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Valve Integrated Pressure Regulator Examination (DOT DUNS #079807449)

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Table of Contents

Valve Integrated Pressure Regulator Examination (DOT DUNS #079807449)	1
Table of Contents	2
1 Introduction and Incident Background	3
2 Incident Description	3
3 Incident Hardware Information and Incident Description	4
3.1 VIPR Identification:	6
3.2 WHA Inspection Chronology	7
4 January 11, 2016 Joint Inspection	7
4.1 Internal VIPR Component Inspection	11
4.2 Chemical Analysis by FTIR	13
4.3 Scanning Electron Microscopy (SEM) with Energy Dispersive Spectroscopy (EDS)	18
4.4 Documentation and Discussion of Internal and External Combustion Patterns	22
5 Opinions and Conclusions	23
Appendix A – Joint Inspection Report	A1
Appendix B – Praxair Grab ‘n Go Literature and Recall Details	B2
Appendix C - FTIR Sampling Description and Log	C3
DAppendix D – SEM/EDS Analysis Summary	D7

1 Introduction and Incident Background

WHA International Inc., (WHA) was requested by the Pipeline and Hazardous Materials Safety Administration (PHMSA) to document and evaluate the valve portion of a ruptured DOT 3AL cylinder that had been involved in a fire during high-pressure oxygen service, and to determine the following:

- Materials of construction of the valve, including polymeric/elastomeric components;
- The degree of exposure of the valve to thermal energy;
- Evidence of a fire on the valve body that may have resulted from the combustion of organic components/contamination in a pure oxygen atmosphere.

This report does not include evaluation of fire patterns on the incident cylinder. Analysis of only the Valve Integrated Pressure Regulator (VIPR) was authorized. In August 2016, the incident cylinder was transferred to WHA in preparation for further joint examination of this evidence; however, authorization for detailed examination and testing of the cylinder was not received by WHA; and, by early 2017 WHA was requested to finalize this report (pertaining to the VIPR inspection only) while disposition of the cylinder was still under consideration.

As requested by PHMSA, the WHA activities included a forensic analysis of the valve components, utilizing, as needed, Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) with Energy Dispersive Spectroscopy (EDS), to determine if the use of inappropriate materials on the valve may have resulted in combustion inside the pressurized oxygen service DOT 3AL cylinder.

The WHA investigation included inspection and documentation of the fire-damaged VIPR and included a joint inspection of the evidence at the WHA Laboratory in Las Cruces, NM, with interested parties in attendance. The joint inspection included disassembly of the VIPR and selection of samples for chemical and elemental analysis.

Appendix A, herein, provides a hyperlinked report documenting the activities undertaken during and after the joint inspection. The hyperlinks in this appendix provide access to all the WHA data, photographs, video, x-ray radiography, chemical, and metallurgical analysis obtained by WHA.

The body of this report provides a summary of the observations and opinions developed by WHA pertaining to the objectives stated above and is based on the investigation completed to date and the materials provided for review. If additional analysis or evidence is performed, then WHA reserves the right to amend any opinions and conclusions stated herein accordingly.

2 Incident Description

The incident VIPR, as shown in **Figure 1**, was identified as a Praxair Grab 'n Go Vantage Advanced Respiratory System valve integrated regulator. Operating instructions for the Grab 'n



Go are provided in **Appendix B** to this report. This figure also shows that the VIPR received by WHA for analysis had been marked with a “T” decal affixed to the shroud identifying it as having been modified according to a voluntary recall by Praxair in 2012, which replaced the O-ring seal between the VIPR and the cylinder as part of routine servicing and filling cycles.

Several press releases regarding the voluntary recall are also provided in **Appendix B**. An O-ring also received with the evidence (**Figure 2**) is consistent with a PTFE-based O-ring typically used successfully in high-pressure oxygen service. Chemical analysis of this O-ring will be discussed later in this report.

It was noteworthy that no description of the incident summarizing the manner in which the equipment was being used or the circumstance of the incident was provided to WHA. This information is preferred by WHA when evaluating the causative factors associated with an oxygen-related fire. WHA has significant expertise and background in investigating high-pressure oxygen fires, mapping the melt-flow propagation of an oxygen-fire kindling chain, and conducting flammability and compatibility tests in high-pressure oxygen.

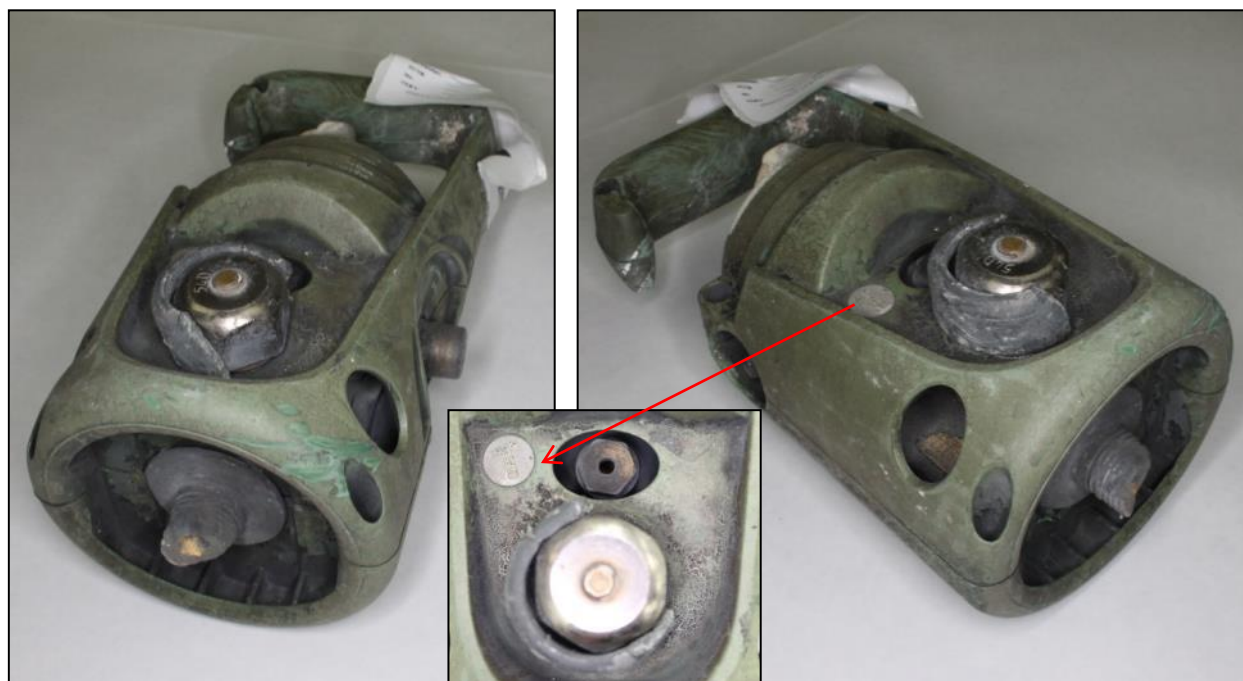


Figure 1 – Incident VIPR and Shroud (As-Received)

3 Incident Hardware Information and Incident Description

WHA initially received and inspected the evidence on August 27, 2015 and documented the as-received condition of the incident VIPR, as shown in **Figure 1** and **Figure 2**. These figures depict the condition of the VIPR and shroud, an O-ring consistent with the VIPR-to-cylinder seal and a metallic segment consistent with a portion of the fragmented cylinder. The VIPR and O-ring shown in these photographs was the subject of the WHA investigation. The cylinder

fragment was not part of the WHA scope and the remainder of the incident cylinder was not provided to WHA with authorization for examination.

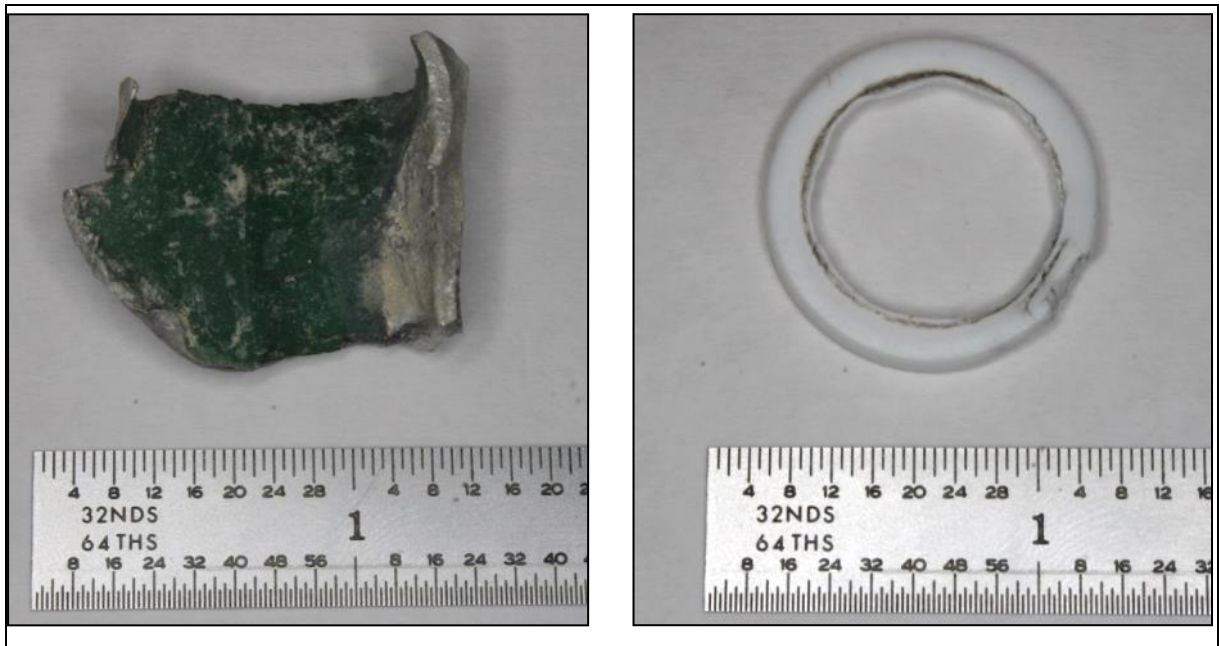


Figure 2 – Metal Fragment (Likely from Cylinder) and O-Ring (Received with Evidence)



Figure 3 – Incident VIPR (Shroud Removed)

Figure 3 and **Figure 4** depict the condition of the VIPR in its as-received condition after removal of the external plastic shroud. The VIPR was heavily covered with soot and combustion products but was generally intact without any inside-to-outside combustion evident in the external examination¹. The predominant area of melt-flow and thermal involvement was observed on the threads of the VIPR, associated with the interface to the cylinder. This region of the VIPR exhibited very heavy involvement and had burned/melted where the original threaded interface to the cylinder had been. The VIPR included the following markings:

3.1 VIPR Identification:

- Praxair Grab 'n Go Vantage VIPR (based on flow range, 0.5 to 25 lpm)
- Post-recall modified based on "T" decal affixed to the plastic shroud
- Valve Markings
 - Serial Number M002270-1003572
 - "CGA 540" marking on fill valve port and cap
 - Burst disc cap marked "32" and "3360" (consistent with 3360 psig nominal rupture pressure)
 - Body marking above threads/flange: ¾-16, UNF-2A

