ASTRO PAK®

CASE STUDY

BLACK RESIDUE ON STAINLESS STEEL SURFACES

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BACKGROUND

Have you ever wiped a vessel and found that the wipe had excessive black debris? This is common on mechanically polished vessels, especially when they have not been properly prepped and cleaned. This discoloration on the wipe is classified as polishing debris consisting of ground-in stainless steel particles and abrasive residue. The residue is a thin film not generally visible on the surface and its removal can be problematic since it consists of very fine particles (metallic debris from sanding/polishing, abrasives, and other compounds or polymers) that are well attached to the surface. Cleanliness evaluation of new equipment including vessels, process equipment and components is recommended prior to FAT testing or installation. Additionally, the condition of surfaces in new equipment and systems for pharmaceutical processing meeting the requirements of ASME - BPE should be documented.

INSPECTION

Inspection and sampling of mechanical polished surfaces is performed with the use of a clean room wipe and alcohol. First, scrub the surface with light to moderate pressure using an alcohol wipe over an approximately one square foot area. Next, inspect the wipe visually and/or under magnification to initially evaluate the severity and physical characteristics of the residue. The residue can appear white to light gray in color, or more commonly, dark gray to black. If excessive residue is encountered, the wipe can be evaluated with advanced analytical techniques to identify the metals or organic compounds present. ICP-MS (Inductively Coupled Plasma – Mass Spectrometry) and EDX (Energy Dispersive X-ray) are used to identify the inorganic elements (metal oxides) present on the wipe from the surface residue. FTIR (Fourier Transfer InfraRed spectroscopy) is used to identify any organic compounds present in the residue.

TESTING RESULTS

The testing results of vessels over the past 5 years are presented below to describe the typical components found in the residue. These tests were often the result of the appearance of rouge on the surface of the vessel or the appearance of a wipe from the equipment surface that was considered excessively colored. The analyses of the wipes are summarized in Attachment "A", from highest to lowest average concentrations. The risk of this film on the surface can be categorized as inhibiting the cleaning and passivation treatment, potentially available for release into the process or product fluids, or a source of rouge and corrosion products.

The elemental analyses lead to the following conclusions. The most prevalent elements (iron and chromium) are from the stainless steel particles present on the surface from polishing (sanding operations). The second most prevalent group (calcium, sodium, potassium and magnesium) of elements are from process fluids or water. Silica (silicates) is found at a similar level of concentration and is one of the more common abrasive media components used in polishing operations. Other abrasive elements including aluminum are also seen at lower concentrations. The last group of elements includes manganese (often present on the surface of stainless steel) and metals found in low concentrations in stainless steel (such as copper, molybdenum, zinc and titanium). Phosphorus is sometimes found on the surface (generally from cleaners or from the stainless steel). Carbon is not quantitatively evaluated since the wiping material is composed of mostly carbon. Organic analyses of the surface residues show a low level of oils and greases, waxes, esters, phthalates, and assorted polymers. These are compounds that can be found in polishing processes and some from the actual wipe.

In all the testing, a blank (clean) part of the wipe is processed in addition to the colored or residue laden section for comparison. The variance in the blank analyses and the variability in the residue analyses due to the low (ppm) levels of the contaminants only yield qualitative results.

REMEDIATION TECHNIQUES

Removal of these films have been attempted in a number of ways, including hand wiping with an alkaline cleaner, electrochemical cleaning (flash electropolishing) and high pressure washing with particulate removal chemistries. Results vary based upon the condition of the surface and the technique employed. Electropolishing of the surface has been shown to effectively remove both the surface contamination and the damage layer often associated with mechanically polished surfaces. Hand wiping removes the majority of the residue, but can require multiple treatment efforts with hot water washing cycles between wiping efforts. Pressure washing or the use of particulate removal chemistries is generally only marginally successful, unless combined with additional mechanical cleaning efforts.

SUMMARY

Analysis of wiping of product contact surfaces is a method to determine characteristics of potential residue or contamination. The residue from wipe samples show that it is mostly stainless steel particles and oxide compounds generated from the mechanical polishing with lower levels of abrasive compounds and water or process fluid residues. Each project presents a different level of contaminants based on existing conditions and polishing or cleaning techniques employed on the equipment surfaces. Sampling and testing of the residue will indicate the source and guide you in the proper treatment technique to be used.

TABLE 1 - ICP-MS, EDX, FTIR

RESIDUE FINDINGS

ELEMENT	AVERAGE CONCENTRATION	SOURCE
Iron	Very High	Stainless steel particles / rouge
Chromium	High	Stainless steel particles
Calcium	Moderate	Water or process fluids
Sodium	Moderate	Water or process fluids
Silica	Moderate	Abrasive media
Potassium	Low	Water or process fluids
Magnesium	Low	Water or process fluids
Nickel	Very Low	Stainless steel particles
Aluminum	Very Low	Abrasive media or SS surface
Manganese	Very Low	Stainless steel surface
Zinc / Phosphorus / Copper / Molybdenum /Titanium	Trace	Water / Abrasive / Stainless steel

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As Astro Pak's Senior Vice President of Technology,Mr. Roll serves as the primary senior technical advisor for corrosion, surface chemistry and stainless steel passivation. With over 30 years of experience in chemical processing, Daryl has been published in MICRO, UltraPure Water Journal and Chemical Engineering for his papers on passivation and rouge control. He is a participant on the ASME BPE Subcommittees for Surface Finish and Materials of Construction requirements and a leading contributor for the Rouge and Passivation Task Groups. Mr. Roll holds a B.A. in Chemistry and Earth Science from the California State University of Fullerton and a Professional Engineer's license from the State of California.