6061 aluminum and have been in service for well over ten years. In accordance with the KSC material selection guide, KTI-5211, these metals are accepted for long-term hydrazine family fuel storage. All of the railcars were filled with MMH in 2007.

In early July 2008, NASA initiated discussions Arch and the Defense Energy Support Center (DESC), Missile Fuels Business Unit, in San Antonio, TX, about the failed MMH sample results. The group decided to:

1. KSC repeat the LT-90 NVR test.
2. KSC trace LT-90's historical NVR levels.
3. DESC/Arch test the retained MMH sample fromVT-1994.
4. DESC/Arch test the MMH inventory contained within five railcars at the Arch facility.

Arch does not have an in-house NVR analysis capability, so the analysis service was sub-contracted to an outside lab. The outside lab could not reproduce the NVR results from VT-1994 that KSC found or produce a typical MMH NVR result on any of the other railcars (all NVRs were reported <0.1 mg/L), well below the historical range of 3-5 mg/L. Therefore, it was decided that samples from all MMH railcars and VT-1994 would be shipped to both KSC/CCAFS laboratories for analysis by the Cape Lab and Wiltech, the NASA-KSC contractor-operated laboratory. The results are presented in Table 2.

Vessel ID	**NVR Test Results (mg/L)**		**Comment**
	Cape Lab	**Wiltech**	
DAF 17054	20	25	VT-1994 source, unique NVR
DAF 17072	10	6	
DAF 17074			Almost Empty not sampled
DODX 7009	12	15	2x runs by WT, typical NVR
DODX 7010	5	3 (28*)	* no source filter, high solids
DODX 7011	5	10	Source of replacement to original LT90 delivery order
VT-1994	N/A	18	Delivered MMH into LT-90, June 12, 2008, from the "retain" sample tested in Dec'08
LT-90	12-18	20.0-28.8	Multiple NVR tests, unique NVR

Table 2, Summary of NVR Results by container and laboratory, August 2008

It was immediately obvious from the Table 2 NVR test results that storage railcar DAF 17054, which was the source of the VT-1994 MMH then transferred into LT-90, all have similar NVR levels at approximately 20 mg/L and higher. Second, both of the NVR's contained unique, needle-like crystals (Figure 2) as compared with the characteristic bubbled resinous MMH NVR (Figure 3). Note; photograph scales are approximately equal.

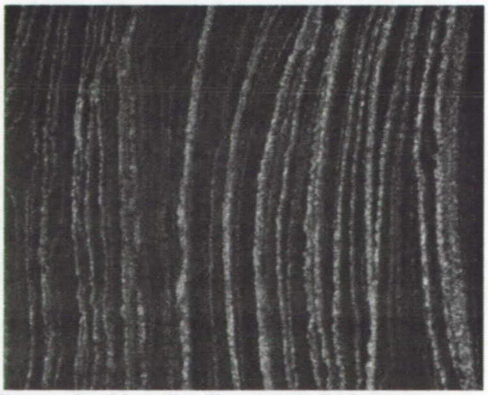

Figure 2. Needle-like crystals from LT-90 and VT1994 MMH NVR.

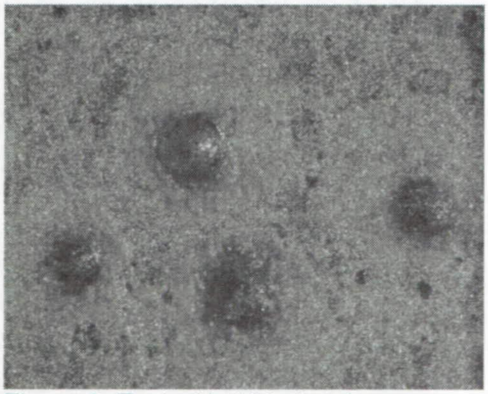

Figure 3. Typical bubbled resinous MMH NVR solids.

On Nov 4, 2008, VT-1994 returned to KSC and the 2400 gallons of high-NVR MMH was transferred from LT-90 back into VT-1994. The product was returned to Arch were the MMH was transferred back into original source railcar DAF 17054. At this point there are two tankers empty but contaminated with unique high-NVR MMH residue, railcar DAF 17054 nearly full of unique NVR MMH. What is this unique NVR, how can the product be recovered, and how can the tankers be returned to routine service?

CHARACTERIZATION OF UNIQUE MMH NVR

It is standard KSC policy to identify all NVR failures to determine if the NVR is either "normal" contamination from the sampling process (typically aerospace-class fluorinated grease), from the system itself, or from an outside source from cross contamination. The KSC Materials Science Division routinely uses a multi-analytical approach in the identification of NVR unknowns using the following techniques:

1. Optical stereo microscopy (OP) using oblique and coaxial illumination to determine the nature and morphology of the contamination.
2. Determination of the solubility of the NVR solids.
3. Fourier transform infrared spectroscopy (FT-IR) to identify the molecular structure.
4. Scanning electron microscopy using energy dispersive spectroscopy (SEM/EDS) support morphology and determine the elemental composition and homogeneity of the residue.

Optical microscopy observation is the first evaluation to determine the morphology and number of phases of an unknown. Whereas oblique illumination provides good topography of solids, it is extremely limited at discerning liquids and almost impossible to see thin clear films. The ability to observe thin films is the strength of coaxial illumination. Coaxial illumination, by the nature of the light being coincident to the optical path, all anisotropic materials (two or more refractive indexes), exhibit interference color of first and higher orders (2nd, 3rd, etc.) in what is call birefringence, as is typical for common clear fluorinated oil shown in Figure 4.

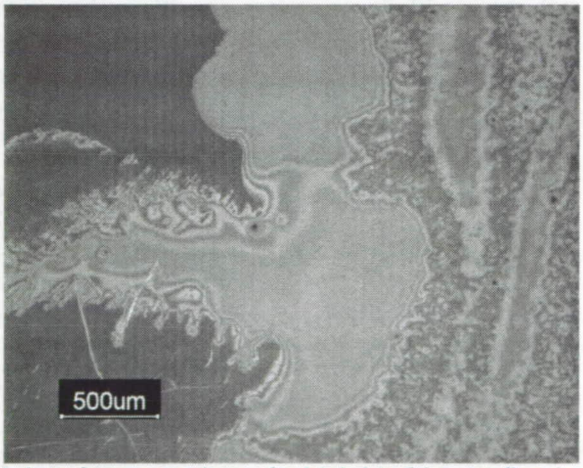

Figure 4. Interference colors of a high birefringent fluorinated oil.

The birefringent colors of the first, second, etc. orders (noted by the red bands) resemble the contour lines in topography. The higher order of colors correlate with the film-thickness of the oil. This optical property is extremely useful in determining sampling for FTIR analysis, where the film thickness is proportional to strongest absorbance. All anisotropic crystals exhibit birefringence colors and therefore provide a quick evaluation of pure crystalline compounds.

In the case of the LT-90 NVR, observation of the NVR dish by optical microscopy using oblique lighting, showed the typical MMH NVR (Figure 3). However the amount of the typical NVR solids did not correlate with the reported weight. Inspection of the NVR dish using coaxial lighting discovered large collections of extremely thin (< 5 microns thick) birefringent crystals covering the outer edges of the NVR dish as seen in Figure 5 and 6. These thin crystals went unnoticed while using oblique lighting. The crystals were observed as evaporation deposit lines created as the last few milliliters of MMH evaporated.

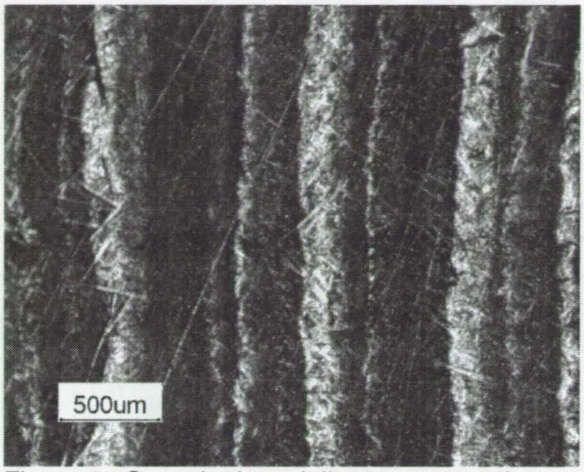

Figure 5. Crystals deposit lines at outer edge of the NVR dish.

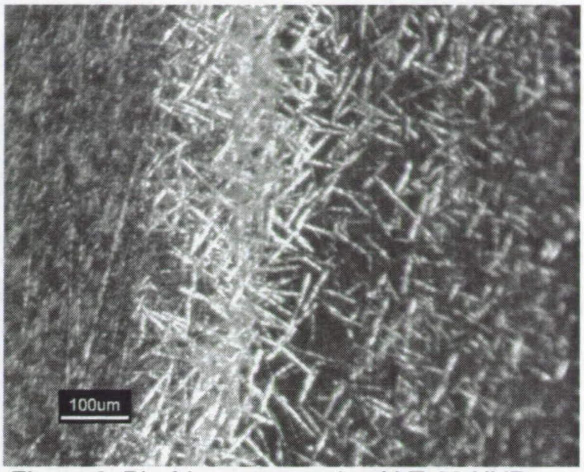

Figure 6. Birefringent crystals of LT-90 NVR.

Solubility is an important physical property of an unknown, not only for determining a suitable cleaning solvent, but also allows transferring the unknown sample for FTIR analysis or other techniques. What must be remembered about solubility is "like dissolves like", so never assume only one solvent will work. A series of solvents, covering the polarity range from non-polar to polar, should be evaluated. MMH would be the best solvent for the MMH NVR but is impractical in the laboratory environment due to its toxicity. Suitable solvents should have low vapor pressure (limited volatility) and high surface tension. These requirements are met by n-decane, amyl acetate, methanol and Castrol® Fluoroclean™ X100 (a fluorinated solvent). Fluoroclean X-100 is the one exception that fails both vapor pressure and surface tension, making it extremely difficult to control. However, Fluoroclean is an efficient solvent for the fluorinated aerospace greases of Braycote and Krytox. In the case of the LT-90 NVR, methanol was found to be the only solvent that dissolved the subject birefringent crystals. The solubility of the crystals in methanol not only allowed the crystals be transferred from the original platinum dishes for FTIR analysis but also allowed the unknown to be re-crystallized into much larger crystals as shown in Figure 7. The extremely high birefringent needles crystals are typical of aromatic organic crystals.

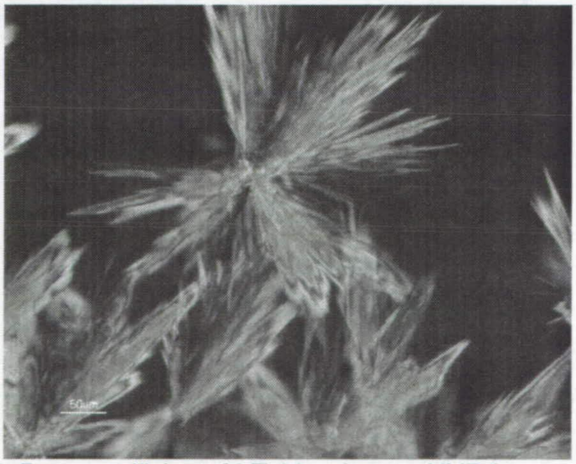

Figure 7. Re-crystallizing of LT-90 unknown NVR from methanol.

Fourier transform infrared spectroscopy (FTIR) is the primary instrument for molecular structure identification. Typical MMH NVR is characterized as a polymerized by-product of MMH. The FTIR spectra of the typical MMH NVR is compared to the unknown crystals as shown in Figure 8. The unknown NVR found in tanker LT-90 and the railcar DFA 17054, was characterized as an aromatic amine. The proposed chemical structure shown in Figure 9.