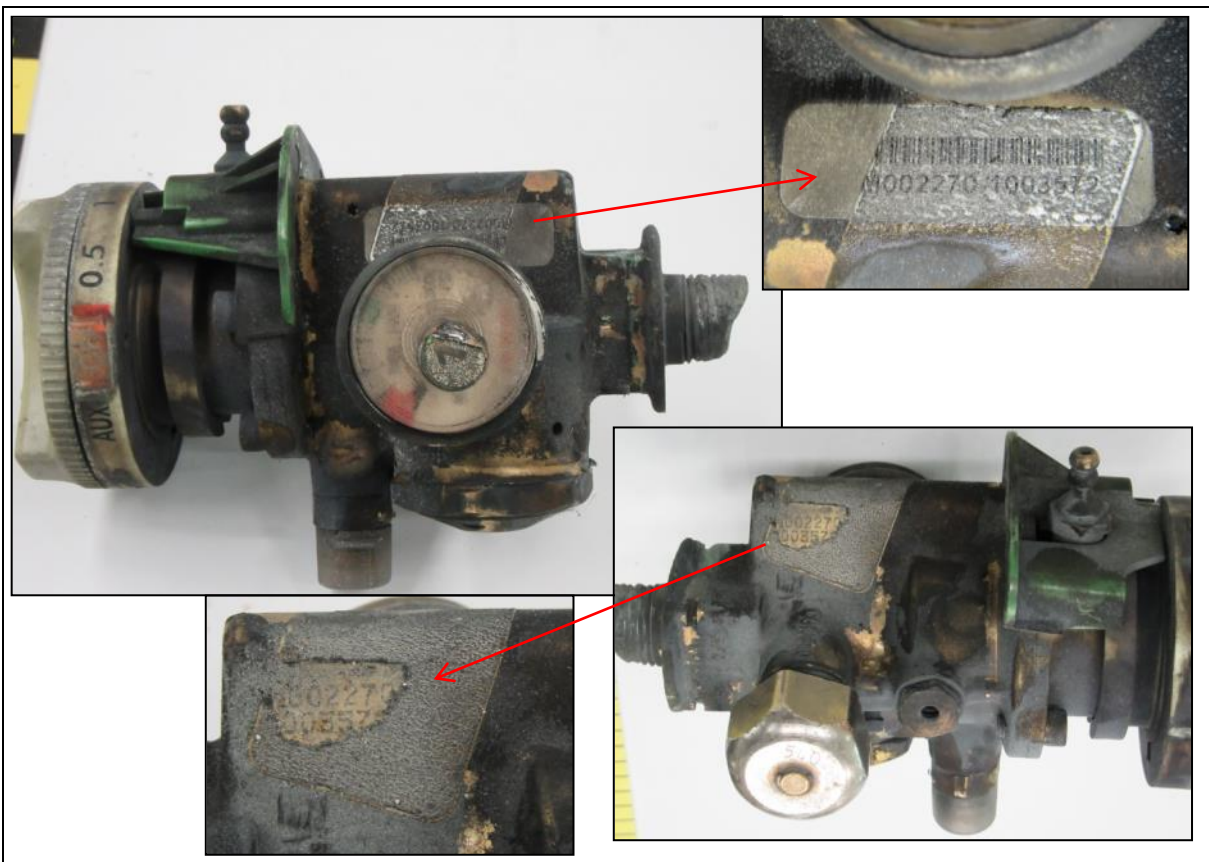


Figure 4 – Grab 'n Go Markings

¹WHA has investigated many "oxygen regulator burnouts" where inside-to-outside combustion is indicated by propagation of internal regulator components burning and breaching through the body of the regulator. This was not observed in the evidence associated with this event.

3.2 WHA Inspection Chronology

WHA initially received the evidence from PHMSA on August 27, 2015 but at that time was still in the process of clarifying the scope of work and funding with the PHMSA contracting officers. WHA then performed an initial visual inspection with Mr. Mark Toughiry of PHMSA on September 25, 2015 at which time the shroud was removed from the VIPR for documentation. No further disassembly was performed at that time. This allowed WHA to perform non-destructive examinations including x-ray radiography, black-light inspections, and microscope examinations in preparation for the joint disassembly which was originally scheduled for December 2015 but was later re-scheduled for January 11, 2016. After the January 11, 2016 joint inspection, WHA completed the chemical analysis and elemental analysis for presentation in this report. The deadline established by PHMSA for completion of the contract work reported herein was initially March 31, 2016. The WHA report presented by that date was not finalized until disposition of the incident cylinder could be determined.

4 January 11, 2016 Joint Inspection

WHA hosted a joint inspection with the interested parties on January 11, 2016. During this inspection, the non-destructive examinations that had previously been performed by WHA were shown to all parties present including visual documentation of the fire-damage, x-ray radiography, boroscope inspection videos, and microscope documentation of the VIPR, especially focused on the threaded area of the VIPR.

By the direction of PHMSA, the parties present were allowed to photograph and document the condition of the evidence but not to interfere with the activities undertaken by WHA during the inspection. To aid PHMSA and the interested parties a complete data package was developed by WHA at that time to summarize the data and documentation obtained and is provided in **Appendix A**, along with hyperlinks to the data obtained.

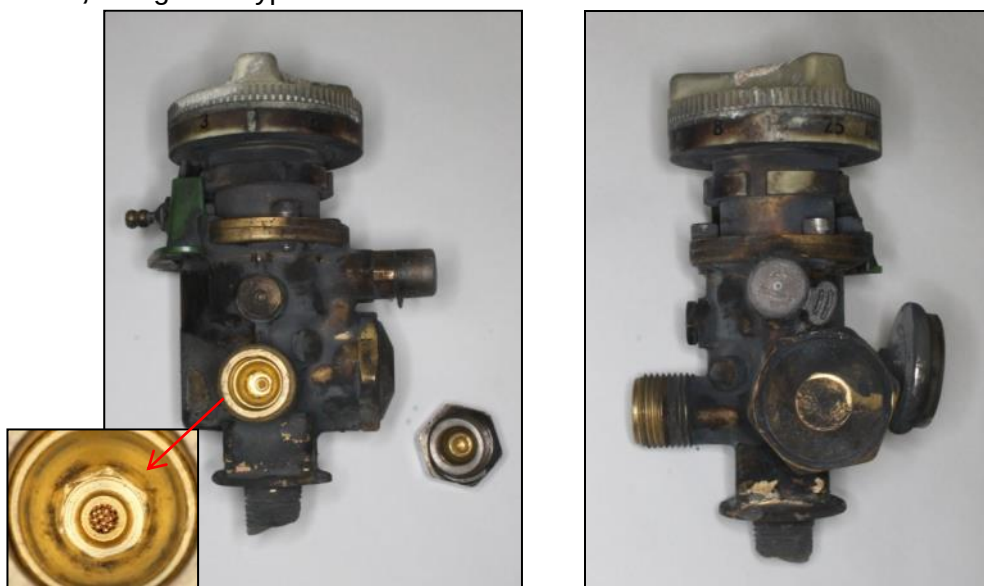


Figure 5 – Views of VIPR (Rotated Clockwise)



Figure 6 – Views of VIPR (Rotated Clockwise) and Flange Close-up

Figure 5 and **Figure 6** provide various views of the incident VIPR from four different sides. Figure 5a (top-left) shows an external view of the VIPR with the fill-port cap removed to expose the fill port assembly and filter. This port, like other ports, was visually undamaged, relatively clean, and intact. The fill-port filter was visually undamaged. Other views in this figure also show the external condition of the VIPR from various sides. The VIPR externally was covered with combustion residue, largely consistent with greyish aluminum-oxide (visually) but did not exhibit inside-to-outside evidence of burn-through. It was noteworthy, however, that the pressure gauge was bent consistent with an impact loading. It was also noteworthy that the flange on the VIPR, at the cylinder-to-VIPR interface, was bent on one side, consistent with an impact or bending moment applied to the body of the VIPR (**Figure 6**). The bend and deformation of the flange was approximately opposite the side of the VIPR threads that exhibited the heaviest melt/consumption of brass.

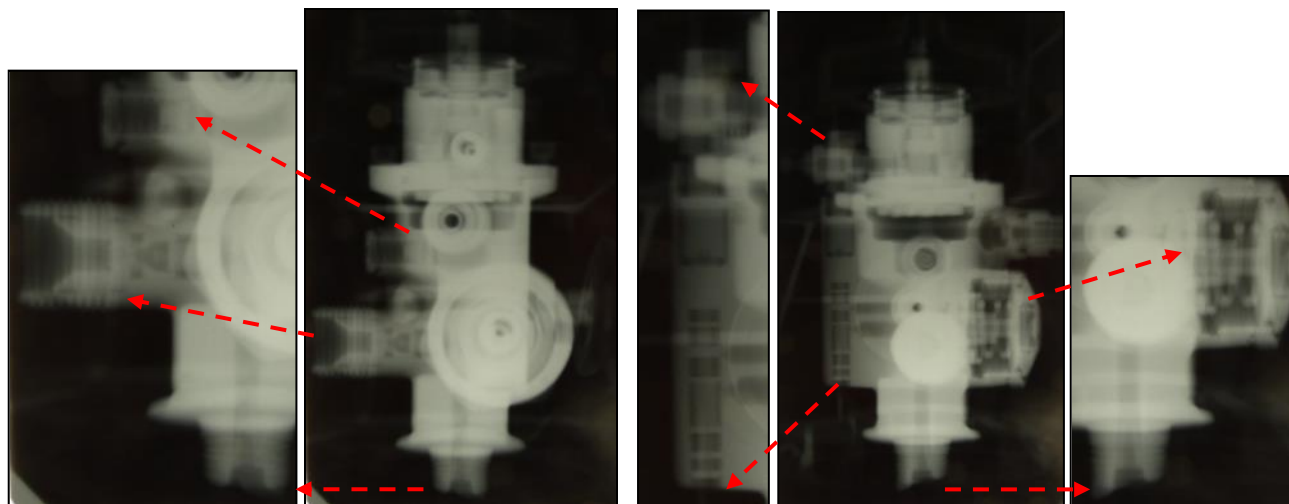


Figure 7 – X-ray Radiographic Views of VIPR (Noting Various Features of Design)

Figure 7 depicts several x-ray radiographic images of the VIPR showing views of the internal components. The x-ray images revealed that the internal components were intact and did not exhibit internal burning or combustion, to the extent that could be discriminated from the images. The fill valve, regulator, flow valve, relief valve, burst disc, and flow head could be observed and did not exhibit indications of damage or internal combustion from the images obtained. The melt/consumption patterns on the cylinder-to-VIPR threads were clear and indicated involvement only of the threads externally without internal propagation being evident in these images. The cylinder-to-VIPR filter was also evident in the x-ray images and appeared visually intact having the expected conical geometry. Further views of this filter using a boroscope confirmed the intact nature of this filter and are hyperlinked in **Appendix A**.

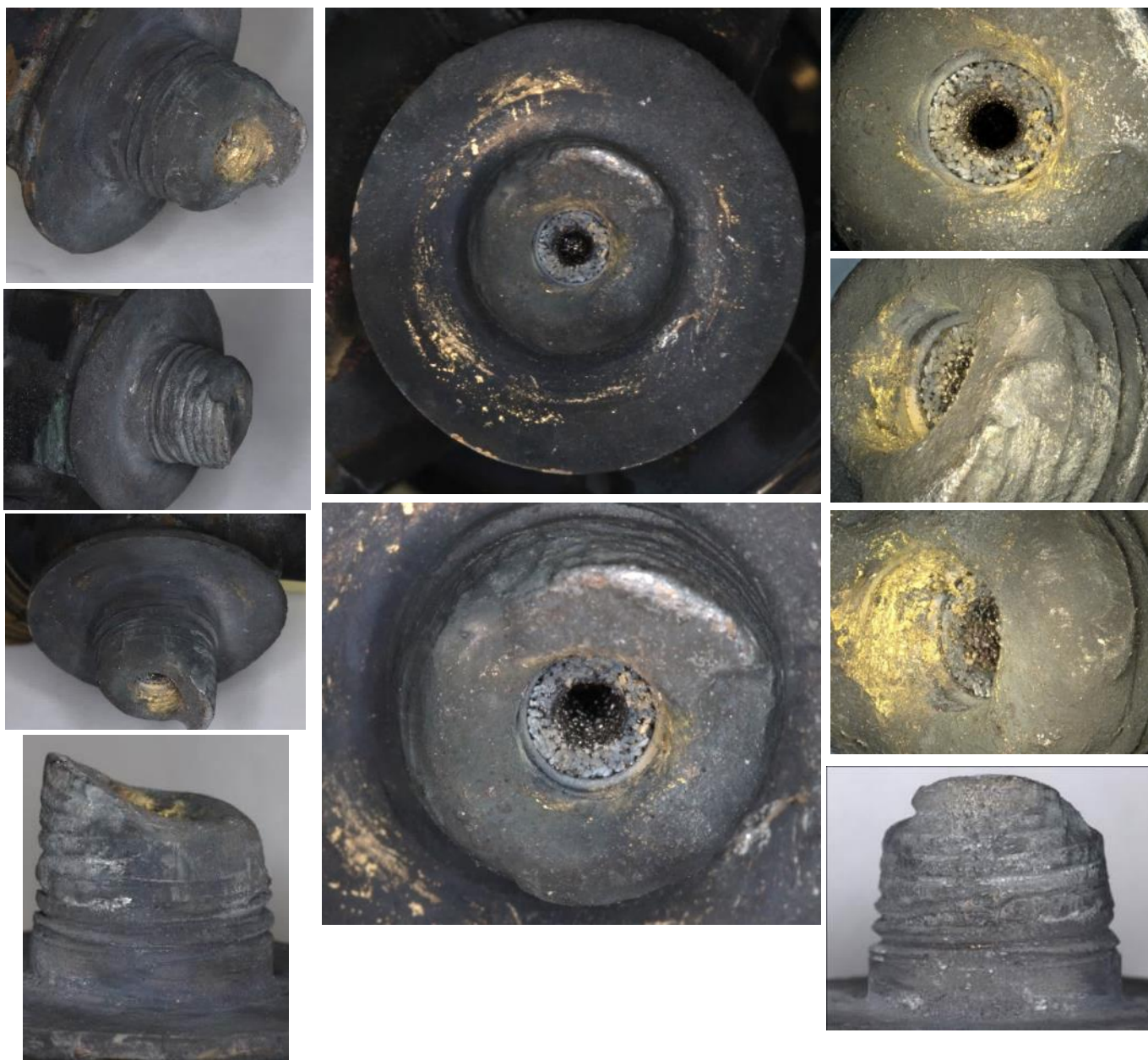


Figure 8 – Magnified Images of the VIPR Thread Damage

Figure 8 depicts magnified images of the flange of the VIPR and the fire-damaged threads. **Figure 9** provides more detailed images of the VIPR thread damage. These images show that the surfaces proximate to the VIPR threads were covered with combustion residue and were primarily comprised of aluminum combustion products (see also EDS summary in **Appendix D**, discussed later). The threads also exhibited melting and/or consumption with a geometric preference for material loss on one side of the threads. The filter was covered with combustion residue but was intact and did not exhibit evidence of compromise or burn-through. The filter's threaded retainer element was missing.

