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Best Practices for the Elemental Profiling of High-Purity Hydrazine

Trace contaminants in high-purity hydrazine (HPH) propellant impact a wide variety of commercial, Department of Defense (DoD), and NASA missions. Depending on thruster design, elemental contaminants must be kept at extremely low levels and are verified as such by routine analysis. A number of these contaminants have recently undergone an assessment to shed light on their quantities present following changes in the HPH supply chain. A round robin analysis utilizing four separate laboratories resulted in unacceptably high variability in the quantification of these contaminants. The principal objective of this technical bulletin is to signal the availability of a new analysis methodology which yields accurate and repeatable quantification by providing best practices for both quantitation methodology and strategies for avoiding sample contamination during analysis.

Background:

Hypergolic propellants (e.g., hydrazine (N2H4)) are used to power monopropellant and bipropellant propulsion systems. Investigations to better understand HPH manufacturing processes and the associated introduction of contaminants have been a priority for the HPH user community after the chemical reaction scheme employed to produce a precursor to HPH, which is used as the feedstock by the Defense Logistics Agency's sole source HPH provider, changed in mid-2018.

Particular concern arose regarding the possible introduction of organic species (e.g., carbonaceous compounds) and elemental content (e.g., cadmium (Cd)) to the final HPH product, as this carries an increased risk of performance degradation and/or flow-path blockage to thruster system valves, softgoods and catalyst beds.

Analysis was completed to identify extraneous unknown carbonaceous materials present in current HPH in addition to comprehensive elemental profiling by four laboratories to develop a full elemental profile of the commodity in relation to heritage HPH stocks.

The elemental laboratory data revealed varying and/or high levels of multiple elements outside of nominal laboratory-to-laboratory variation, and sample-to-sample variation was independently confirmed. The NESC concluded that, while there was variation in the elemental content of the samples, there was also an apparent inconsistent handling of samples within each of the four laboratories that, in some cases, led to widely varying elemental assay results.

Causes of Analysis Variability:

There was an unexpectedly wide variation in elemental assay results from the analysis of single batch-sourced samples from four laboratories. Further analysis revealed that the provided samples themselves exhibited variation, possibly as a result of pre-laboratory handling. However, in some cases, large discrepancies were determined to have been caused by differences in analytical procedures and methods at the laboratories themselves. Refinement of analytical methodology for HPH, other hydrazine derivative sample handling and processing, as well as the instrumental analysis methodology for extended elemental content, are essential to gaining accurate and equivalent results from multiple laboratories performing this type of analysis.

Best Practices for HPH Elemental Analysis:

The detailed best practice recommendations for conducting the elemental analysis process are described in [1]. Briefly:

A. Glassware usage should be minimized in all steps of the analytical process to minimize sample contamination.

B. Blanks for water and acid stock solutions used in sample preparation should be prepared alongside, and analyzed with, each batch of samples analyzed to ensure any contamination is accounted for from the process.

C. When using platinum evaporation dishes, adequate cleaning between samples should be ensured.

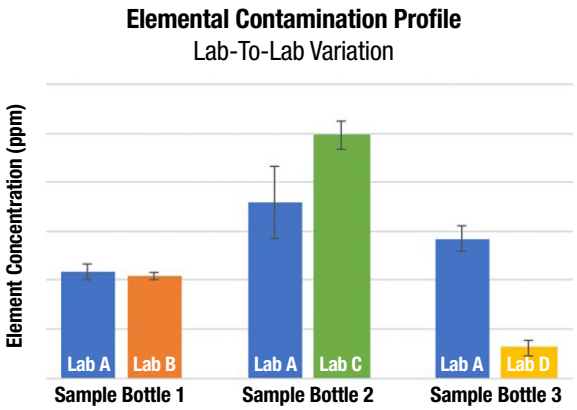
D. Method detection limits and reporting limits should be established for all elements in analysis for proper reporting of trace elemental levels.

E. For ICP-OES (Inductively Coupled Plasma - Optical Emission Spectrometry) or ICP-MS (Inductively Coupled Plasma - Mass Spectrometry) analysis, survey the elements that are to be analyzed and determine what possible interferences may exist for those elements, which should be addressed prior to analysis.

F. Samples should be analyzed in duplicate or triplicate, when possible.

References:

1. NASA Lesson Learned Information System entry No. 29801. Available from https://llis.nasa.gov/lesson/29801



Lab-to-lab variation in analysis of exemplar elemental content across multiple sample bottles of single-source commodity.

For information, contact Donald Parker at donald.s.parker@nasa.gov and Janelle Coutts at janelle.coutts@nasa.gov.

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