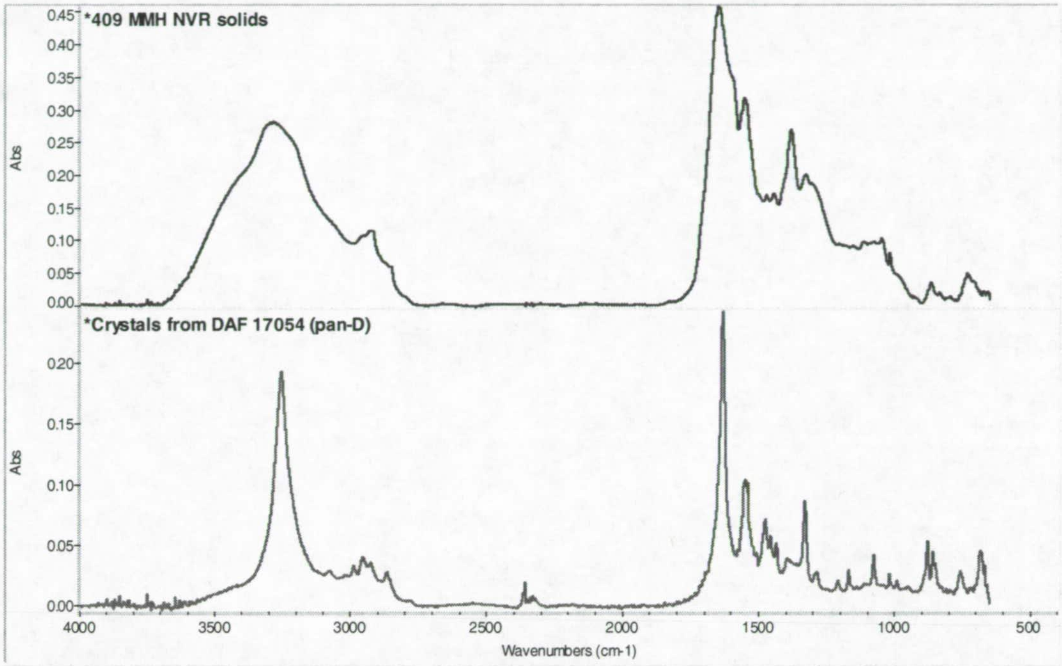

Figure 8. FTIR Spectra of typical MMH polymerized NVR, top (blue), as compared to the crystals found in both LT-90 and DAF 17054, bottom (red).

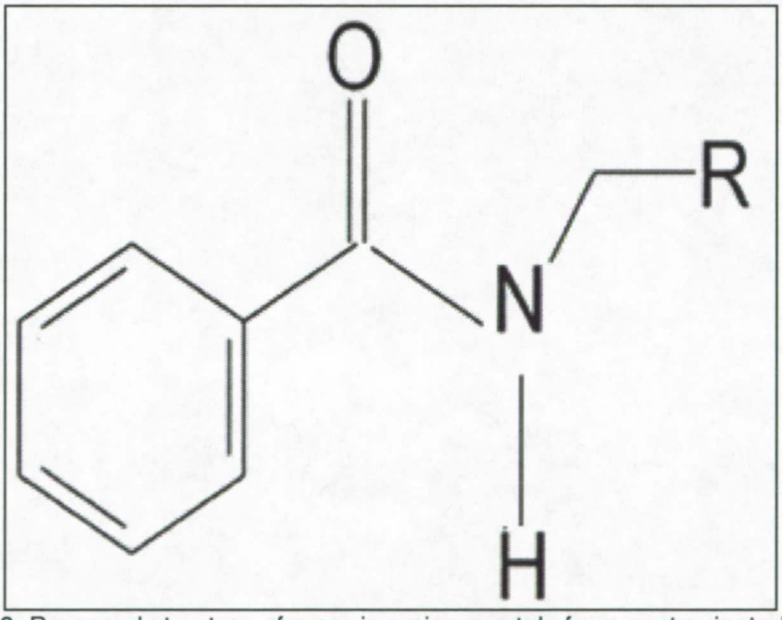

Figure 9. Proposed structure of organic amine crystals from contaminated MMH.

SEM/EDS elemental analysis confirmed the composition of the crystals to be homogeneous material with only carbon, oxygen and nitrogen elements present.

This information has been shared with DESC and Arch for their evaluation of the Lake Charles plant systems and components as to what and where this substance may have originated.

MMH HIGH-NVR RECOVERY OPTIONS

Several options have been considered and some tested to determine the most economical way to return the contaminated containers to specification grade MMH service and how to mitigate the NVR.

1. High-NVR MMH: Dilution of affected MMH with "good" MMH. A calculated 70%-typical-to-30%-unique NVR MMH blend should lower the final NVR below the 10 mg/L limit. This will not remove the "the out of family" aromatic amine contaminate but will minimize any effect of contaminate on performance by diluting it to below the allowable limit. Lab experiments with this "hybrid" NVR produced of results of 9.8 mg/L and 12.0 mg/L. The dilution appeared to only reduce the size and alter to more plate-like morphology of the aromatic amine crystals. Given enough dilution, the original contamination will be below the limit of detection. However, we do not currently plan to determine the limit of detection. Other potential MMH recovery methods could entail re-distillation of the DAF 17054 MMH at Arch's facility. However, further determinations are beyond the scope of this paper.

2. Tankers: There is concern of a residue film remaining on the interior surface of the tankers when the high-NVR MMH was drained from both LT-90 and VT-1994. During various NVR test runs, it was observed that the unique NVR is readily soluble in MMH. Therefore, LT-90 and VT-1994 could be "flushed" with typical MMH to remove and dilute any remaining NRV. This theory was tested on Dec 21, 2008, by flushing LT-90 with about 50 pounds (~ 7 gallons) of typical ~ 3 mg/L NVR MMH the full-length of the tanker, washing over the area where the last bit of fuel would have flowed when LT-90 was drained into VT-1994 the previous month. This flush was captured and measured to have 10.3 mg/L NVR and no evidence of the birefringent crystals. Thus, the NVR increased about 3-fold but lost its unique birefringence. Alternatively, lab testing demonstrated the unique NVR readily dissolved in methanol, therefore, 2-propanol (isopropyl alcohol) should provide similar solvency. 2-propanol is a standard solvent used at KSC for cleaning hypergolic propellant fuel systems and components. This tanker recovery option is currently under review by NASA

3. Future care must be taken with tracking the contaminated MMH now held in railcar DAF 17054 so it will not unknowingly re-enter the general-use inventory. Its fate is currently undetermined.

SUMMARY

Routine quality control testing is vanguard towards ensuring mission success and safety. Specification testing of the LT-90 MMH flagged a failed product before it could enter Shuttle ground support equipment. More specifically, the NVR test discovered an out-of-family contaminant thereby preventing the fuel's potential use in flight hardware and avoiding costs associated with removal of an out-of-specification product and resultant contamination.

However, basic quality test methods only produce a single data point of pass-fail criteria. Complete characterization of the unknown is required to trace back to the potential contamination source. Numerous analytical techniques beyond the basic Mil-spec test methods should be routinely applied to characterize typical NVRs. It is standard KSC policy to identify all NVR failures to determine if the NVR is "normal" contamination is due to sampling, from the system itself, or from an outside source from cross contamination. KSC's analytical testing traced the unique MMH NVR contamination back to the source. Furthermore, solubility testing revealed a practical route to decontaminate tankers LT-90 and VT-1994 so they may be economically returned to service.

Further evaluation of the vendor system component materials and processes is recommended to identify and prevent future contamination recurrence.

ACKNOWLEDGEMENTS

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Robert Lee, Aerospace Fuels Laboratory, Cape Canaveral Air Force Station, FL

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CL 2008LH02572001 – 24Jun2008 MIL-PRF-27404 + SE-S-0073 test report from LT-90
CL 2008LH02899001 – 15Aug2008 MIL-PRF-27404 + SE-S-0073 test report from LT-90
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KSC-MSL-2008-0366 - Identification of MMH NVR Resample from LT-90
KSC-MSL-2009-0379 - Identification of NVR of a 70:30 Mix from LT-90 and Known MMH
KSC-MSL-2008-0410 - Identification of MMH NVR From Trailers
KSC-MSL-2008-0451 - Identification of MMH NVR From Railcars

CL = Cape Lab
WT = Wiltech
KSC-MSL = NASA Material Science Laboratory

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By:

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Renaissance Las Vegas

Introduction

- In-orbit, the Space Shuttle Orbiter uses bipropellant propulsion systems
  - MMH and N2O4
  - OMS for altitude adjustment
  - RCS for attitude control and steering
- MMH manufacture
  - By Arch Chemicals
  - Stored on-site in…
    - 6061 Al
    - 304L SS
    - railcars

MMH Specifications

Analysis	MIL-PRF-27404C Procurement	SE-S-0073 Shuttle Use
Qualitative	Colorless, homogeneous liquid	None
Assay, % by wt.	98.3	98.0 (min.)
Density @ 77° F (25° C)	None	Engineering Info Only
Water, % by wt.	1.5 (max.)	2.0 (max.) plus soluble impurities
Nonvolatile Residue, mg/L	None	10 (max.)
Particulate, mg/L	10 (max.)	None

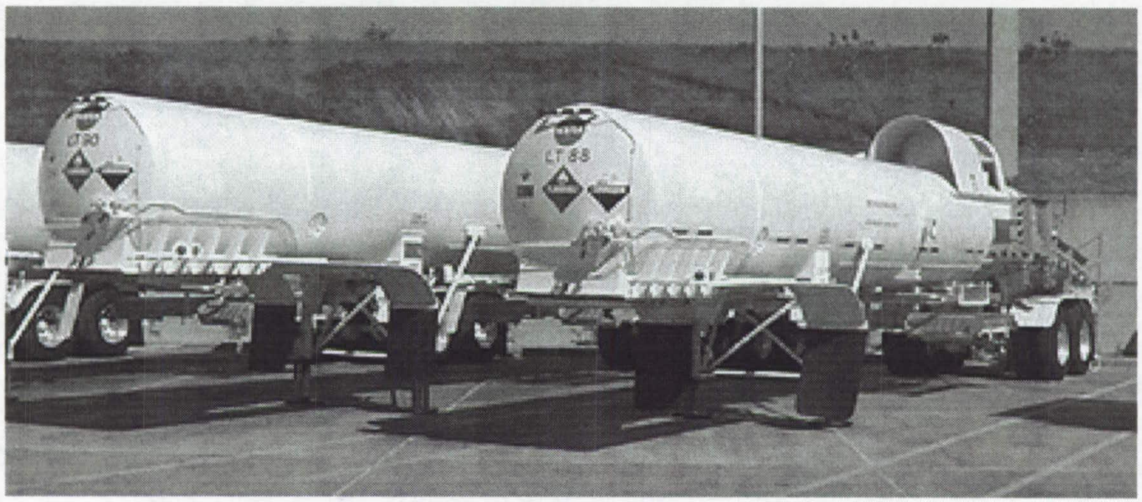
Many of the SE-S-0073 requirements are mirrored the MIL specifications

MMH NVR Test Method

- Gradual evaporation of MMH sample in platinum or Pyrex glass evaporation dishes (~3-4 hours)
- Explosion proof hot plate in a fume hood
- Sample size is limited to 40 mL plus 10 mL water to prevent auto ignition
- The NVR is calculated by weight difference
- Extrapolated to mg/L (0.40 mg max from 40 mL sample)

Routine Delivery Test Anomaly

- Tankers constructed of 304L SS and have long MMH service history
- Routine vendor tanker delivery, VT-1994, received at KSC on June 12, 2008
- Transferred into KSC tanker, LT-90
- Post transfer LT-90 sample analysis failed SE-E-0073 NVR test requirement



Summary of NVR Test Results

Vessel ID	NVR Test Results (mg/L) Cape Lab	Wiltech	Comment
DAF 17054	20	25	VT-1994 source, unique NVR
DAF 17072	10	6	
DAF 17074			Almost Empty not sampled
DODX 7009	12	15	2x runs by WT, typical NVR
DODX 7010	5	3 (28*)	* no source filter, high solids
DODX 7011	5	10	Source of replacement to original LT90 delivery order
VT-1994	N/A	18	Delivered MMH into LT-90, June 12, 2008, from the "retain" sample tested in Dec'08
LT-90	12-18	20.0-28.8	Multiple NVR tests, unique NVR