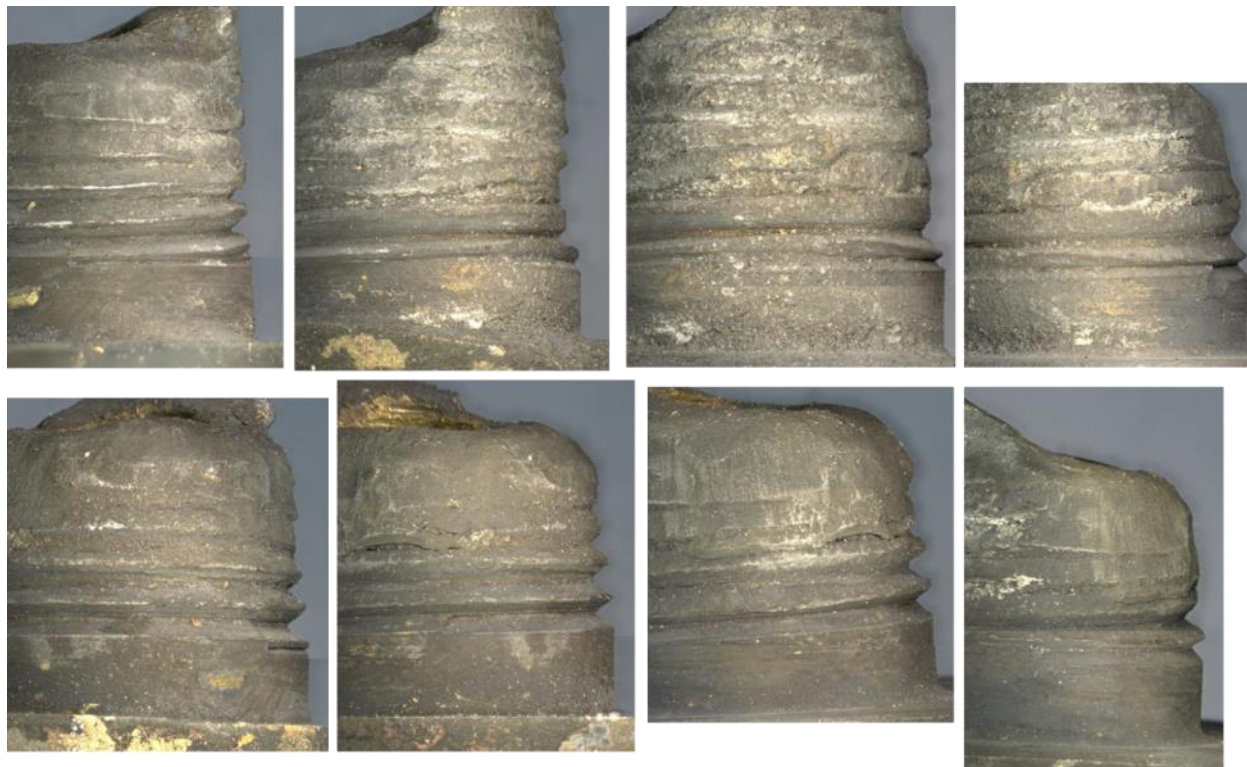


Figure 9 – Magnified Images of the VIPR Threads (Clockwise Rotation)

During the January 11, 2016 inspection, the Grab 'n Go VIPR was disassembled under WHA direction to evaluate whether internal combustion could be identified and to obtain samples for chemical and elemental analysis. Each component was removed and internal ports of the VIPR were documented visually, microscopically, photographically and with the aid of a boroscope where indicated. Chemical samples were also taken from each part or subassembly and will be discussed later in this report².

²The WHA data package is incorporated as **Appendix A**. If the hyperlinks in that report are followed, the full documentation developed by WHA during the investigation can be accessed, including videographic and photographic documentation of the disassembly and condition of the internal components of the VIPR.

4.1 Internal VIPR Component Inspection

During the joint inspection, WHA disassembled and inspected components within the VIPR body, looking for evidence of internal combustion or propagation patterns involving the internal components. Summary documentation of the condition of these component assemblies is shown in **Figure 10** and **Figure 11**. **Figure 11** provides a general sequence approximating the removal of components from the VIPR body. The following components were inspected:

- Burst Disk (rupture disc and gasket intact)
- Fill Valve Assembly (inlet filter, poppet, spring, and O-rings intact)
- Pressure Gauge and “Snubber” Orifice (components intact and orifice visibly clean)
- Regulator Assembly (PTFE Seat and O-rings intact; evidence that external combustion products had projected through the regulator atmospheric vent port; but, no evidenced of internal combustion)
- Relief Valve Assembly (valve components, spring, O-rings intact; evidence of external heat affecting the lubricant consistency, but internal combustion patterns not evident)
- Auxiliary Port Assembly (poppet and O-rings intact)
- Flow Head Assembly (components and O-rings intact)
- Shuttle Valve Assembly (components, O-rings, and backup rings intact; evidence of external combustion residue projecting into portions of the shuttle valve, but, no evidence of internal combustion evident)

The components listed above were inspected and documented by all parties present during the WHA joint inspection. During disassembly, WHA observed combustion product deposits inside some component housings. However, analysis revealed the residue was not associated with internal combustion. Several components have ports drilled into them that allow communication with the external environment, such as the regulator atmospheric sensing port (**Figure 10**). The combustion deposits were consistent with the external fire depositing combustion products on these components through the external ports onto surfaces within the VIPR housing. However, the VIPR did not exhibit internal fire-damage, melt-flow patterns, or propagation patterns that were consistent with fire originating inside or within the VIPR.



Figure 10 – Regulator Atmospheric Vent Port Illuminated from Exterior



A - Burst Disk and Gasket



B - Fill Valve Assembly



C - Pressure Gauge and Snubber Orifice



D - Regulator Assembly, PTFE Piston, Spring and Inlet Filter (lubricant on filter surface)



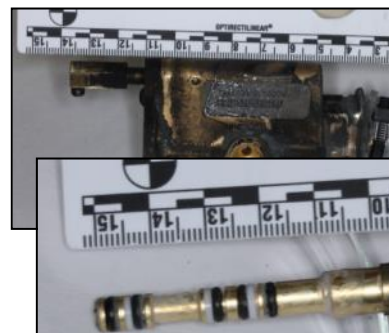
E - Relief Valve Assembly



F - Auxillary Port Assembly



G - Flow Head Assembly



H - Shuttle Valve



Figure 11 – VIPR Internal Components Disassembled

Figure 11 shows that all the internal soft goods, springs, and valve assemblies were intact and did not appear to be fire-damaged or substantially changed from their original condition. As component assemblies were removed, documentation of the condition of the ports was obtained with still photography and with boroscope examination (as necessary to visually access deeper porting). No evidence of internal combustion or combustion propagation was observed (see **Appendix A** documentation package).

4.2 Chemical Analysis by FTIR

Chemical samples were taken and Fourier Transform Infrared Spectroscopy (FTIR) was performed to evaluate the classification of the plastics, elastomers, and lubricants, to evaluate whether contamination such as hydrocarbon could be identified. Approximately 56 separate samples were obtained by WHA and analyzed by FTIR. **Appendix C** provides a summary of all the samples taken by WHA for chemical analysis and all FTIR spectra are included in the data package referenced in **Appendix A**. **Table 1** provides a general summary of the FTIR analysis.

Table 1 – General Summary of FTIR Softgood Analysis

Softgood/Lubricant	Identification	Comment
Lubricant ^a	Perfluoropolyether grease (Krytox GPL, or equivalent)	The white-colored lubricant observed on several internal components was consistent with a Krytox GPL or 240AC oxygen-compatible perfluoropolyether.
Burst Disc Gasket	Polyamide	Consistent with a polyamide material, like Nylon or equivalent
Fill Valve O-rings	FKM	Consistent with FKM material, like DuPont Viton or equivalent
Cylinder O-Ring	PTFE	Consistent with PTFE material, like DuPont Teflon or equivalent
Regulator Seat	PTFE	Consistent with PTFE material, like DuPont Teflon or equivalent
Regulator piston large O-Ring	FKM	Consistent with FKM material, like DuPont Viton or equivalent
Regulator piston small O-ring	EPDM	Consistent with a mixture of EPDM and PTFE or equivalent.
Regulator Filter (lubricant transfer)	Perfluoropolyether grease (Krytox GPL, or equivalent)	The white-colored lubricant transferred onto the regulator filter was consistent with a Krytox GPL (or equivalent) compatible perfluoropolyether grease
Shuttle Valve O-Rings Black O-rings White Backup Rings	FKM PTFE	Consistent with FKM, Viton or equivalent Consistent with PTFE, Teflon or equivalent.
^a Many samples exhibited a mixture of the base material covered by a layer of perfluoropolyether lubricant.		

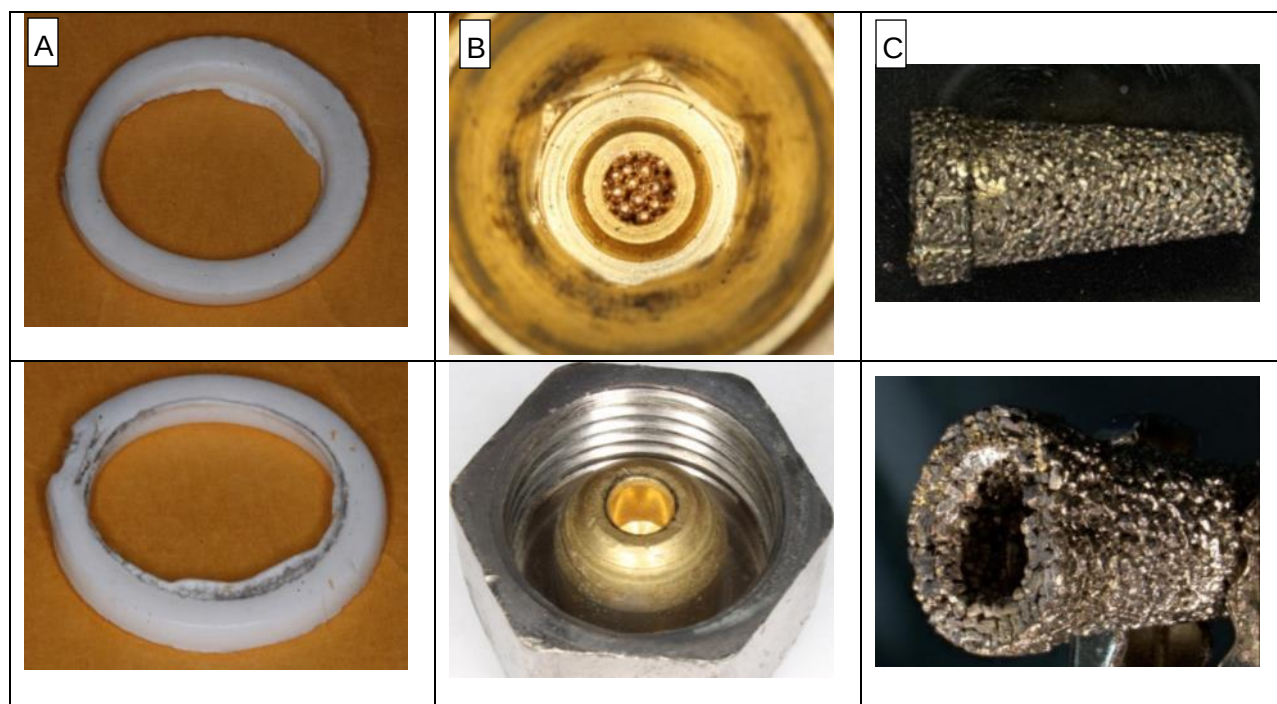


Figure 12 – Photographs of the Cylinder O-ring seal (A), Inlet Fill Port Filter and Cap (B), and Cylinder-to-VIPR Filter (C)

Figure 12 depicts the condition of what was provided to WHA as the cylinder O-ring (determined by FTIR to be PTFE) and two of the VIPR filters. The inlet fill port filter and protective cap are also shown in this figure. These components were visually clean but solvent rinses of the filter indicated detectable levels of a hydrocarbon contaminant. The cylinder-to-VIPR filter is also depicted and after careful removal from the VIPR threads, was observed to exhibit an intact and largely clean appearance. However, detectable levels of hydrocarbon were also observed on this filter for both careful solvent rinsing of the interior conical bore and solvent rinsing of the external conical structure. During these solvent rinses the filter was positioned so that careful solvent rinses would drain from the filter in such a manner that the interior and exterior would not be cross-exposed.

Figure 13 depicts the results of the FTIR sampling, comparing the fill port filter and the cylinder filter (interior and exterior). Each sample taken from these filters exhibited aliphatic hydrocarbon functionality (~2800 to 3000 wavenumbers) consistent with contamination. **Figure 14** depicts the FTIR sampling of the cylinder-to-VIPR filter, which also exhibited predominant hydrocarbon contamination and weaker functionality for Dioctyl Sebecate (discussed later). These samples also exhibited functionality consistent with a perfluoropolyether grease (**Figure 14**, Krytox or equivalent, ~900 to 1300 wavenumbers), which was a common finding with many samples, considered a result of the lubrication of the VIPR assemblies with an oxygen-compatible grease during assembly.

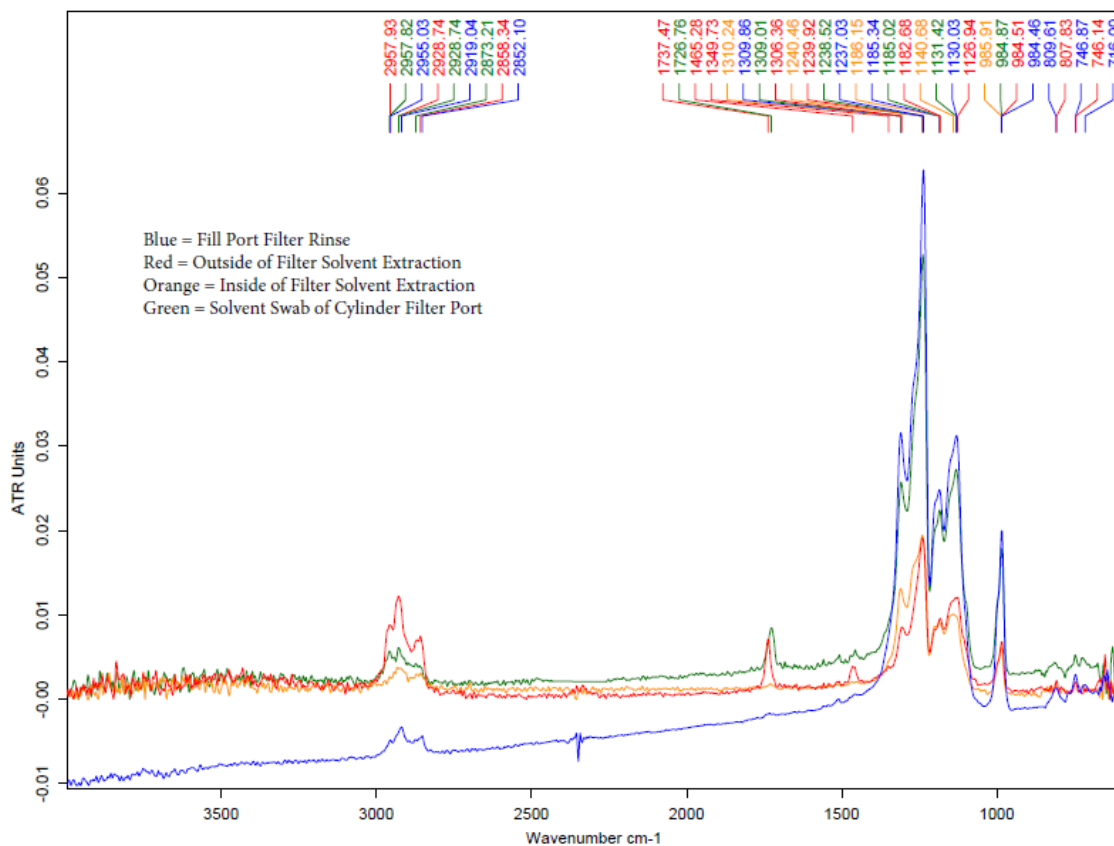


Figure 13 – FTIR Comparisons of VIPR Fill Port and Cylinder-to-VIPR Filter

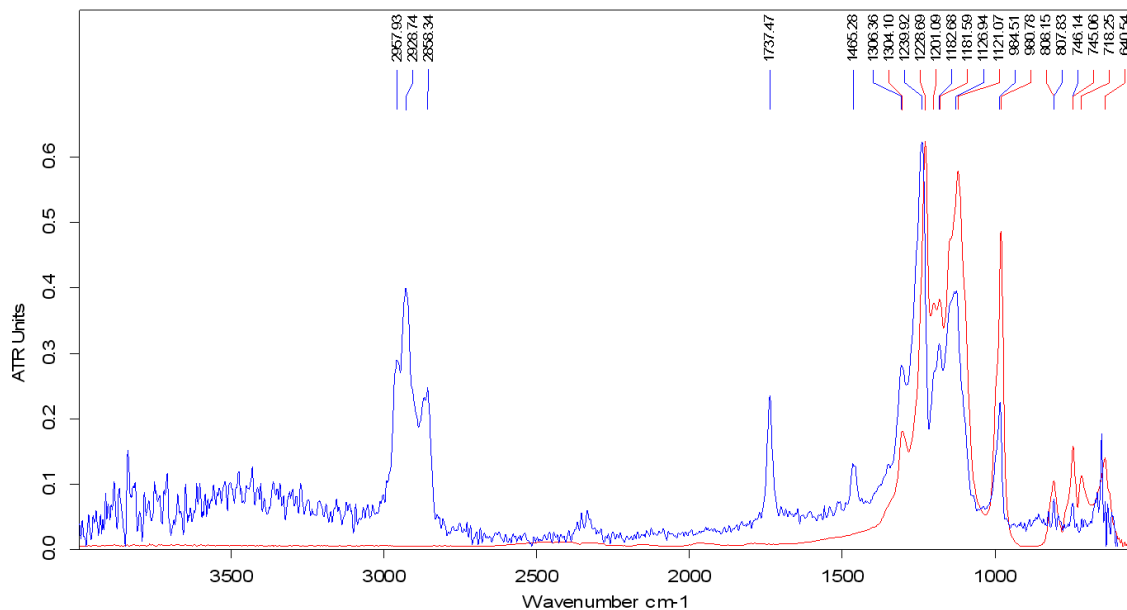


Figure 14 – FTIR Comparison of Exterior Solvent Rinse of Cylinder-to-VIPR Filter with Krytox

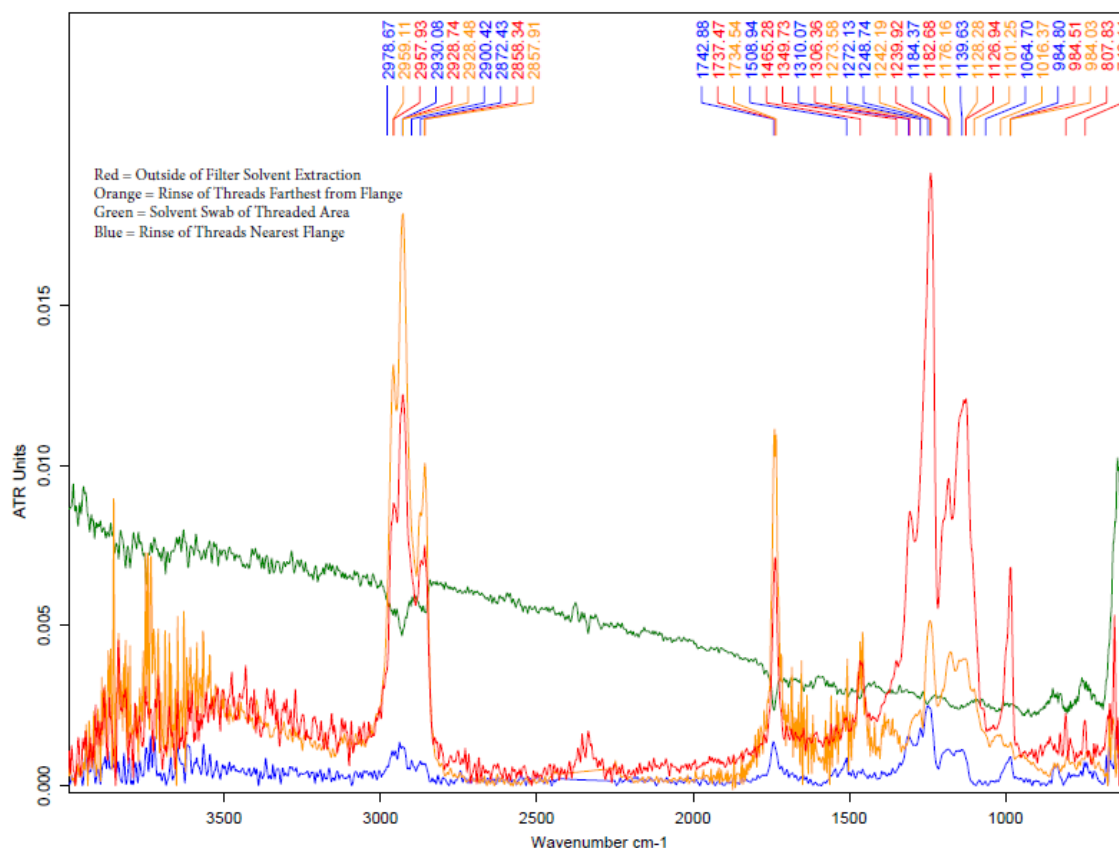


Figure 15 – FTIR Comparisons of Solvent Rinses of VIPR Threads

Figure 13 also depicts a solvent swab of the cylinder-to-VIPR port surfaces after the filter was removed (blue line). This sample also exhibited evidence of contamination with hydrocarbon.

Figure 15 depicts a comparison of several solvent rinses on the VIPR threads compared to the solvent rinse of the exterior of the cylinder-to-VIPR filter. These comparisons show that measurable levels of predominantly hydrocarbon and Krytox-like grease could still be detected on the threads after the fire, especially in the areas most damaged away from the flange region (orange compared to blue lines). The FTIR analysis indicated that the VIPR exhibited hydrocarbon contamination extending from the fill port inlet through to the cylinder filter and into the VIPR/cylinder threads.