Propellant NVR Usual suspects

- Where did the NVR failure come from?
- Valid reason for NVR Failures - mistakes do happen
  - Container cross contamination
  - Contamination reaction can create by-products or polymerization
    - Higher NVR samples tend to be yellow in color
  - Sampling and analysis process contamination
    - Classic failure mechanism - fluorinated lubricants from GSE valves or fittings
    - Debris from the lab - fiberglass and cotton fibers
- Routine optical inspection of NVR to baseline “Normal”

NVR Identification - Multi Analytical Approach

- Optical microscopy – particle morphology
- Microextraction – solubility
- Fourier transform infrared spectroscopy (FTIR) for molecular identification
- Scanning electron microscopy energy dispersive spectroscopy (SEM/EDS) morphology and elemental analysis

Optical Microscopy

- Morphology of the NVR
  - Nature of the NVR either solids or liquids
  - Positive identification of specific particles
- Coaxial Illumination
  - Observe optical property of birefringence
  - Crystallinity
  - Can see thin films where no other lighting techniques can
    - Delaminated glass
    - Biological (biofilms)
    - Liquids

Stereomicroscopy Reflected Light Illumination

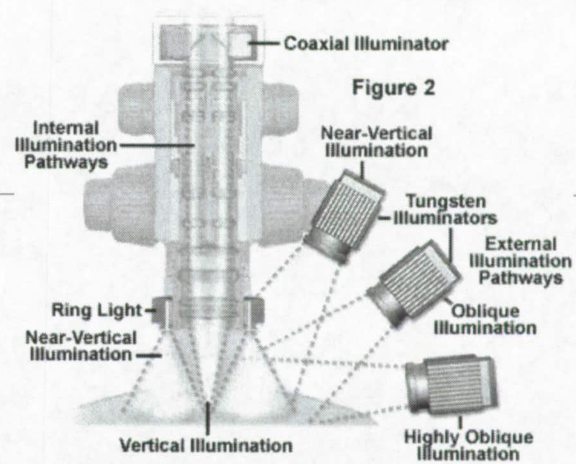
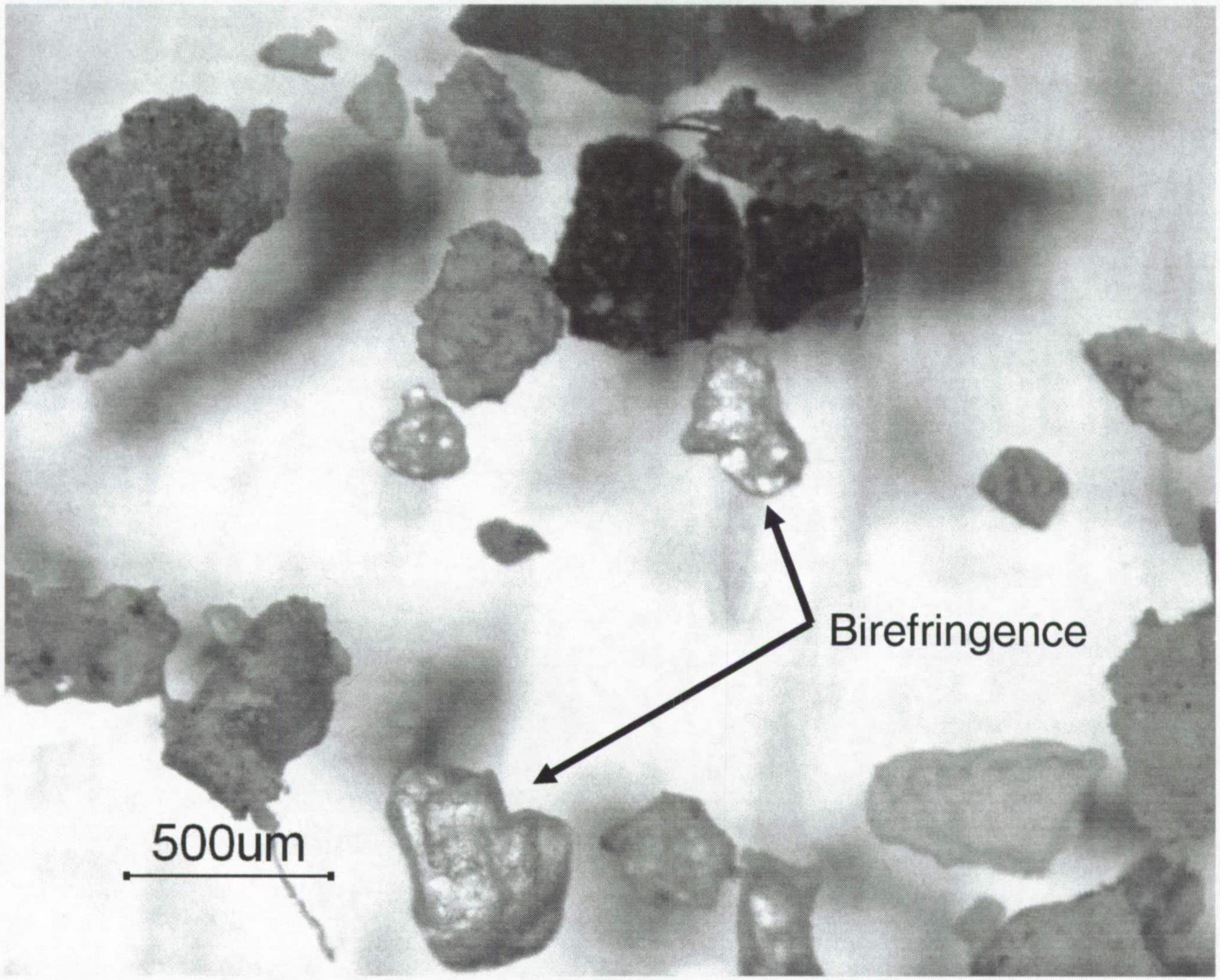
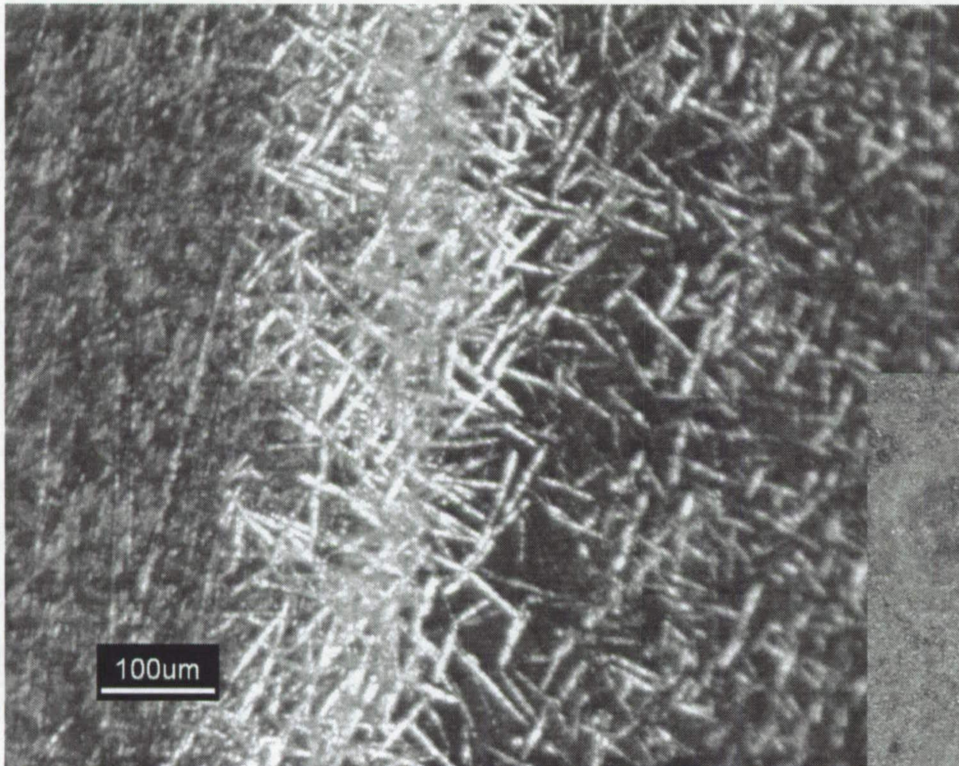


Figure 2

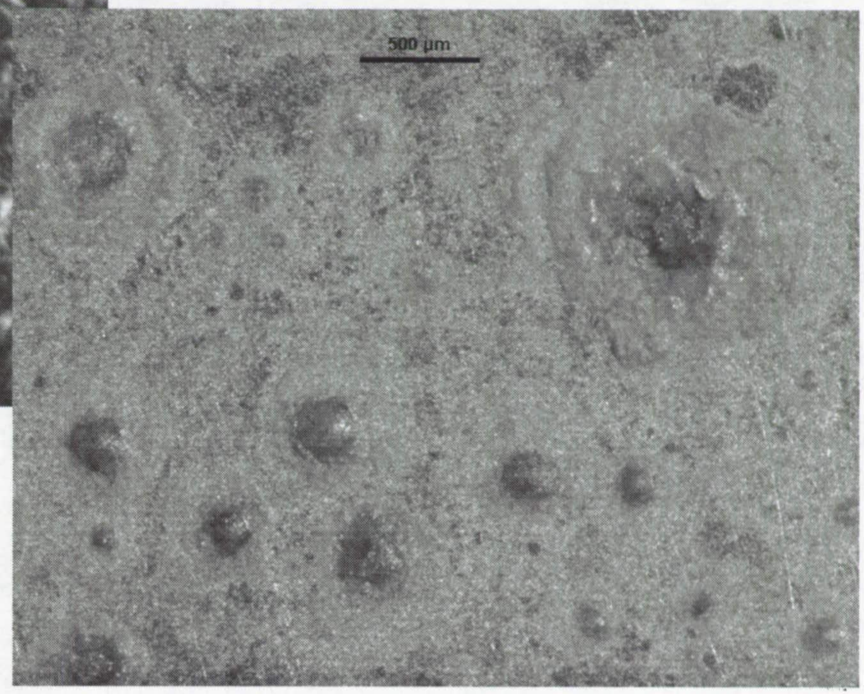
Example of Coaxial Illumination



MMH NVR Morphology

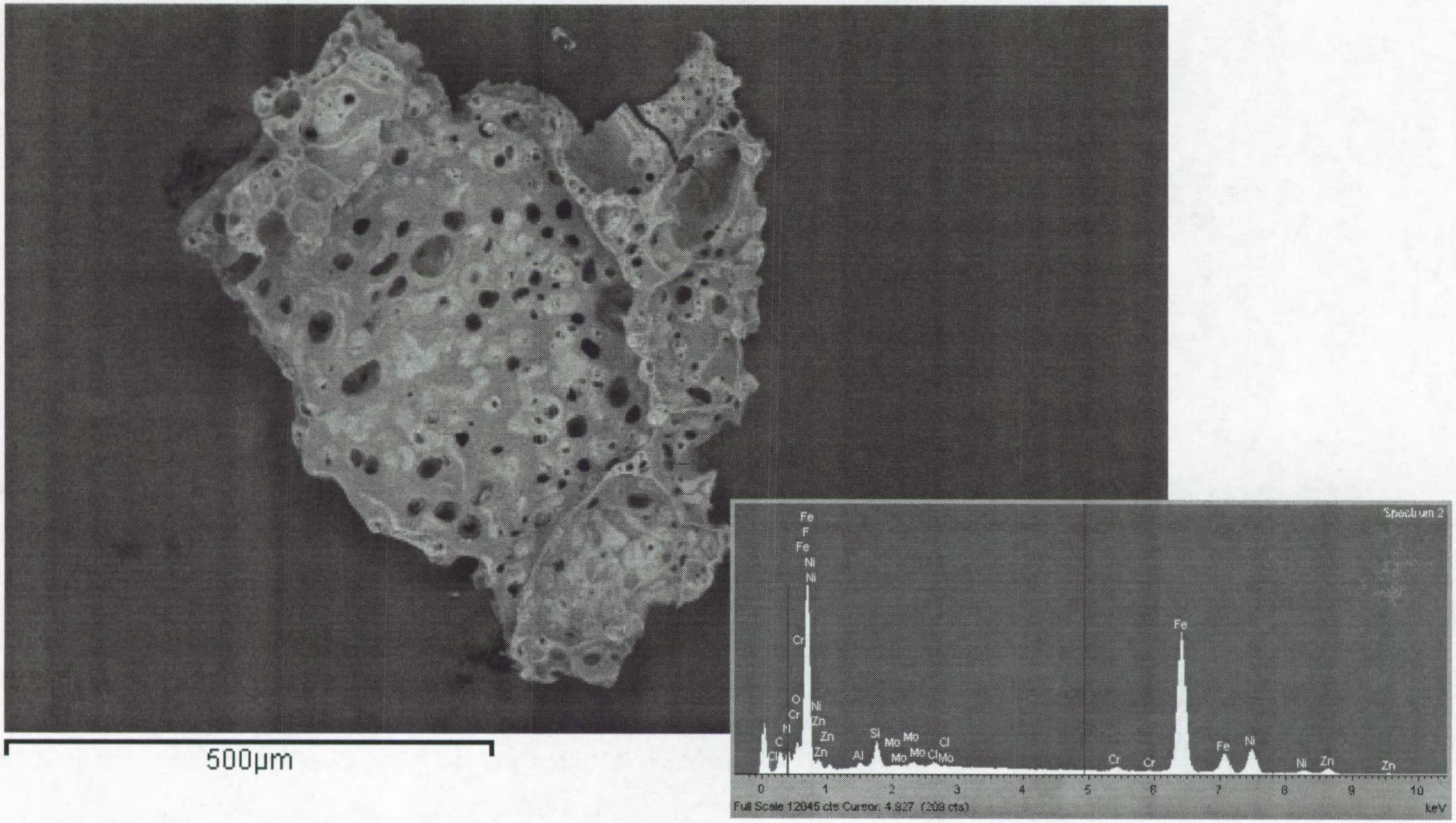


Historical resinous
MMH NVR



Unique needle-like
LT-90 NVR

Typical Resinous MMH NVR



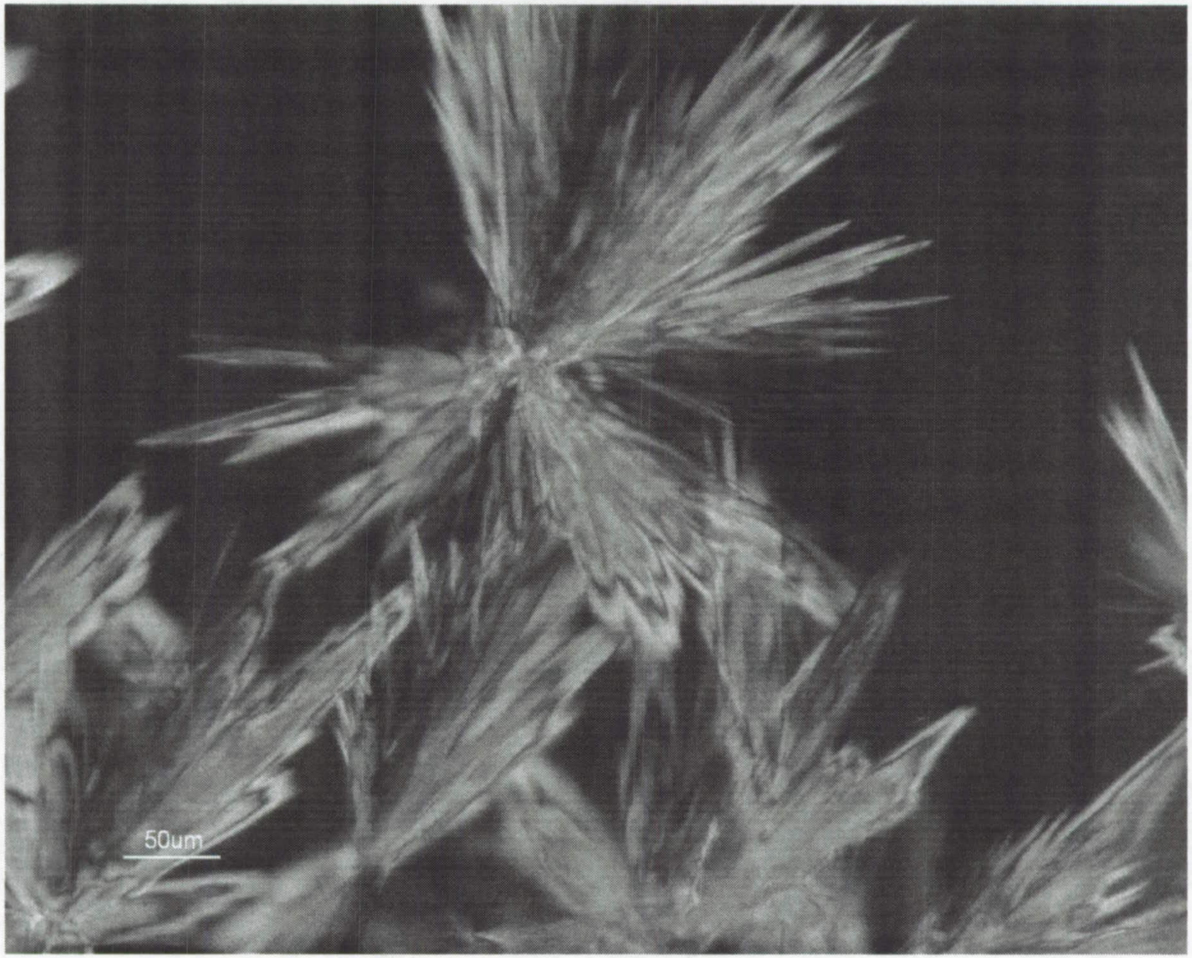
SEM with Four Quad Back Scatter Detector, note SS-reaction products

Micro-extraction

- Relative solubility-intrinsic physical property
  - Micro sample particle < 20 micrometers
- Transferring sample to other techniques
  - FTIR
  - Recrystallization → multiple small samples into single large sample
- Like dissolves like
  - Never assume only one solvent is needed; cover polarity range
    - Fluoroclean X-100
    - N-nonane
    - Amyl acetate
    - Methanol → Worked well with the LT-90 NVR (as does MMH)

LT-90 NVR Crystals

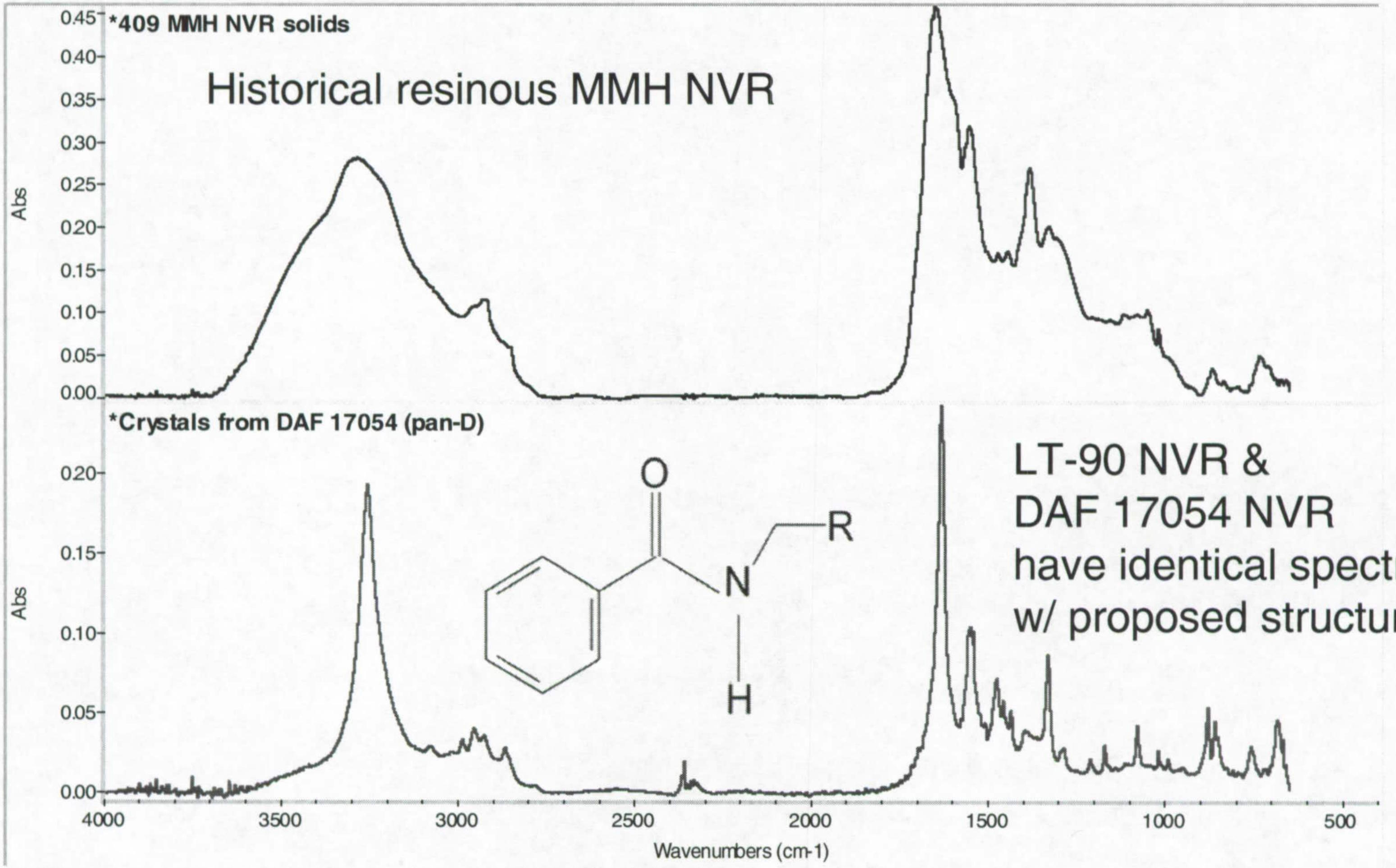
- Isolated Crystals From Methanol



Kennedy Space Center
Center Operations Directorate

FTIR Comparison

Operations Support Division



Conclusions

- LT-90 Contamination
  - Unique C, N, O, H compound
  - Very pure, birefringent crystals
  - NVR contaminant was traced back to the vendor storage tank
- Specification testing will remain a proactive defense against substandard products
  - Alleviates concern of performance on orbit
- NVR is the best test for concentrating unknown contaminates
- Multi-analytical approach in addition to specification testing is needed to better understand the nature of the potential contaminates

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- This project was internally funded by NASA

NASA
Engineering Directorate
Materials Science Division
Kennedy Space Center, Florida

August 5, 2008

KSC-MSL-2008-0366

SUBJECT: Identify Residue in NVR Pans

CUSTOMER: George Krewson/Wiltech/WT

1. FOREWORD

1.1. The non-volatile residue (NVR) of the monomethyl hydrazine (MMH) product sampled from tanker LT-90 failed NVR (20 to 30.0 mg/liter). The NVR limit is 10 mg/liter. A resample of the MMH from LT-90 was taken and the NVR residues were submitted for identification of the residue. A total of four NVR samples were submitted, two primary and two back-up.

1.2. The NVR samples were labeled as follows:

NVR Pan Identification	Sample Source	NVR Results mg/40 mls
G	Primary	0.9
D	Primary	0.9
#5	Back-up	0.9
#3	Back-up	0.9

2. PROCEDURES

2.1. The NVRs were analyzed with optical microscopy (OM) using coaxial illumination.

The solids were micro-extracted with methanol and analyzed by Fourier transformed infrared spectroscopy (FTIR), scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS) and gas chromatography /mass spectrometry GC/MS.

3. RESULTS AND CONCLUSIONS

3.1. Inspection of the NVR dishes with OM identified birefringent needle crystals in all samples.

3.2. Elemental analysis of the crystals by SEM/EDS identified major elements to be carbon, oxygen and nitrogen with trace sodium, chloride and calcium.