As shown in **Table 1** and **Appendix C**, the regulator piston and cylinder-to-VIPR seals were identified as a PTFE material, widely considered a superior oxygen compatible plastic, which exhibits high Autogenous ignition temperatures (AIT, >450 °C or greater) and low heats of combustion (Hc, ~2000 cal/gram or less). The VIPR design incorporated FKM (Viton, or equivalent) O-rings in the fill port and the regulator piston seal (large seal), which are also considered to be an oxygen-compatible elastomer (AIT ~300 °C; Hc ~3000 cal/gram). The VIPR included EPDM O-rings on the regulator's small seal and an O-ring that served as a relief seat seal, which has lower oxygen compatibility (AIT ~ 206 °C; Hc ~9200 cal/gram) than Viton, but did

not exhibit evidence of fire involvement. A lubricant was also identified in the chemical analysis as consistent with Krytox (perfluoropolyether lubricant), which is considered a superior oxygen compatible lubricant similar to PTFE materials. None of the internal plastics or elastomers exhibited evidence of ignition or combustion, as indicated in the photographs in **Figure 11** (see also the **Appendix A** documentation package).

Some investigations on Grab-n-Go VIPR fires have attributed critical contamination to an EPDM o-ring gland seal that provides the seal between the VIPR and cylinder. In the present investigation, the gland seal o-ring was PTFE and not EPDM. However, these allegations persist. Contamination from EPDM gland seals have been attributed to a plasticizer incorporated into the seal to provide flexibility, primarily Dioctyl Sebacate (DOS), that is alleged to have migrated from the o-ring during service. **Figure 16** shows FTIR spectra for this plasticizer (plasticizer spectra only, not identified with this evidence).

As shown, DOS plasticizers exhibit a characteristic stretch in the aliphatic hydrocarbon region, located between approximately 2800 cm^{-1} and 3100 cm^{-1} . DOS also exhibits a characteristic carbonyl stretch at 1737 cm^{-1} . It is noteworthy that the carbonyls relative absorbance peak amplitude is characteristically greater than the aliphatic hydrocarbons relative peak amplitude.

In contrast to **Figure 16** for DOS (only), the contamination found in the VIPR and cylinder were predominantly aliphatic hydrocarbons inconsistent with only DOS. **Figure 13**, **Figure 14**, and **Figure 15** depict weak carbonyl stretches; however, the ratio of the carbonyls relative absorbance to the hydrocarbons relative absorbance indicates that an unidentified hydrocarbon-based contaminant was predominant in the samples. If the contamination were solely due to DOS, the spectrum would show a larger carbonyl peak as compared to the aliphatic hydrocarbon peak, which was not the case. Therefore, the hydrocarbon-based contaminant observed on the incident components was primarily an unidentified saturated hydrocarbon³.

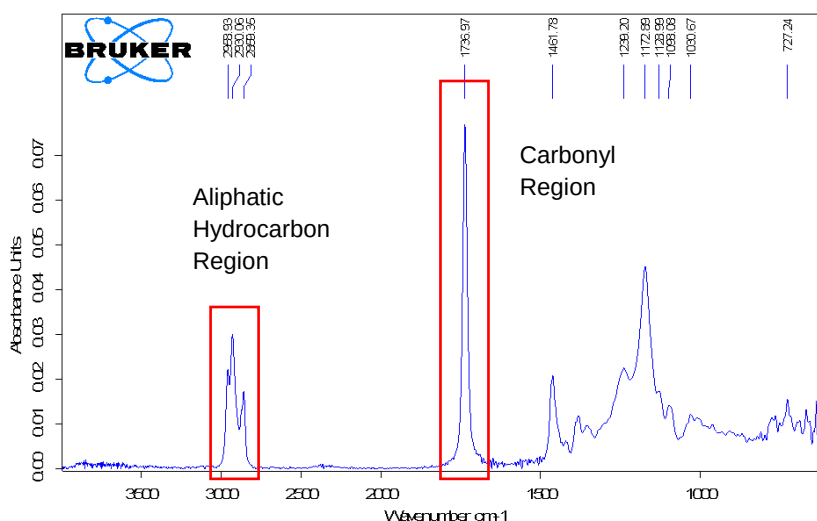


Figure 16 - Dioctyl Sebacate Characteristic FTIR Spectra

³Some spectra shown in Appendix C also exhibit indications of unsaturated alkene modalities.

The presence of this hydrocarbon extending from the VIPR fill port to the VIPR cylinder interface filter (through the VIPR) was consistent with the unidentified hydrocarbon contamination being introduced from external sources into the VIPR/cylinder package.

The flange and thread area of the VIPR, proximate to the cylinder-to-VIPR O-ring gland, did not exhibit melt-flow or evidence of significant combustion. This was also consistent with the PTFE O-ring, which was recovered and provided to WHA as the cylinder seal. It is noteworthy that for an initial local origin involving the O-ring at the cylinder to VIPR interface, the combustion patterns would be expected to exhibit intense burning at the O-ring gland followed by burnout and venting past the VIPR flange. These patterns were not observed at the flange area in the physical evidence. Instead, the evidence indicated heavier melt/flow and thermal involvement in the cylinder-to-VIPR threads, well below the flange O-ring seal.

The chemical analysis indicated that hydrocarbon contamination⁴ of the VIPR was observed in several locations from the fill port of the VIPR through the internal cavities and into the cylinder-to-VIPR threads. The extreme flammability and poor compatibility of hydrocarbon oils and greases is an indication that the origin of the incident was probably not within the internal cavities or components of the VIPR, since neither kindling nor combustion was observed⁵.

4.3 Scanning Electron Microscopy (SEM) with Energy Dispersive Spectroscopy (EDS)

Analysis of the elemental composition of the surface residues associated with the thermally-damaged areas of the VIPR was performed with a Scanning Electron Microscope (SEM) using Energy Dispersive Spectroscopy (EDS). By cooperative agreement, the SEM/EDS analysis was performed by WHA personnel at New Mexico State University (NMSU), near the WHA laboratories. WHA experience indicates that analysis and elemental characterization of the surfaces residues is useful for propagation analysis. Therefore, full metallurgical characterization was not necessary during this phase of the investigation.

The SEM/EDS work was scheduled initially during the joint inspection on a few areas of the VIPR threads and on the cylinder-to-VIPR filter; however, a fixture prepared by WHA for holding and positioning the VIPR in the chamber did not provide sufficient clearance to the EDS detector and the EDS work was halted until the fixture could be revised to provide better access inside the SEM chamber. After reworking the WHA positioning fixture (**Figure 17**), WHA performed SEM/EDS on the cylinder threads of the VIPR, which have been shown to exhibit the heaviest melt-flow and consumption patterns. **Figure 17** depicts the holding fixture that was utilized along with notations for the clocking of the VIPR threads, as designated by WHA, for the elemental analysis of the surface residues. The revised holding fixture provided very secure

⁴Hydrocarbon functionality was weak in some locations but was readily identifiable throughout. Hydrocarbon contamination in oxygen systems is an extreme hazard, readily ignited with low ignition energy and producing a vigorous combustion event.

⁵Weak carbonyl functionality was observed in some samples, which can be associated with EPDM plasticizers. However, since the only EPDM materials that were identified were associated with the regulator, and since the upstream filter leading to the regulator did not exhibit carbonyl functionality, it was considered unlikely that the carbonyl stretches were associated with the EPDM downstream in the VIPR regulator. Instead, the predominantly saturated hydrocarbon functionality was consistent with another unknown contaminant source.

positioning of the VIPR threads to allow positioning in a wide variety of orientations within the SEM chamber. In this way, the VIPR body and threads were maintained in an intact condition and the SEM/EDS analysis was performed in a manner that was non-destructive to the VIPR.

In general, the threads were analyzed at the 12, 3, 6, and 9 o'clock positions and EDS elemental analysis was obtained from the bottom remaining portions of the threads up to the flange. The heaviest melt/flow and consumption patterns on the brass threads was designated as the approximate 12 o'clock position. The least melt/flow and consumption was observed at the approximate 6 o'clock position (see **Figure 17**).



Figure 17 – SEM Chamber Positioning Fixture and Orientation of VIPR Threads for Elemental Characterization of Residues

The initial analysis performed confirmed that the body of the VIPR was consistent with a type of brass having a high copper and zinc content. Brass is a superior oxygen compatible metal, not considered flammable at the understood service conditions. The SEM/EDS analysis on the cylinder-to-VIPR filter indicated that it was a sintered filter, believed to be generally sintered bronze with nickel plating. This material is a superior oxygen-compatible material combination that has exhibited a good history of successful use. A summary of the SEM/EDS analysis is provided in **Appendix D**⁶. The following observations briefly summarize the SEM/EDS elemental analysis on the VIPR threads:

- Zinc (Zn) content was greater for regions of the thread that experienced more material loss (12, 3, and 9 o'clock positions). WHA experience indicates this is consistent with high temperatures and combustion and considers it possible that Zn may preferentially precipitate to the surface during combustion.

⁶See also the full documentation package incorporated in **Appendix A**, which by following the hyperlinks includes all SEM/EDS elemental analysis.

- Generally observed more elemental Fluorine (F) exhibited near the flange area and lesser near damaged areas (exception 9 o'clock region). The Fluorine observed is generally considered consistent with the presence of a perfluoropolyether grease having been on the threads, flange, and O-ring prior to the combustion event. Since the cylinder-to-VIPR O-ring was identified as PTFE, the fluorine may also be consistent with the O-ring, especially near the flange. However, Fluorine was observed away from the flange in the damaged and middle regions of the threads, which is considered more consistent with perfluoropolyether-based lubricant on the threads.
- Aluminum (Al) content was greatest for the 6 o'clock thread position (region of thread that experienced less material loss). The following summarizes each position:
 - 12 o'clock position: Al content (~10%) generally consistent throughout thread position from damaged end to flange. This was the approximate position observed to exhibit the heaviest brass consumption and/or melt/flow and exhibited the heaviest thread distortion.

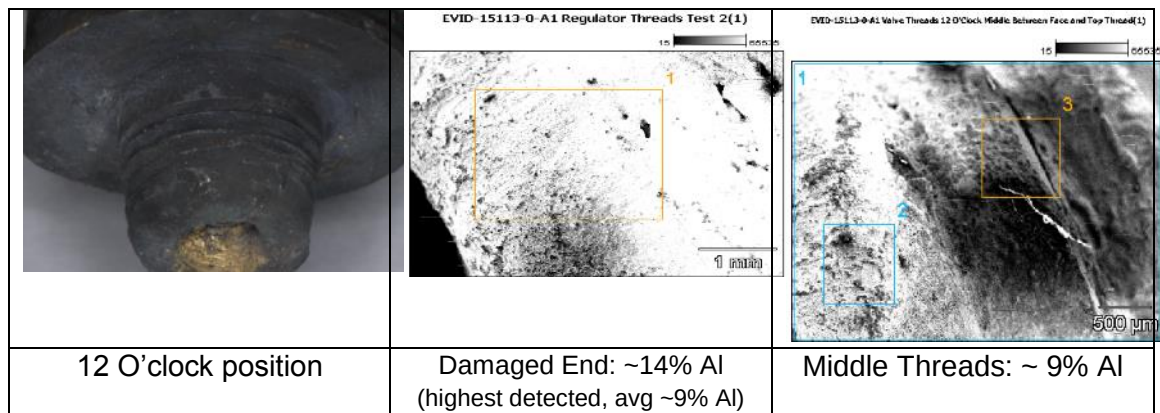


Figure 18 – 12 O'clock Position SEM Images

- 3 o'clock position: Al content generally higher on damaged end (~12%) than flanged end (~6%)
- 9 o'clock position: Al content (~12%) consistent throughout thread region from damaged end to flange, highest amount of Al (~18%) on thread in one location near the flange; however, overall, the Al concentration was reasonably consistent along this position.
- 6 o'clock position: Al content generally increasing from damaged end (~18%) to flanged end (~30%). This position exhibited heavy aluminum and aluminum combustion products clinging to the threads, probably having been less effected by aluminum combustion before the VIPR was ejected from the cylinder.