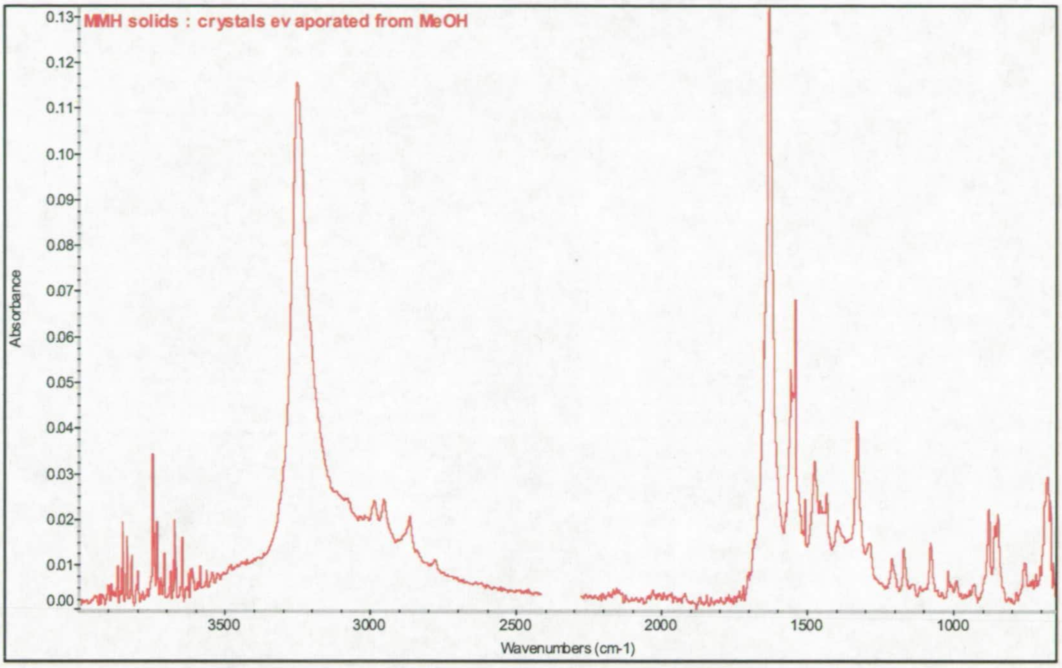
3.3. FTIR analysis of the crystals identified them as a simple amine, potentially a polymerization by-product. See appendix for spectra.

3.4. Analysis by GC/MS indicated that the primary compound is similar to methyl hydroxylamine.

EQUIPMENT:
Thermo Nicolet 6700 Fourier-Transform Infrared Spectrometer, with Continuum Microscope, ECNs 2191216/2191218
Nikon Stereo microscope MSZ-1500, ECN 3059651

PRIMARY INVESTIGATOR: ______________________________
Philip M. Howard/NE-L2

Appendix



FTIR of amine crystals from MMH (LT-90).

NASA
Engineering Directorate
Materials Science Division
Kennedy Space Center, Florida

July 23, 2008

KSC-MSL-2008-0345

SUBJECT: Identify Residue in NVR Pans

CUSTOMER: George Krewson/Wiltech/WT

1. FOREWORD

1.1. The non-volatile residue (NVR) of the monomethyl hydrazine product sampled from tanker LT-90 failed NVR (20 to 30.0 mg/liter). The NVR limit is 10 mg/liter. The NVR residue from both the flush and sample bottles were submitted for identification of the residue. A total of four NVR samples were submitted.

1.2. The NVR samples were labeled as follows:

NVR Pan Identification	Sample Source	NVR Results mg/40 mls
G	Flush Bottle	1.1
#5	Flush Bottle	1.2
D	Sample Bottle	0.9
#3	Sample Bottle	0.8

2. PROCEDURES

2.1. The NVRs were analyzed with optical microscopy (OM) using coaxial illumination.

2.2. The solids were micro-extracted with methanol and analyzed by Fourier transformed infrared spectroscopy (FTIR).

3. RESULTS AND CONCLUSIONS

3.1. Inspection of the NVR dishes with OM identified birefringent needle crystals in all samples as shown in Figures 1 through 4 below.

Figure 1. NVR Pan G

Figure 2. NVR Pan D

Figure 3. NVR Pan #3

Figure 4. NVR Pan # 5

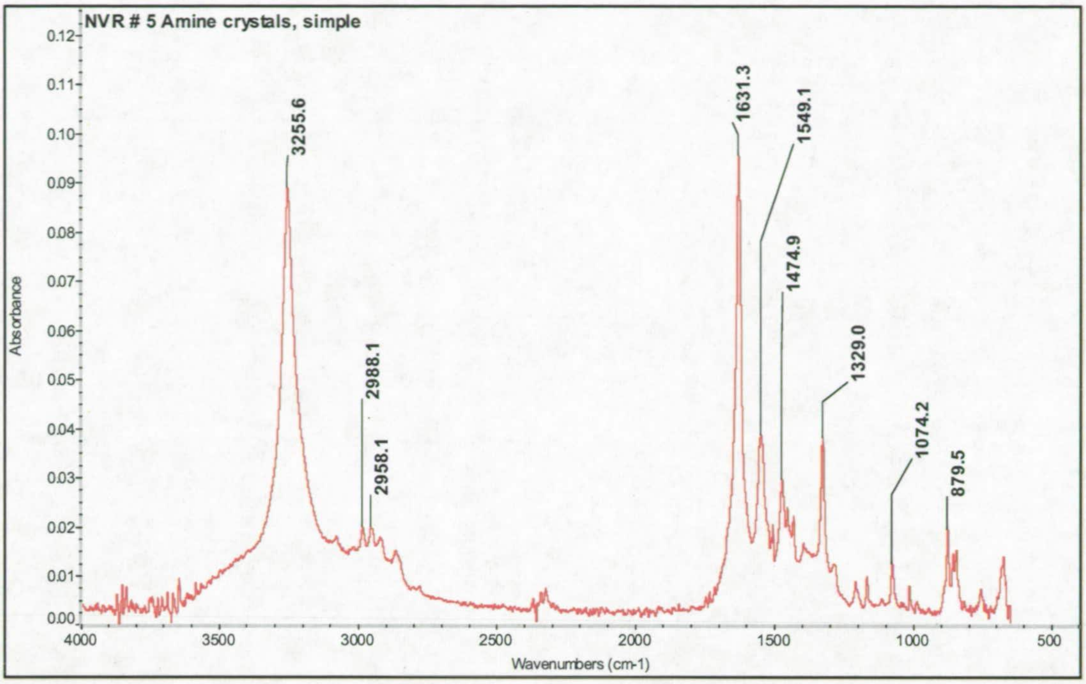
3.2. FTIR analysis of the crystals identified them as a simple amine, potentially a polymerization by-product. See appendix for spectra.

EQUIPMENT:
Thermo Nicolet 6700 Fourier-Transform Infrared Spectrometer, with Continuum Microscope, ECNs 2191216/2191218
Nikon Stereo microscope MSZ-1500, ECN 3059651

PRIMARY INVESTIGATOR: ______________________________

Philip M. Howard/NE-L2

Appendix



FTIR of Amine crystals.

NASA
Engineering Directorate
Materials Science Division
Kennedy Space Center, Florida

October 21, 2008

KSC-MSL-2008-0451

SUBJECT: NVR from Railcars 7009 and 7011

CUSTOMER: George Krewson/Wiltech/WT

1. FOREWORD

1.1. The non-volatile residue (NVR) from two monomethyl hydrazine (MMH) Arch Chemicals railcars were submitted for identification and characterization. The railcars were identified as 7009 and 7011.

1.2. The NVR of the samples were labeled as follows:

Railcar	NVR Pan Identification	NVR Results mg/40 milliliters
7009	8	0.6
7009 Duplicate	5	0.6
7011	C	0.3
7011 Duplicate	10	0.6

1.3. A previous Arch Chemicals MMH shipment to Kennedy Space Center had failed NVR and was found to be contaminated with anisotropic amide crystals, see results of report KSC-MSL-2008-0410 as shown in Figure 1.

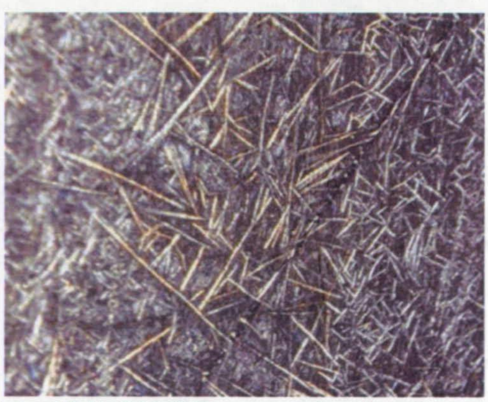


Figure 1. Original MMH NVR contamination from trailer LT-90.

2. PROCEDURES

2.1. The NVR pans were analyzed with optical microscopy (OM) using coaxial illumination.

2.2. The relative solubility of the solids were determined using a micro-extraction technique.

2.3. The solids were also analyzed by Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS).

3. RESULTS AND CONCLUSIONS

3.1. Inspection of the NVR pans with OM showed that the samples contained tan to brown colored blistered solids with gas bubble inclusions as shown in Figures 2 and 3. The solids were observed to be electrostatic upon manipulation. No anisotropic crystals were observed.

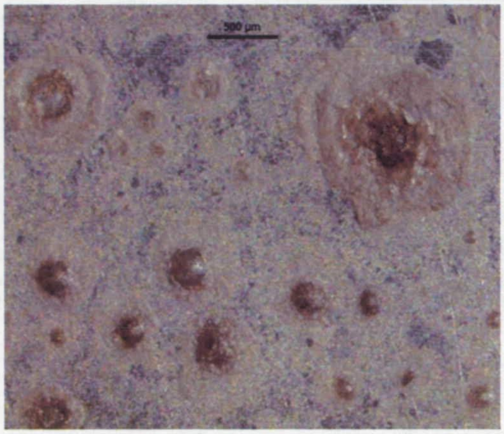


Figure 2. Blistered NVR from pan C.

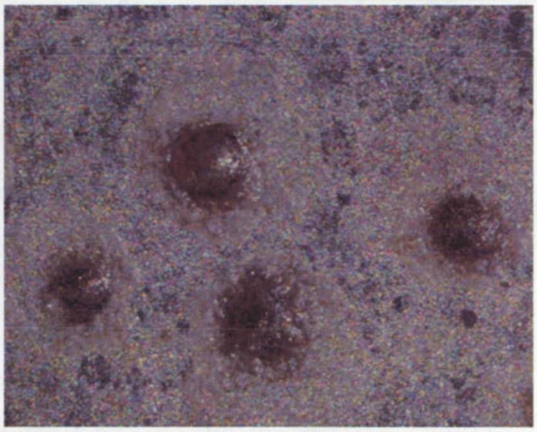


Figure 3. Zoom of pan C.

3.2. SEM/EDS analysis of the NVR solids identified the majority of the materials to be organic (carbon, oxygen, and nitrogen) associated with stainless steel corrosion by-products of iron and nickel oxides.

3.3. Micro-extraction of the MMH solids displayed limited solubility in n-nonane, amyl acetate, Fluoroclean X-100, methanol and water. The limited solubility with a wide range of solvents is consistent with an organic polymer.