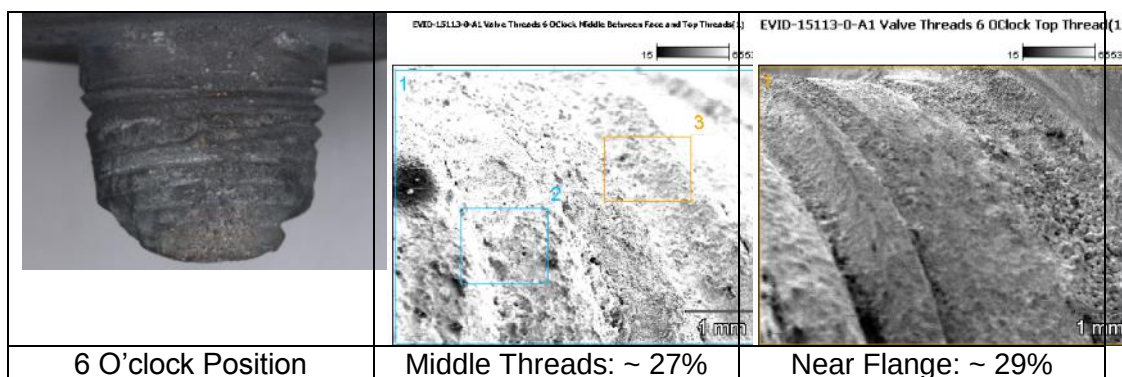


Figure 19 – 6 O'clock Position SEM Images

The heavier concentration of Al proximate to the 6 O'clock position was consistent with the VIPR probably being ejected before this region was as fully involved as compared to the 6 O'clock region. The length of the remaining threads in the 6 O'clock position was also closer to the pre-incident length. The 12 O'clock position exhibited greater mass loss and was probably involved with the aluminum combustion from the cylinder at an earlier stage in the propagation after ignition.

- Generally more lead (Pb) and molybdenum (Mo) were observed at the damaged end of thread position. It was noteworthy that the 6 o'clock position observed this trend for Mo but there was no Pb observed in this region (region of lesser material consumption). Lead content is common to brass, but, the molybdenum is not common to either brass or 6061 Aluminum (assumed cylinder alloy). Molybdenum disulfide is, however, used in some Krytox grease compounds⁷.
- Carbon (C) and Oxygen (O) were typically higher on the damaged end of threads (12, 3, 9 o'clock positions) where extreme heat and oxidation patterns were observed. The exception was at the 6 o'clock position where oxygen (O) remained consistent throughout the threaded region and carbon (C) was generally higher on the damaged end (region of lesser material consumption).
- Copper (Cu) content was generally consistent throughout each thread region, except positions 3 and 9 where Cu content was generally higher at the flanged end (lesser at damaged end). WHA experience indicates that combustion can be expected to change the alloy concentrations where mixing and precipitation of light alloy and alloy oxides develop.

⁷The Krytox that WHA provided comparison to is Krytox GPL206, which has a white coloration and consistency similar to that observed on many of the VIPR components. Krytox 240AC is also similar and both are commonly used as oxygen compatible greases. However, Krytox GPL 210-217 contains molybdenum disulfide added as an extreme pressure additive for highly loaded components; but, has a gray color. It does have the same/similar PFPE oil that they use in the Krytox mixtures. The samples taken by WHA were mostly white but some had a tint of grey. However, color is difficult to judge visually especially after a combustion event.

4.4 Documentation and Discussion of Internal and External Combustion Patterns

The VIPR exhibited combustion and slag deposits on the majority of its external components and external surfaces. However, external fire damage to the VIPR was generally limited to the cylinder-to-VIPR thread connection. Fire damage to the thread connection was also heaviest on the lower 12 o'clock region of the threads away from the flange and flange O-ring seal. VIPR damage to the cylinder thread post is shown in **Figure 20**. Noteworthy bending, consistent with impact, was also observed on the flange and the pressure gauge at 6 o'clock (**Figure 6**).

Figure 8 and **Figure 9** depict various views of the VIPR threads, VIPR flange region proximate to what would have been the cylinder and cylinder O-ring gland. These figures depict the very heavy melting and melt-flow associated with the lower segments of the VIPR threads. The SEM/EDS analysis was consistent with evidence of heavy aluminum combustion products deposited on the VIPR threads and suggested that aluminum combustion had developed initially in the lower segment of the threads proximate to the 12 o'clock region⁸.

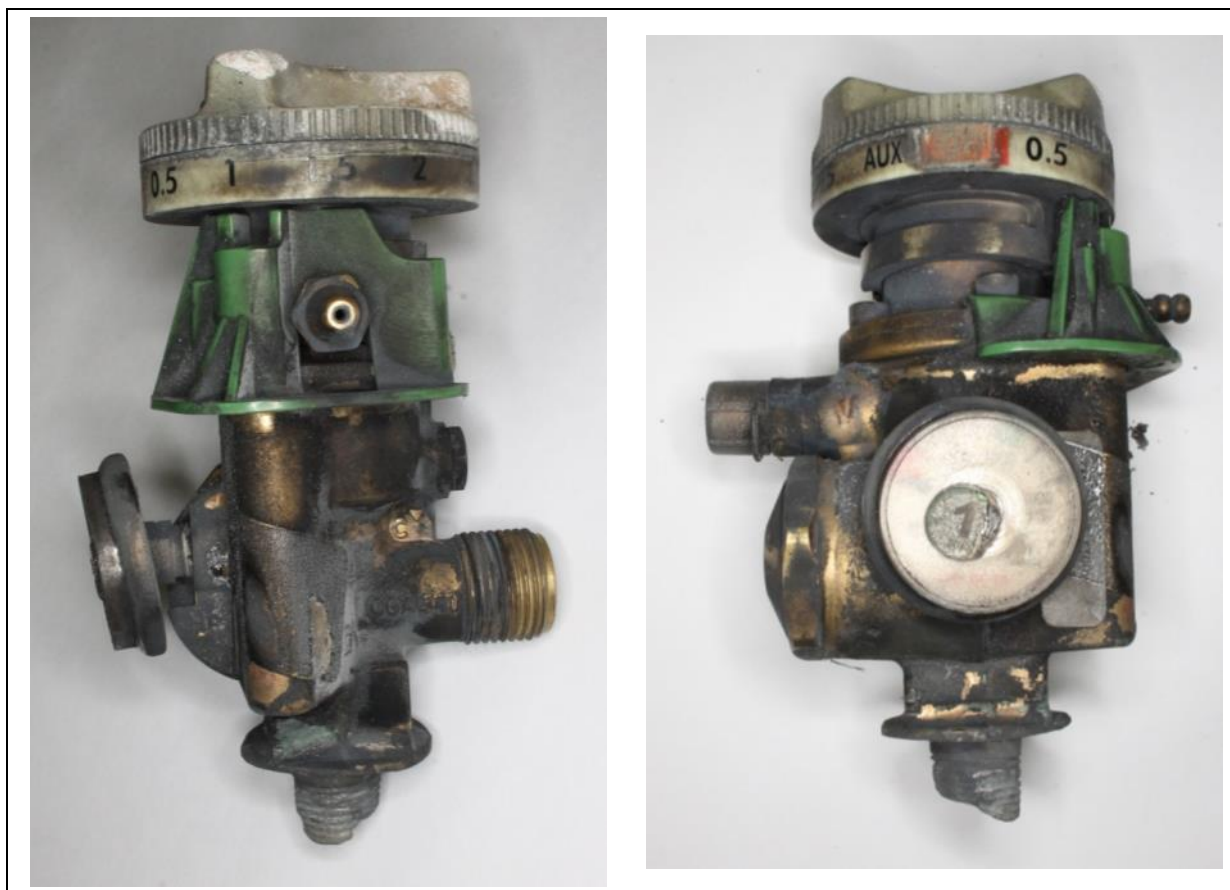


Figure 20 – External views of the incident VIPR

Experience with brass combustion in high-pressure oxygen indicates that it will experience self-extinguishing combustion, if promoted by another material, but will not readily self-sustain

⁸WHA analysis of origin is limited due to the lack of information on the aluminum cylinder combustion patterns.

combustion at the elevated oxygen pressures understood to have been present. This characteristic is observed in the physical evidence with the threads melting in some locations but resisting being fully promoted. However, aluminum in high-pressure oxygen conditions, once ignited will readily support energetic self-sustained combustion⁹. The combustion event, once the aluminum was kindled, would be expected to dominate combustion and continue until the VIPR was ejected with force and/or the cylinder ruptured under the extreme combustion conditions.

It was especially noteworthy that the cylinder threads and VIPR threads were heavily involved; however, the cylinder O-ring seal and associated area of the VIPR flange exhibited much less damage and very little, if any, melt-flow. The flange and thread area of the VIPR, proximate to the cylinder-to-VIPR O-ring gland, did not exhibit melt-flow or evidence of significant combustion. This was also consistent with the PTFE O-ring which was recovered and provided to WHA as the cylinder seal. This observation favors an origin likely to involve lower thread area of the cylinder and VIPR and not a region around the cylinder-to-VIPR O-ring seal. WHA experience indicates that had the cylinder O-ring ignited first, the fire patterns would have emanated from the area of the cylinder gland and heavy consumption patterns would be expected to involve the flange region of the VIPR and the associated O-ring seal. Since this O-ring is the pressure seal, the fire and high-pressure oxygen would have been expected to rapidly vent from this region and would not have been expected to propagate into the lower threads before venting oxygen externally. These patterns were not observed at the flange area in the physical evidence. Instead, the evidence indicated heavier melt/flow and thermal involvement in the cylinder-to-VIPR threads, well below the flange O-ring seal. As stated above, however, WHA has not analyzed the cylinder and its propagation patterns.

Significantly, the VIPR threads exhibited hydrocarbon contamination, which was observed extending from the fill valve filter to the cylinder-to-VIPR threads through the cylinder filter (see FTIR analysis). Experience indicates that hydrocarbon contamination, even in low levels, will migrate readily with the gas flow. It is noteworthy that the cylinder inlet filter was observed to be directly exposed to combustion energy in the fire event; however, **Figure 12** shows the inlet filter is still intact and relatively undamaged (see also **Figure 8**). The lack of damage to the filter was consistent with an origin external to the VIPR and not developing from within the VIPR (i.e., inside-to-outside) since internal combustion must propagate through the filter in order to kindle the lower threads and cylinder wall.

5 Opinions and Conclusions

As requested by PHMSA, the WHA activities included a forensic analysis of the valve components, utilizing, as needed, Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) with Energy Dispersive Spectroscopy (EDS), to determine if the use of inappropriate materials in the valve may have resulted in combustion inside the pressurized oxygen service DOT 3AL cylinder. This report does not include evaluation of fire

⁹It is noteworthy that based on WHA testing aluminum is difficult to ignite due to a very protective surface oxide but will self-sustain combustion if a sufficiently high ignition energy can be delivered.



patterns on the incident cylinder. Analysis of only the Valve Integrated Pressure Regulator (VIPR) was requested.

Based on the investigation completed to date, the chemical and metallurgical analysis, and the materials provided for review, the following opinions and conclusions (limited to the VIPR observations and analysis) have been developed. Detailed analysis and inspection of the incident cylinder, if requested, could influence the conclusions stated herein. If additional information is provided or additional inspection/analysis is performed, then we reserve the right to amend these opinions and conclusions accordingly:

- 1) Based on the thermal damage, consumption, and melt/flow patterns on the VIPR, the heaviest burning was consistent with the cylinder-to-VIPR thread interface in the lower portions of the threads away from the cylinder O-ring seal, based on the lack of burning at the VIPR flange. The initial ignition and first material to ignite could be associated with the lower portion of the threads; however, WHA has not analyzed the corresponding areas of the cylinder. Since the o-ring seal was not heavily burned and was recovered as part of the evidence, and since the internal pressure would cause fire to flow outward if the seal ignited in the early stages, the evidence is consistent with an origin away from the VIPR-to-cylinder gland seal. Cylinder pressure would be expected to eject fire early if seal ignition occurred early and to produce less combustion than is observed at the threads.
- 2) The physical evidence was inconsistent with initial ignition and propagation from within the VIPR. Inside-to-outside propagation from the VIPR would be expected to have led to propagation patterns inside the VIPR extending through the cylinder filter, which was not observed. The burn patterns, described previously, were focused outside of the VIPR and at the lower interface to the cylinder threads.
- 3) The brass comprising the VIPR body and threads is a superior material that resisted ignition and propagation, as was observed in the evidence; and, would be expected from WHA combustion testing of brass in high-pressure oxygen. The majority of the softgoods and lubricants comprising the VIPR construction were PTFE-based, FKM-based, and Krytox-like (perfluorinated polyether grease), which are generally considered oxygen compatible and have been successfully used in oxygen service. At least two O-rings in the regulator portion of the VIPR were consistent with EPDM, which exhibits lower compatibility with oxygen (i.e., more easily ignited) but is an elastomer that exhibits good mechanical properties and is currently being used in many oxygen components. However, neither the EPDM nor any other internal softgood exhibited evidence of ignition, combustion, or propagation.