3.4. FTIR analysis of the crystals identified a polymerized amine by-product as shown in Figure 4.

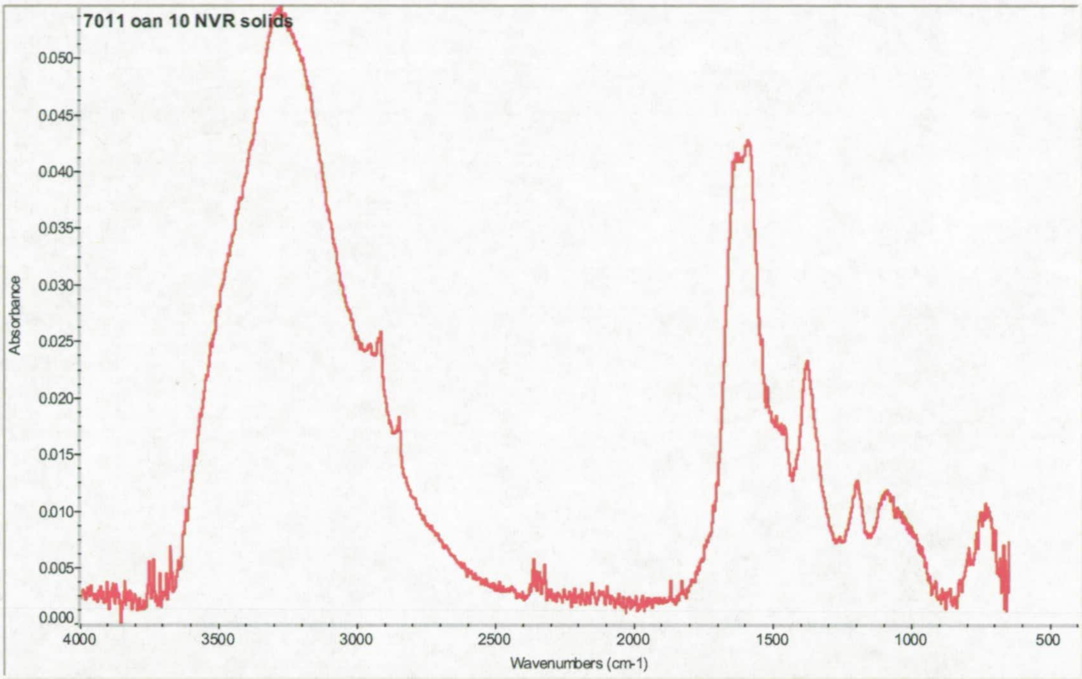


Figure 4. FTIR spectrum of MMH NVR solids from railcar 7011 pan 10 (polymerized amine).

3.5. The SEM four quad back scatter electron detector (QBSD) was used to observe the atomic contrast of iron and nickel solids. The iron and nickel solids appear as the brighter areas Figures 4 and 5.

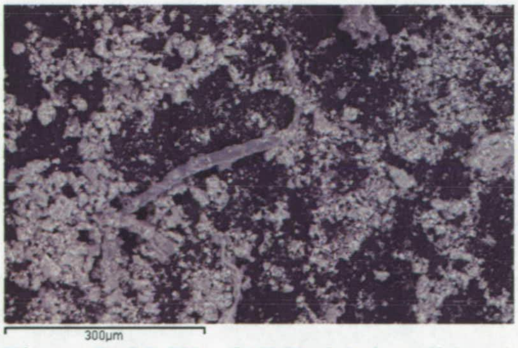


Figure 5. QBSD of NVR debris solids.

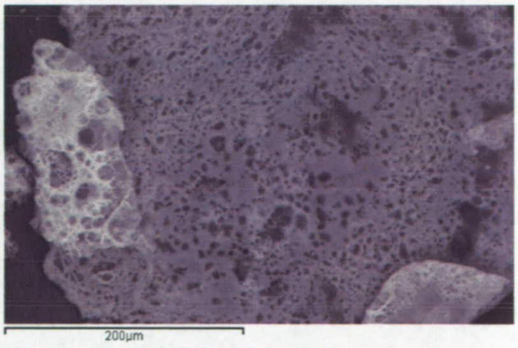


Figure 6. QBSD of the blister.

EQUIPMENT:
Thermo Nicolet 6700 Fourier-Transform Infrared Spectrometer, with Continuum Microscope, ECNs 2191216/2191218
Nikon Stereo microscope MSZ-1500, ECN 3059651
Zeiss EVO 50 SEM with Oxford INCA EDS, ECNs 2191213/2191214

RELATED DOCUMENTATION: KSC-MSL-2008-0345
KSC-MSL-2008-0379
KSC-MSL-2008-0382
KSC-MSL-2008-0410

CONTRIBUTOR: H. Kim/NE-L2-C

PRIMARY INVESTIGATOR: ______________________________
Philip M. Howard/NE-L2-C

NASA
Engineering Directorate
Materials Science Division
Kennedy Space Center, Florida

September 5, 2008

KSC-MSL-2008-0410

SUBJECT: NVR Analysis from MMH Trailers

CUSTOMER: George Krewson/Wiltech/WT

1. FOREWORD

1.1. The non-volatile residue from three monomethyl hydrazine (MMH) sources were submitted for identification. Two samples were submitted from the Arch Chemicals MMH vendor trailers, DAF 17054 and DAF 17072, and the third was a re-sample of the Kennedy Space Center MMH trailer, LT-90.

1.2. The samples were identified as following:

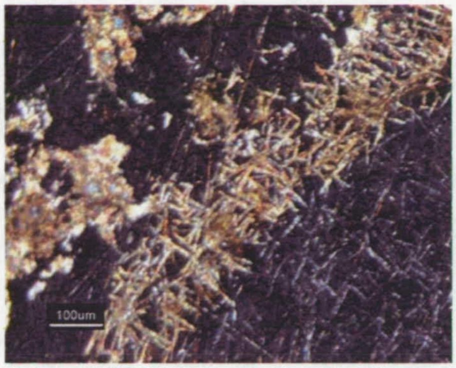
Product Source	Platinum S/N	NVR (mg/40 mls)
DAF 17054	B	1.1
DAF 17054	D	0.9
DAF 17072	2	0.2
DAF 17072	5	0.3
LT-90 KSC	8	0.9
LT-90 KSC	11	0.7

2. PROCEDURES

The NVRs were inspected by optical microscopy (OM) and analyzed by Fourier transform infrared spectroscopy (FTIR).

3. RESULTS AND CONCLUSIONS

3.1. OM inspection of the NVR residues from the DAF 17054 and LT-90 trailers confirmed the presence of anisotropic birefringent crystals as shown in Figures 1 and 2. The NVR from the DAF 17072 trailer appeared black and did not contain crystalline materials.



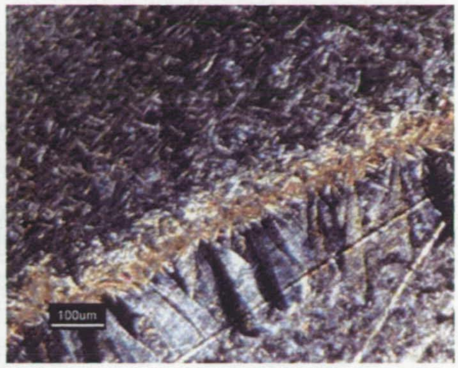


Figure 1. NVR from DAF 17054. Figure 2. NVR from LT-90.

3.2. FTIR confirmed that the molecular composition of the crystals from DAF 17054 and LT-90 were identical as show in Figure 3.

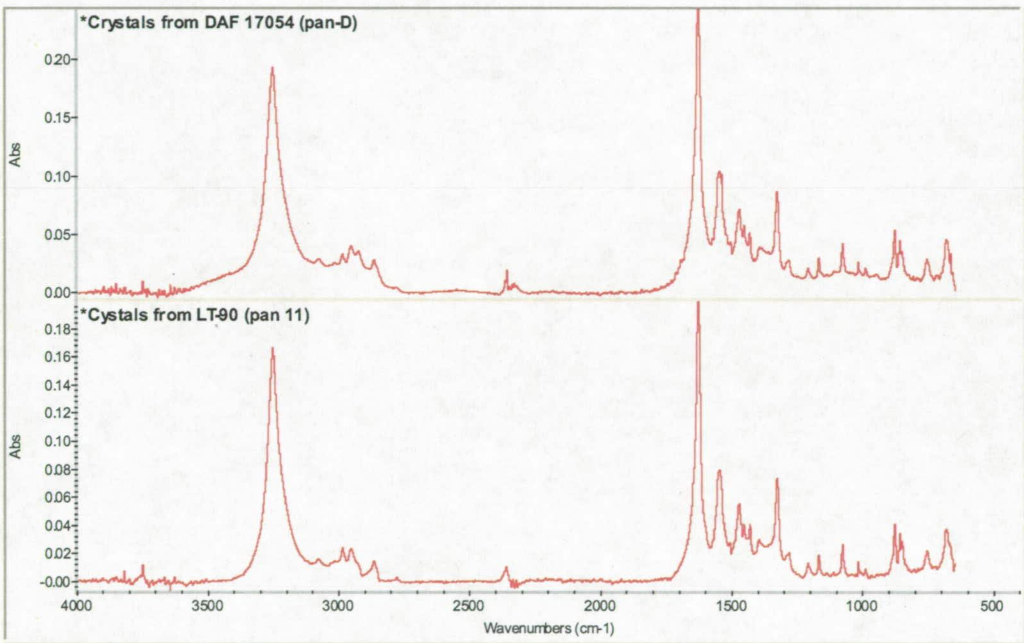
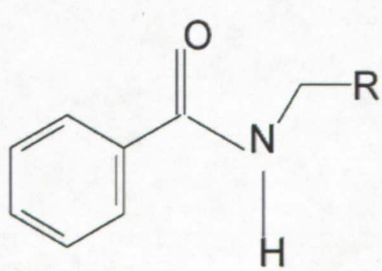


Figure 3. FTIR of the crystals from DAF 17054 and LT-90 NVRs.

3.3. The FTIR spectra identified the crystals as an amide which has a proposed structure of the following.



3.4. The black residue from NVR DAF 17072 was identified as a type of hydrocarbon, totally un-unrelated to the residue collected from the other Arch Chemicals DAF 17054 trailer and the KSC LT-90 trailer.

EQUIPMENT:
Thermo Nicolet 6700 Fourier-Transform Infrared Spectrometer with Continuum microscope, ECNs 2191216/2191218
Nikon Stereo microscope MSZ-1500, ECN 3059651

PRIMARY INVESTIGATOR: ______________________________
Philip M. Howard/NE-L2-C

Characterization of Monomethylhydrazine (MMH) Non-Volatile Residue

Chuck Davis and Philip M. Howard
NASA
Kennedy Space Center, FL

ASTRACT

The Space Shuttle program has a unique propellant purity requirement for determination of non-volatile residue (NVR) in monomethylhydrazine (MMH). This requirement differs from the Military Specification procurement specification by requiring a NVR analysis with a limit of less than or equal to 10 milligrams per liter. In June 2008, a routine MMH replenishment delivery was transferred into a NASA KSC owned tanker for future delivery to the Space Shuttle pad MMH storage tank. Per Shuttle standard operating procedure, the receiving tanker was sampled and analyzed for purity and surprisingly it failed the Shuttle use NVR specification limit. Detailed examination of the NVR revealed that it was fundamentally different than the typical MMH NVR. This paper will examine various aspects of NVR determination in MMH and the analytical characterization processes used to identify the NVR.

INTRODUCTION

Monomethylhydrazine (MMH) is used as a fuel with dinitrogen tetroxide (N_2O_4) oxidizer in the NASA Space Shuttle orbiters' Orbital Maneuvering System (OMS) and Reaction Control Systems (RCS). Shuttle Program document, SE-S-0073, Fluid Procurement and Use Control Specification, for the various fluids and propellant typically mirror the Military procurement "PRF" specifications. However, SE-S-0073, Table 6.3-9 for (MMH) has a unique requirement to measure the non-volatile residue (NVR) in MMH instead of the particulate that is required by the MIL-PRF-27404C, Propellant Monomethylhydrazine. The rationale for this difference is that particulate can be mitigated with filters but NVR cannot. On a re-usable system such as the Shuttle OMS/RCS, NVR accumulation over years of service could cause decreased performance and potentially operational failures.

MMH Analysis	MIL-PRF-27404C Procurement	SE-S-0073, Table 6.3-9 Shuttle Use
Qualitative	Colorless, homogeneous liquid	None
Assay, % by wt.	98.3 (min.)	98.0 (min.)
Density @ 77° F (25° C)	None	Engineering Info Only
Water, % by wt.	1.5 (max.)	2.0 (max.) plus soluble impurities
Nonvolatile Residue, mg/L	None	10 (max.)
Particulate, mg/L	10 (max.)	None

Table 1: Specification Analysis Requirement Comparison.

MMH NVR DETERMINATION METHOD

Given there is no industry standard method for MMH NVR determination, the aerospace propellant community laboratories developed a method based on the NVR determination in hydrazine from MIL-PRF-26536, Propellant Hydrazine. The method summary is:

1. Low temperature (135 °C) evaporation of MMH sample in platinum or Pyrex glass evaporation dishes (~ 3-4 hours) on an explosion-proof hot plate contained inside a fume hood. Sample size

is typically limited to 40 milliliters (mL) with 10 mL of water added to prevent auto ignition. Larger sample volumes are impractical due to the extremely slow evaporation rate needed to prevent auto ignition and the ability to manage a potential fire.

2. The evaporation dish is removed from the hot plate when 1-2 mL remains, then the dish is evaporated to dryness in a 40°C oven for thirty minutes to the final dryness. The dishes are stored inside desiccators to equilibrate to room temperature before weighing. Care must be taken to not "over-bake" the NVR dish.

3. NVR is calculated by weight difference of the dish after ambient temperature equilibration.

4. The total NVR is extrapolated to the mg/L value by multiplying the calculated NVR by 25. A maximum NVR weight of only 0.40 mg (from the 40 mL sample) is needed to fail the limit of 10 mg/L. Much of the reproducibility is due to this large dilution factor and by applied heating rate.

Even though this method is technique-sensitive; reproducible results have been obtained during 25+ years of use by NASA. Historically, MMH NVR is generally in the range of 3-5 mg/L.

A classic reason for either the NVR or Particulate failure is due to contamination with the fluorinated grease used to lubricate the system valves and fittings. This type of failure is typical in satellite propellant fueling operations, where the ratio of the MMH product to internal system volume is low. Grease is liberated from the components when opened to take the analysis sample.

LT-90 MMH NVR FAILURE

Liquid Tanker, LT-90, Figure 1, is one of four KSC-based tankers used for hypergolic fuel storage and delivery at KSC. On June 12, 2008, KSC received the vendor tanker VT-1994, containing approximately 18,000 pounds (~ 2460 gallons) of MMH conforming to MIL-PRF-27404C. The VT-1994 contents were transferred into LT-90 and sampled post-transfer per standard KSC operating procedure. The initial sample failed NVR results at 16 mg/L. The following week, on June 24, the tanker's fill/drain valve was sparged with helium and allowed to re-fill with MMH from within the tanker, and then a second sample was taken. This sample also failed the NVR level test at 18 mg/L, thus confirming the initial failure. Both LT-90 analyses were performed by the Aerospace Fuels Laboratory at Cape Canaveral Air Force Station (CCAFS - Cape Lab).

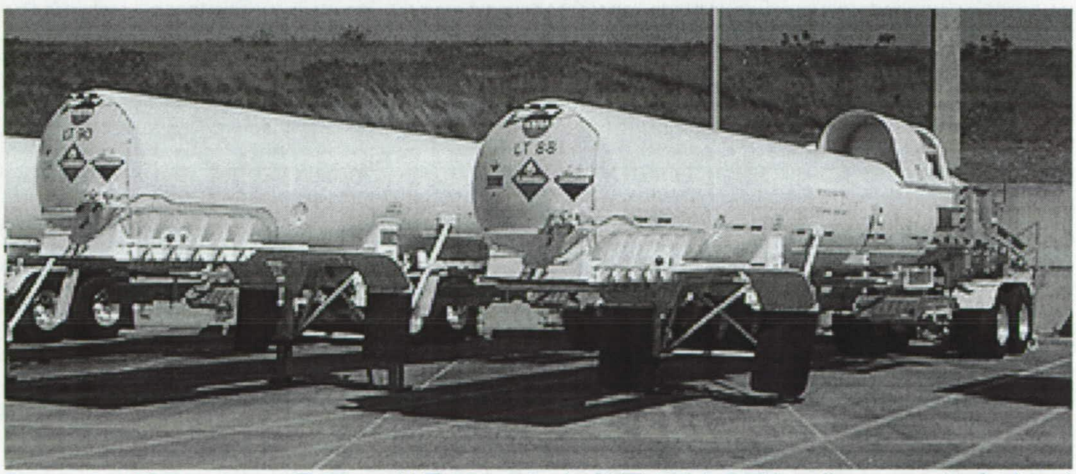


Figure 1: KSC tankers LT-88 and LT-90, typical of VT-1994, 2500-gallon capacity fuel tanker.

The tankers are of identical design and constructed of 304L stainless steel (SS). Each has been in continuous MMH-service with typical MMH sample test results since their last five-year recertification testing process. MMH is manufactured and stored by Arch Chemicals Corp., Lake Charles, LA. Arch uses railcars for storage vessels. The DODX-series railcars are constructed of 304L SS and entered service in 2007. The DAF-series railcars are constructed of 6061 aluminum and have been in service for well over ten years. In accordance with the KSC material selection guide, KTI-5211, these metals are accepted for long-term hydrazine family fuel storage. All of the railcars were filled with MMH in 2007.

In early July 2008, NASA initiated discussions Arch and the Defense Energy Support Center (DESC), Missile Fuels Business Unit, in San Antonio, TX, about the failed MMH sample results. The group decided to:

1. KSC repeat the LT-90 NVR test.
2. KSC trace LT-90's historical NVR levels.
3. DESC/Arch test the retained MMH sample fromVT-1994.
4. DESC/Arch test the MMH inventory contained within five railcars at the Arch facility.

Arch does not have an in-house NVR analysis capability, so the analysis service was sub-contracted to an outside lab. The outside lab could not reproduce the NVR results from VT-1994 that KSC found or produce a typical MMH NVR result on any of the other railcars (all NVRs were reported <0.1 mg/L), well below the historical range of 3-5 mg/L. Therefore, it was decided that samples from all MMH railcars and VT-1994 would be shipped to both KSC/CCAFS laboratories for analysis by the Cape Lab and Wiltech, the NASA-KSC contractor-operated laboratory. The results are presented in Table 2.

Vessel ID	NVR Test Results (mg/L) Cape Lab	Wiltech	Comment
DAF 17054	20	25	VT-1994 source, unique NVR
DAF 17072	10	6	
DAF 17074			Almost Empty not sampled
DODX 7009	12	15	2x runs by WT, typical NVR
DODX 7010	5	3 (28*)	* no source filter, high solids
DODX 7011	5	10	Source of replacement to original LT90 delivery order
VT-1994	N/A	18	Delivered MMH into LT-90, June 12, 2008, from the "retain" sample tested in Dec'08
LT-90	12-18	20.0-28.8	Multiple NVR tests, unique NVR

Table 2, Summary of NVR Results by container and laboratory, August 2008

It was immediately obvious from the Table 2 NVR test results that storage railcar DAF 17054, which was the source of the VT-1994 MMH then transferred into LT-90, all have similar NVR levels at approximately 20 mg/L and higher. Second, both of the NVR's contained unique, needle-like crystals (Figure 2) as compared with the characteristic bubbled resinous MMH NVR (Figure 3). Note; photograph scales are approximately equal.

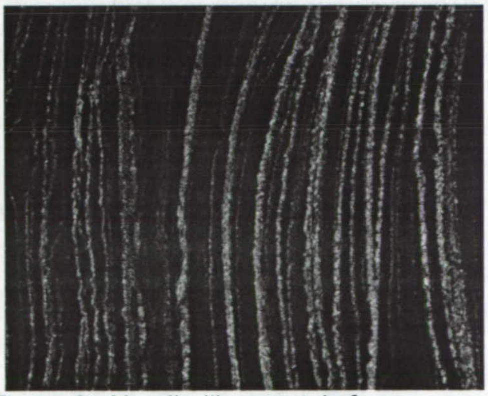


Figure 2. Needle-like crystals from LT-90 and VT1994 MMH NVR.

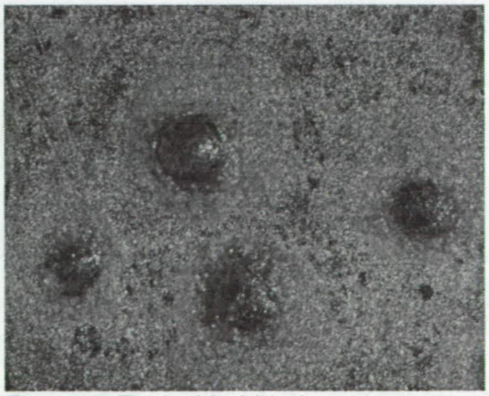


Figure 3. Typical bubbled resinous MMH NVR solids.

On Nov 4, 2008, VT-1994 returned to KSC and the 2400 gallons of high-NVR MMH was transferred from LT-90 back into VT-1994. The product was returned to Arch were the MMH was transferred back into original source railcar DAF 17054. At this point there are two tankers empty but contaminated with unique high-NVR MMH residue, railcar DAF 17054 nearly full of unique NVR MMH. What is this unique NVR, how can the product be recovered, and how can the tankers be returned to routine service?

CHARACTERIZATION OF UNIQUE MMH NVR

It is standard KSC policy to identify all NVR failures to determine if the NVR is either "normal" contamination from the sampling process (typically aerospace-class fluorinated grease), from the system itself, or from an outside source from cross contamination. The KSC Materials Science Division routinely uses a multi-analytical approach in the identification of NVR unknowns using the following techniques:

1. Optical stereo microscopy (OP) using oblique and coaxial illumination to determine the nature and morphology of the contamination.
2. Determination of the solubility of the NVR solids.
3. Fourier transform infrared spectroscopy (FT-IR) to identify the molecular structure.
4. Scanning electron microscopy using energy dispersive spectroscopy (SEM/EDS) support morphology and determine the elemental composition and homogeneity of the residue.

Optical microscopy observation is the first evaluation to determine the morphology and number of phases of an unknown. Whereas oblique illumination provides good topography of solids, it is extremely limited at discerning liquids and almost impossible to see thin clear films. The ability to observe thin films is the strength of coaxial illumination. Coaxial illumination, by the nature of the light being coincident to the optical path, all anisotropic materials (two or more refractive indexes), exhibit interference color of first and higher orders (2^{nd}, 3^{rd}, etc.) in what is call birefringence, as is typical for common clear fluorinated oil shown in Figure 4.

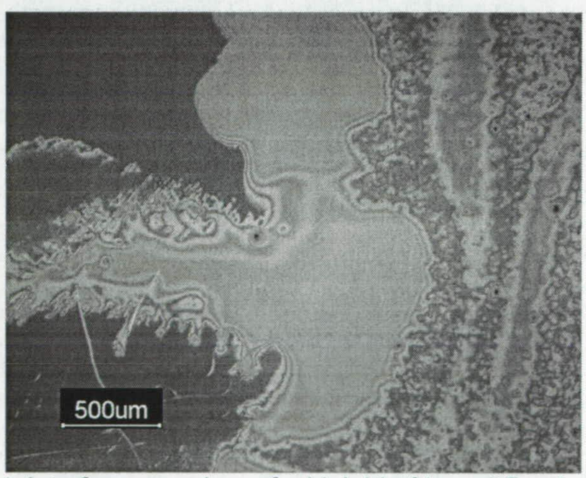


Figure 4. Interference colors of a high birefringent fluorinated oil.

The birefringent colors of the first, second, etc. orders (noted by the red bands) resemble the contour lines in topography. The higher order of colors correlate with the film-thickness of the oil. This optical property is extremely useful in determining sampling for FTIR analysis, where the film thickness is proportional to strongest absorbance. All anisotropic crystals exhibit birefringence colors and therefore provide a quick evaluation of pure crystalline compounds.

In the case of the LT-90 NVR, observation of the NVR dish by optical microscopy using oblique lighting, showed the typical MMH NVR (Figure 3). However the amount of the typical NVR solids did not correlate with the reported weight. Inspection of the NVR dish using coaxial lighting discovered large collections of extremely thin (< 5 microns thick) birefringent crystals covering the outer edges of the NVR dish as seen in Figure 5 and 6. These thin crystals went unnoticed while using oblique lighting. The crystals were observed as evaporation deposit lines created as the last few milliliters of MMH evaporated.