- 4) Evidence of hydrocarbon contamination was observed in the chemical analysis of the VIPR filters and threads¹⁰. Figures 13 -15 are consistent with an outside-to-inside migration of hydrocarbon contaminant that migrated through the VIPR and to the cylinder-to-VIPR threads. Since the inlet filter (fill-port) exhibited the same type of contamination as observed elsewhere, it is considered more likely than not that the source was external due to handling and/or external filling operations.
- 5) Some previous investigations have implicated DOS, a plasticizer in EPDM o-rings sometimes used as the gland seal on these systems, as the type of hydrocarbon contamination associated with the critical kindling chain. In the present investigation; however, a PTFE VIPR-to-cylinder o-ring was installed, rather than EPDM. Further, the hydrocarbon identified by the chemical analysis reported herein was predominantly an aliphatic-based hydrocarbon with weak carbonyls stretching. The ratio of the carbonyls relative absorbance to the aliphatic hydrocarbons relative absorbance was inconsistent with DOS and indicated that an unidentified hydrocarbon-based contaminant was predominant in the samples. Further, the presence of this hydrocarbon extending from the VIPR fill port filter to the VIPR cylinder interface filter (through the VIPR) was consistent with the unidentified hydrocarbon contamination being introduced from an external source(s) into the VIPR/cylinder package.
- 6) Based on the observed bending of the VIPR-to-cylinder flange, the ignition energy was probably external impact of the VIPR/cylinder package, potentially with rotational friction at the threads. The most ignitable material observed would have been the hydrocarbon contamination. While hydrocarbon-based contaminants are known to ignite readily in sufficient concentration in high-pressure oxygen, they still require ignition energy to initiate.
- 7) While WHA test experience indicates that aluminum is very difficult to ignite directly in this configuration, a critical kindling chain evidently developed and may have involved a combination of the following: a) hydrocarbon contamination, b) Krytox lubricants, c) aluminum thread shavings, d) thin sections of aluminum threads, e) and/or other materials in the cylinder thread-to-VIPR interface. While Krytox is considered an oxygen-compatible lubricant, it is still flammable under the conditions that existed and could decompose to potentially react with aluminum under elevated temperature conditions. Disruption of the aluminum surface oxides due to impact, VIPR rotation (i.e., friction and severe thread interaction) would be expected to provide sufficient energy for a critical kindling chain to initiate.

¹⁰Some of the hydrocarbon was detected at a low level; however, its presence after a fire is significant. Further, even low levels of hydrocarbon contamination are of concern in a high-pressure oxygen system.

Appendix A – Joint Inspection Report



CONFIDENTIAL

15113-0 Inspection Summary and Data Package

Report Number:

R-WHA-15113-0-A-EX1

February 16, 2016

Prepared for:

Mr. Mark Toughiry

US Department of Transportation (DOT)

1200 New Jersey Ave., SE

East Building, 2nd Floor

Washington, DC 20590

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15113-0 Inspection Summary and Data Package

Prepared by:



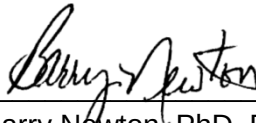
Marcus Johnson, BSME

Reviewed by:



Jared Hooser, MSME

Approved by:



Barry Newton, PhD, PE

Table of Contents

1	Introduction	1
2	Pre-Inspection Documentation.....	1
3	Inspection	2
3.1	Visual Examination	2
3.2	Borescope	2
3.3	Microscope	2
3.4	VIPR Disassembly	3
3.5	FTIR Sampling and Analysis.....	3
3.6	SEM-EDS Analysis.....	3
3.7	Further Analysis.....	4



1 Introduction

This document provides general summary of activities and the resulting documentation for the following inspection:

Date: Friday, January 8, 2016
Location: WHA International, Inc.
Test Facility
5605 Doña Ana Road
Las Cruces, NM 88007
Case: Valve Integrated Pressure Regulator Analysis for PHMSA
Protocol: WHA Forensic and Laboratory Procedures
Purpose: Disassembly, inspection, and analysis on the incident hardware

Table 1 lists the evidence identifying numbers (IDs) of the evidence items received by WHA.

Table 1 – WHA Evidence ID numbers.

WHA Evidence ID	Description
EVID-15113-0-A1	Valve (VIPR) (Bar Code: M002270 / 1003572; Partial Label: _002270; _00357_)
EVID-15113-0-A2	Green metal fragment
EVID-15113-0-A3	White O-ring

2 Pre-Inspection Documentation

In order to facilitate timely progress during the inspection, parts of the incident hardware were visually and microscopically examined and documented by WHA personnel prior to the inspection. Videos of these pre-inspection examinations are located in the folder: [05 - Microscopy/VIPR Microscope Overview Video](#)

The areas visually examined prior to the inspection were as follows:

- Incident VIPR (EVID-15113-0-A1)
 - Face of cylinder threads proximate to filter
 - Cylinder interface flange/root
 - Cylinder interface flange
 - Cylinder interface threads

After visual examination, the incident VIPR was transported by WHA personnel to American X-Ray Inspection Services (A.X.I.S.) in El Paso, TX for radiographic examination. The resulting X-



ray images were used during the joint inspection to guide decisions about disassembly of the incident VIPR. Photographic, microscopic, and radiographic images resulting from the pre-inspection examinations are available in the folders: [03 - Visual Inspection](#), [05 - Microscopy](#), and [06 - Radiography](#).

3 Inspection

3.1 Visual Examination

Each piece of incident hardware was visually examined prior to any chemical sampling or disassembly. Photographs from the visual examination of the incident hardware are located in the folder: [03 - Visual Inspection](#).

3.2 Borescope

Camera borescopes with fiber-optic lighting were used to visually examine the following areas on the incident VIPR (EVID-15113-0-A1) prior to disassembly.

- Cylinder-to-VIPR inlet port(with and without filter)
- Fill port (with and without filter)
- Regulator port (after removal of components)
- Rupture disc port (after rupture element removal)

The videos and photos from the borescope examination are located in the folder: [04 - Borescope](#).

3.3 Microscope

All microscopic examination was conducted by WHA personnel using a Keyence® Digital Microscope. The microscopic examination process was video documented; the video files are located in the folder: [05 - Microscopy\VIPR Microscope Overview Video](#)

Prior to chemical sampling and disassembly, the following areas of the incident hardware were examined and imaged with the digital microscope:

- Incident VIPR (EVID-15113-0-A1) [05 - Microscopy\VIPR Inlet](#)
 - Face of cylinder-to-VIPR threads proximate to filter
 - Cylinder interface inlet filter
 - Cylinder interface inlet flange
 - Cylinder interface flange/root (O-ring position)
 - Cylinder interface threads

The following parts were examined and imaged with the digital microscope after disassembly of the incident VIPR (EVID-15113-0-A1) but prior to chemical sampling of the parts.



- Incident VIPR (EVID-15113-0-A1) [05 - Microscopy\VIPR Inlet](#)
 - Cylinder interface inlet filter

3.4 VIPR Disassembly

Disassembly of the incident VIPR was discussed and performed in a manner agreed to by all parties present at the inspection. The disassembly process was video and photo documented. Videos and photos of the disassembly are located in the folder: [09 - VIPR Disassembly](#)

3.5 FTIR Sampling and Analysis

All FTIR sampling was performed using standard laboratory procedures. FTIR samples were taken from the following areas of the incident hardware:

- Incident VIPR (EVID-15113-0-A3)
 - Cylinder interface threads (various areas including flange)
 - Cylinder interface o-ring (as recovered)
 - Cylinder interface filter (after removal)
 - Fill port (various sections including poppet after removal)
 - Pressure gauge port and socket up to snubber
 - Burst disc and gasket
 - Regulator assembly and selected internal components
 - Relief valve and selected internal components
 - Various internal soft-goods and lubricants

A detailed description of the FTIR sampling methods used during this inspection and a log sheet of all samples taken along with videos of the sampling process are located in the folder: [07 - FTIR\Sampling](#)

The resulting FTIR data files from each sample and a summary table of results are located in the folder: [07 - FTIR\Analysis](#)

3.6 SEM-EDS Analysis

Scanning Electron Microscope (SEM) with Energy Dispersive X-ray (EDS) was performed to characterize the residues and surfaces of the VIPR cylinder threads after chemical analysis samples had been removed from the threads (see FTIR sampling above). The entire VIPR body was mounted so that no destructive sectioning was necessary to conduct the analysis. The mounting in the SEM chamber was video documented as shown in the files located at the link below. All SEM photomicrographs and EDS scans showing the elemental breakdown of the threads scanned during the SEM-EDS analysis are also provided in the folder: [08 - SEM EDS](#).

As desired by the experts, no changes to the VIPR were made to accomplish the analysis and replication of the SEM/EDS (or additional analysis) can be performed at the discretion of the



parties. Potential destructive sectioning has NOT been performed, as agreed, but is still under consideration.

3.7 Further Analysis

Further metallurgical analysis and destructive sectioning of the VIPR threads, and potentially the cylinder, if provided, was considered by all experts at the WHA joint inspection. However, at the current stage of the investigation the aforementioned data was desired before a decision could be made pertaining to further destructive sectioning. Therefore, sectioning has NOT been performed at this stage of the investigation.

All current evidence, FTIR samples and SEM/EDS positions are maintained by WHA and can be reanalyzed at the discretion of the parties.



Appendix B – Praxair Grab ‘n Go Literature and Recall Details



PRAXAIR

Grab 'n Go®

Advanced Respiratory Systems

Before using or working with the Grab 'n Go system, please read and understand the usage and safety instructions provided in this booklet.

Introduction

The Praxair *Grab 'n Go*® III and *Grab 'n Go Vantage*™ Advanced Respiratory Systems provide an easy-to-use package for those who need oxygen supplied from portable cylinders. They're easy to use because the oxygen regulator and pressure gauge are combined and permanently attached to the gas cylinder as one unit. This innovative design eliminates difficulties locating and attaching a separate regulator, saving you time and money.

These operating instructions will:

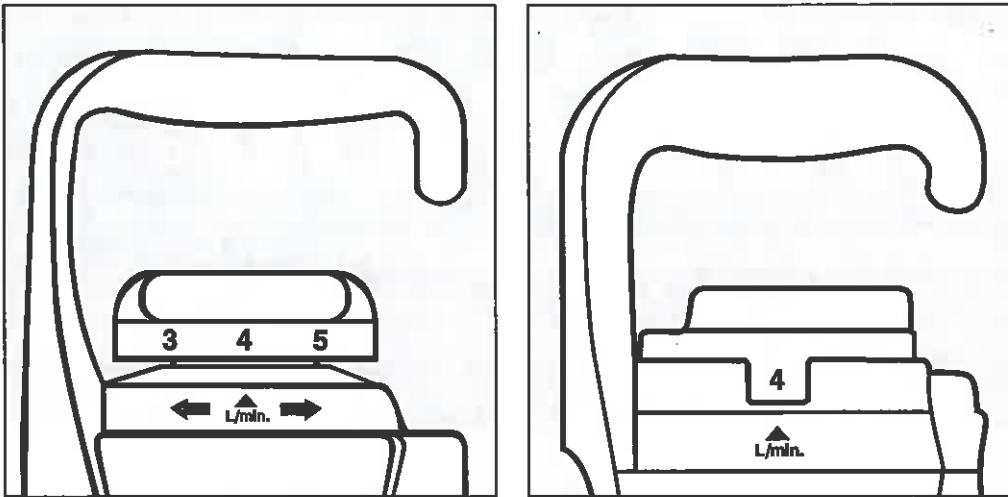
- (1) introduce you to the features of the *Grab 'n Go* III and *Grab 'n Go Vantage* units;
- (2) review the safety precautions for working with oxygen; and
- (3) explain the setup and use of the *Grab 'n Go* III and *Grab 'n Go Vantage* units to ensure that you can use it properly and safely.