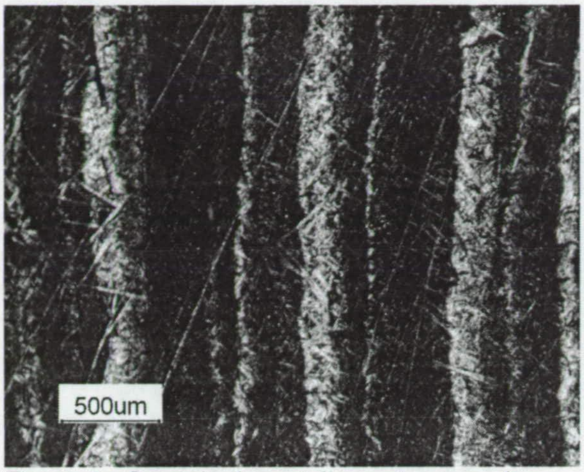


Figure 5. Crystals deposit lines at outer edge of the NVR dish.

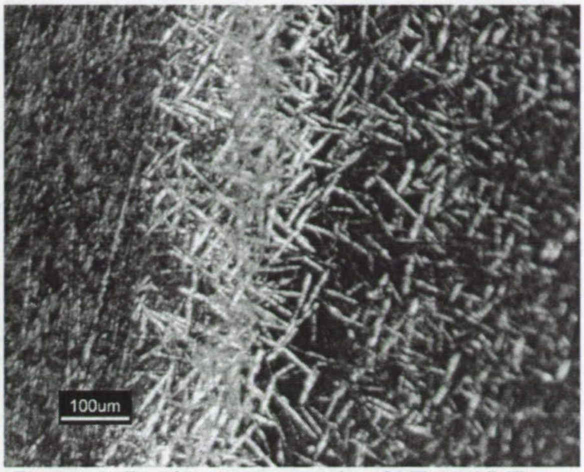


Figure 6. Birefringent crystals of LT-90 NVR.

Solubility is an important physical property of an unknown, not only for determining a suitable cleaning solvent, but also allows transferring the unknown sample for FTIR analysis or other techniques. What must be remembered about solubility is "like dissolves like", so never assume only one solvent will work. A series of solvents, covering the polarity range from non-polar to polar, should be evaluated. MMH would be the best solvent for the MMH NVR but is impractical in the laboratory environment due to its toxicity. Suitable solvents should have low vapor pressure (limited volatility) and high surface tension. These requirements are met by n-decane, amyl acetate, methanol and Castrol® Fluoroclean™ X100 (a fluorinated solvent). Fluoroclean X-100 is the one exception that fails both vapor pressure and surface tension, making it extremely difficult to control. However, Fluoroclean is an efficient solvent for the fluorinated aerospace greases of Braycote and Krytox. In the case of the LT-90 NVR, methanol was found to be the only solvent that dissolved the subject birefringent crystals. The solubility of the crystals in methanol not only allowed the crystals be transferred from the original platinum dishes for FTIR analysis but also allowed the unknown to be re-crystallized into much larger crystals as shown in Figure 7. The extremely high birefringent needles crystals are typical of aromatic organic crystals.

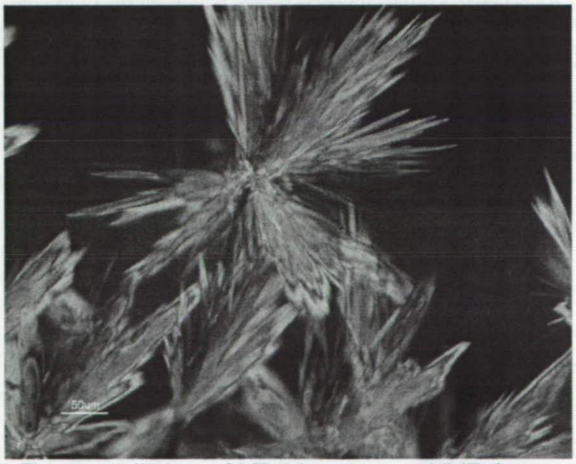


Figure 7. Re-crystallizing of LT-90 unknown NVR from methanol.

Fourier transformed infrared spectroscopy (FTIR) is the primary instrument for molecular structure identification. Typical MMH NVR is characterized as a polymerized by-product of MMH. The

FTIR spectra of the typical MMH NVR is compared to the unknown crystals as shown in Figure 8. The unknown NVR found in tanker LT-90 and the railcar DFA 17054, was characterized as an aromatic amine. The proposed chemical structure shown in Figure 9.

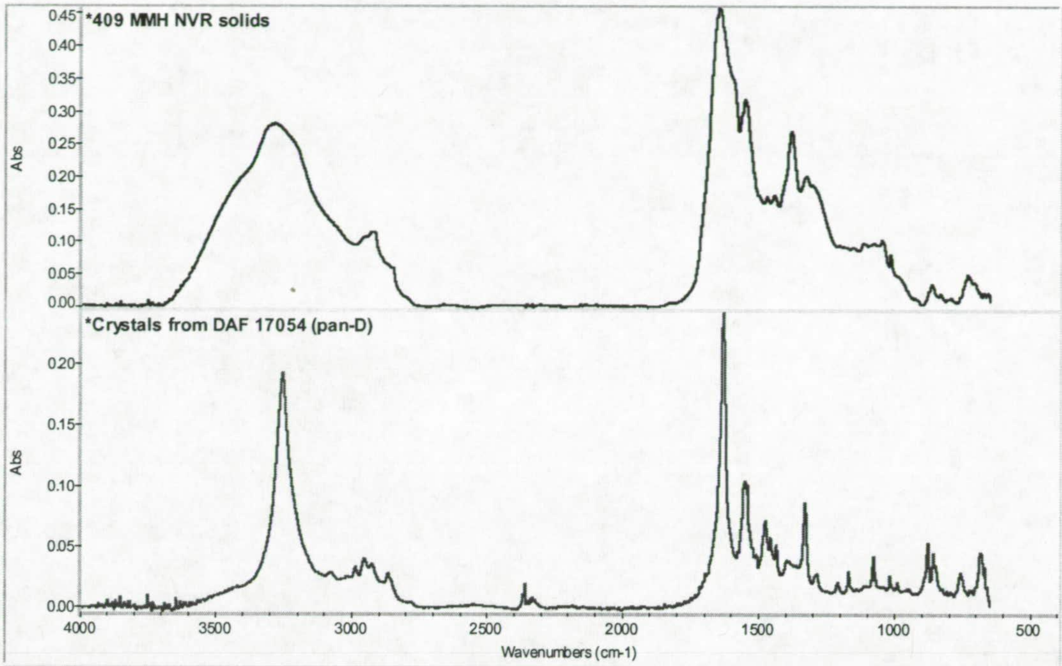


Figure 8. FTIR Spectra of typical MMH polymerized NVR, top (blue), as compared to the crystals found in both LT-90 and DAF 17054, bottom (red).

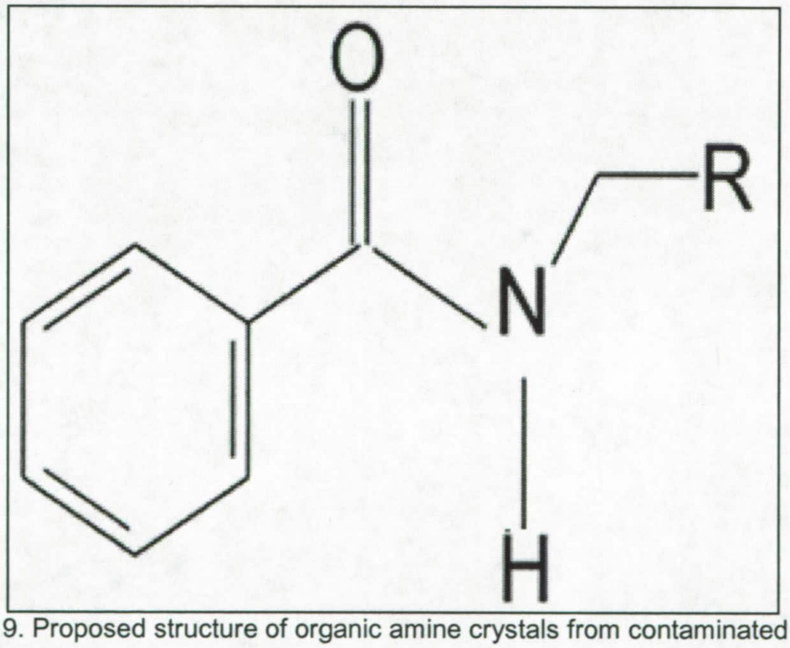


Figure 9. Proposed structure of organic amine crystals from contaminated MMH.

SEM/EDS elemental analysis confirmed the composition of the crystals to be homogeneous material with only carbon, oxygen and nitrogen elements present.

This information has been shared with DESC and Arch for their evaluation of the Lake Charles plant systems and components as to what and where this substance may have originated.

MMH HIGH-NVR RECOVERY OPTIONS

Several options have been considered and some tested to determine the most economical way to return the contaminated containers to specification grade MMH service and how to mitigate the NVR.

1. High-NVR MMH: Dilution of affected MMH with "good" MMH. A calculated 70%-typical-to-30%-unique NVR MMH blend should lower the final NVR below the 10 mg/L limit. This will not remove the "the out of family" aromatic amine contaminate but will minimize any effect of contaminate on performance by diluting it to below the allowable limit. Lab experiments with this "hybrid" NVR produced of results of 9.8 mg/L and 12.0 mg/L. The dilution appeared to only reduce the size and alter to more plate-like morphology of the aromatic amine crystals. Given enough dilution, the original contamination will be below the limit of detection. However, we do not currently plan to determine the limit of detection. Other potential MMH recovery methods could entail re-distillation of the DAF 17054 MMH at Arch's facility. However, further determinations are beyond the scope of this paper.

2. Tankers: There is concern of a residue film remaining on the interior surface of the tankers when the high-NVR MMH was drained from both LT-90 and VT-1994. During various NVR test runs, it was observed that the unique NVR is readily soluble in MMH. Therefore, LT-90 and VT-1994 could be "flushed" with typical MMH to remove and dilute any remaining NRV. This theory was tested on Dec 21, 2008, by flushing LT-90 with about 50 pounds (~ 7 gallons) of typical ~ 3 mg/L NVR MMH the full-length of the tanker, washing over the area where the last bit of fuel would have flowed when LT-90 was drained into VT-1994 the previous month. This flush was captured and measured to have 10.3 mg/L NVR and no evidence of the birefringent crystals. Thus, the NVR increased about 3-fold but lost its unique birefringence. Alternatively, lab testing demonstrated the unique NVR readily dissolved in methanol, therefore, 2-propanol (isopropyl alcohol) should provide similar solvency. 2-propanol is a standard solvent used at KSC for cleaning hypergolic propellant fuel systems and components. This tanker recovery option is currently under review by NASA

3. Future care must be taken with tracking the contaminated MMH now held in railcar DAF 17054 so it will not unknowingly re-enter the general-use inventory. Its fate is currently undetermined.

SUMMARY

Routine quality control testing is vanguard towards ensuring mission success and safety. Specification testing of the LT-90 MMH flagged a failed product before it could enter Shuttle ground support equipment. More specifically, the NVR test discovered an out-of-family contaminant thereby preventing the fuel's potential use in flight hardware and avoiding costs associated with removal of an out-of-specification product and resultant contamination.

However, basic quality test methods only produce a single data point of pass-fail criteria. Complete characterization of the unknown is required to trace back to the potential contamination source. Numerous analytical techniques beyond the basic Mil-spec test methods should be routinely applied to characterize typical NVRs. It is standard KSC policy to identify all NVR failures to determine if the NVR is "normal" contamination is due to sampling, from the system itself, or from an outside source from cross contamination. KSC's analytical testing traced the unique MMH NVR contamination back to the source. Furthermore, solubility testing revealed a practical route to decontaminate tankers LT-90 and VT-1994 so they may be economically returned to service.

Further evaluation of the vendor system component materials and processes is recommended to identify and prevent future contamination recurrence.

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CL = Cape Lab
WT = Wiltech
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