Shown on steel cylinder

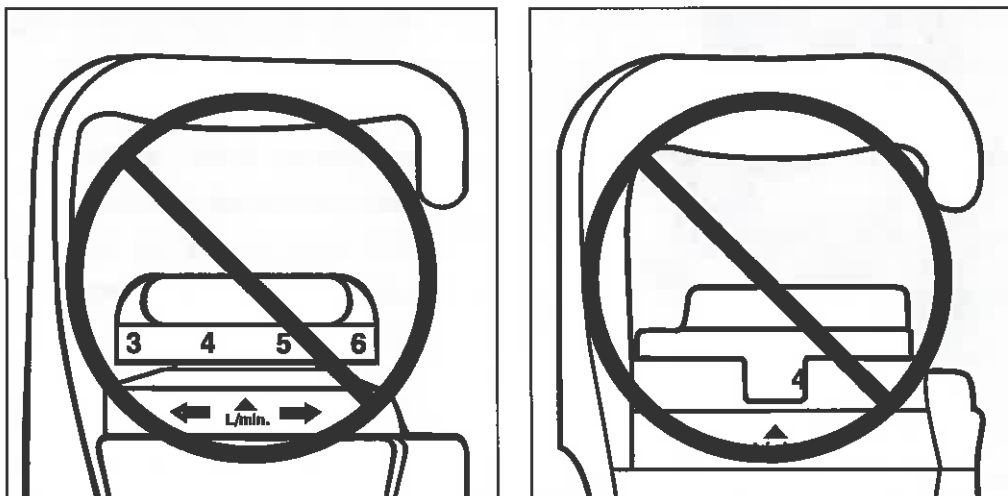
Shown on aluminum cylinder

Correct



The **Grab 'n Go** systems operate **ONLY** when positioned at the flow rates marked on the flow-adjusting knob. The flow rates are marked in liters per minute. If the intended flow rate is not marked on the flow-adjusting knob, **DO NOT ATTEMPT TO USE THE SYSTEM**. Consult your physician for a different flow rate or a different system.

Incorrect



DO NOT OPERATE THE SYSTEM BETWEEN MARKED FLOW RATE SETTINGS. Remember oxygen flows properly only when the flow-adjusting knob is stopped at one of the numbered click stops. Always verify flow with the patient's end of the tubing before applying.



Series III and Vantage features

The *Grab 'n Go* system contains an integral regulator; a flow-adjusting knob to set the flow of oxygen in clearly numbered increments; an easy-to-read gauge that displays a continuous indication of cylinder pressure; and a durable plastic housing that doubles as a convenient carrying handle.

Product Description

The *Grab 'n Go III* system provides flow rates from 0.5 to 15 LPM. The *Grab 'n Go Vantage* series, with its distinctive all white flow-adjusting knob, provides flow rates from 0.5 to 25 LPM and a 50 psig auxiliary connection to supply demand valves or portable ventilators at flow rates up to 100 LPM.

Flow Rate's Available (Liters Per Minute)

Grab 'n Go III									
0.5	1.0	1.5	2.0	2.5	3.0	4.0	5	10	15
Grab 'n Go Vantage									
0.5	1.0	1.5	2.0	3.0	4.0	6.0	8.0	15	25

Auxiliary 50 psig Port Availability

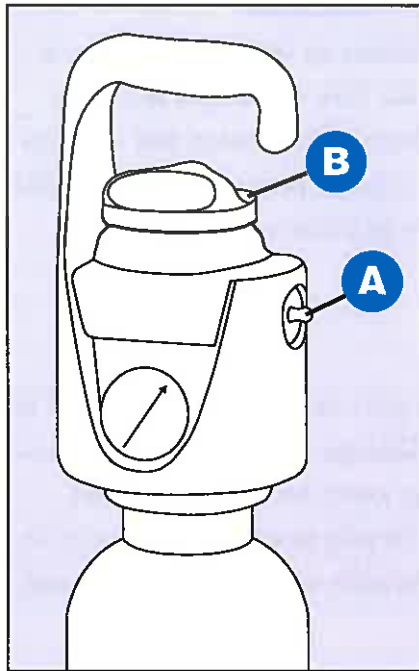
Grab 'n Go III	No
Grab 'n Go Vantage	Yes

Maintenance:

For the user, there is no special maintenance required. Flow is metered through a series of fixed orifices inside the regulator. The *Grab 'n Go III* and the *Grab 'n Go Vantage* units are maintained and tested by Praxair in accordance with manufacturer's instructions.

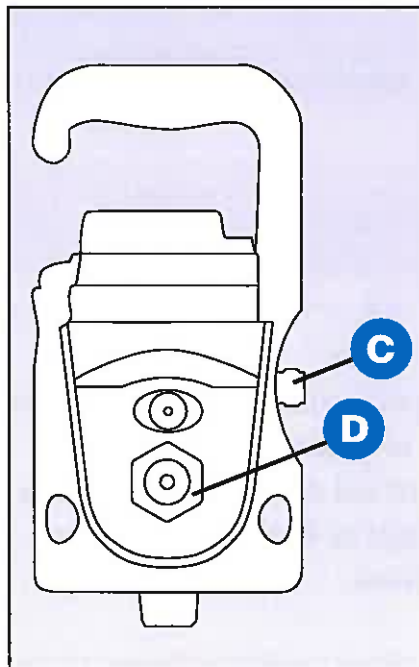
Safety Precautions

Front view



A Outlet Port
B Flow-adjusting Knob

Back view



C Auxiliary Port
D Fill Port (Never Remove Cap)

Before using or working with this system, please read and understand the following safety information.

While it is very simple to use the *Grab 'n Go* system, remember that pressurized oxygen vigorously accelerates combustion. Combustibles that burn in air burn hotter and brighter in oxygen. Oil, grease, and other hydrocarbons can ignite violently (explode) in the presence of high-pressure oxygen.

The Praxair *Grab 'n Go* Portable Medical Oxygen System is equipped with a pressure-reducing regulator. This unit also contains several safety features that relieve pressure if a malfunction occurs. Continuous or intermittent escape of gas from the regulator indicates a malfunction or a leak.

Here are some other precautions to keep in mind:

- Use the unit only with equipment designed and cleaned for oxygen service.
- Keep the cylinder, regulator, and connections free of oil, grease, and other hydrocarbons. **Do not lubricate!**
- Do not smoke or allow open flames in areas where oxygen is stored or in use.
- Never wrap the unit in bedcovers or carry it under clothing.
- Always turn the unit off when not in use so that no oxygen flows from the outlet.
- The unit is designed for use above 32°F (0°C), low temperature or ice may delay or prevent the flow of oxygen.
- Do not use or store the cylinder near heat or flame. Do not expose the cylinder to direct sunlight for long periods or to any condition where temperature exceeds 130°F (54.5°C).
- Keep the unit out of reach of children.
- Store the unit indoors to protect it from temperature extremes and the elements.
- Clean cover with a clean lint-free cloth and water. **DO NOT** use flammable liquids such as alcohol to clean the unit.

- This device is **ONLY** for use by, or on the order of, a physician. Do not activate the unit unless you have been trained in its proper functioning or are under competent supervision.
- Avoid extended use of this unit except on the advice of a physician.
- The cylinder contains high pressure U.S.P. (United States Pharmacopoeia) grade oxygen.
- Never attempt to service the *Grab 'n Go* system or to remove the regulator from the cylinder. If the system malfunctions or develops a leak, turn it off immediately, place it outdoors, mark the unit as unserviceable and notify your Praxair representative.
- Never use the outlet port (A) when the Auxiliary port (C) is in operation.
- **DO NOT** take a steel cylinder into an MRI environment.

Equipment Storage, Handling, and Setup

The *Grab 'n Go* system operates **ONLY when positioned at the flow rates marked on the flow-adjusting knob. If the Intended flow is not marked on the flow-adjusting knob, **DO NOT ATTEMPT TO USE THE SYSTEM**. Consult your physician for a different flow rate or a different system.**

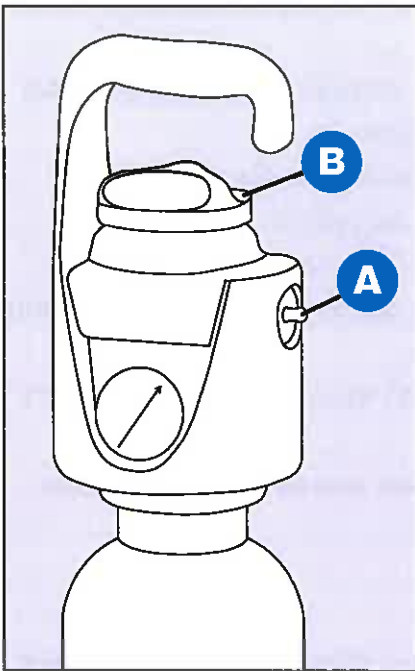
1. Store *Grab 'n Go* systems indoors in a safe area where they will not fall over or be damaged by falling objects. Never store a system in an elevated location because a fall could seriously damage the regulator or cylinder. If a system is dropped or knocked over, check to see that components are not damaged and connections remain secure.

2. Inspect the regulator's barbed outlet port for physical damage, traces of dust, dirt, oil, or grease. If you spot oil or grease or other contaminants or if you suspect their presence, do not use the unit. Do not attempt to remove the oil or grease or repair the unit. Notify Praxair or Praxair's representative immediately and have the system serviced by qualified personnel.

2B. If your unit is equipped with the auxiliary port, inspect the regulator's barbed outlet port (A) and auxiliary port (C) for physical damage, traces of dust, dirt, oil or grease. If you spot oil, grease or other contaminants or if you suspect their presence, do not use the unit. Do not attempt to repair or remove the oil or grease from the unit. Notify Praxair or Praxair's representative immediately and have the system serviced by qualified personnel.

3. Handle systems safely. Portable oxygen therapy systems are inherently subject to greater abuse than are stationary units. Avoid jarring or dropping the system.

Front view



A Outlet Port
B Flow-adjusting Knob

4. Attach the oxygen supply tubing to the regulator's barbed outlet port (A) located in the access hole on the side of the housing. Rotate flow-adjusting knob (B) clockwise to one of the numbered click stops. Always verify flow with the patient's end of the tubing before applying. Make sure oxygen tubing is never folded, kinked or crushed.

Intended use

The *Grab 'n Go* system is to be used **only** at the marked liters per minute (L/min) flow rate settings. If settings other than those marked on the knob are required, do not use the *Grab 'n Go* system. Consult your physician for different flow rate or a different system. The *Grab 'n Go Vantage* unit should be selected when a flow rate of 25 L/min is required or a 50 psi auxiliary outlet to supply a demand valve or portable ventilator is required.

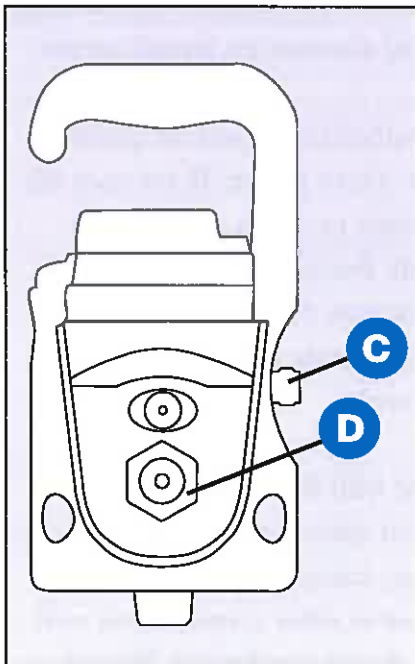
Directions for use

1. Check the pressure gauge to ensure presence of adequate oxygen contents. To turn the system on, rotate the flow-adjusting knob (B) clockwise from the OFF position. The knob has a limited range of travel, about 300 degrees. Click stops mark each flow rate.

Caution: Never force the flow-adjusting knob.

Warning: Do not use between the clickstops at marked flow-rate settings. If settings other than those marked on the knob are needed, do not use the *Grab 'n Go* system. Always verify flow with the patient's end of the tubing before applying.

Back view



C Auxiliary Port
D Fill Port (Never Remove Cap)



Giving caregivers an even greater hand.

2. To turn the system off, turn flow-adjusting knob (B) counterclockwise to the OFF position. If you still hear a hissing sound, do not try to force the knob past the positive stop at the OFF position. Hissing indicates a leak. Remove the system from service, place it outdoors, mark the unit as unserviceable and notify your Praxair representative.

Warning: Do not attempt to use or repair a leaking system.

3. When using the auxiliary port on the *Grab 'n Go Vantage* unit, connect the oxygen supply hose to the auxiliary port DISS threaded connector. Rotate the flow-adjusting knob (B) clockwise until the “AUX” setting is visible in the flow setting window and verify flow at the device. Never attach a patient directly to the auxiliary port (C). The auxiliary port’s (C) flow rate is not regulated and excessive flow to the patient will result. When the flow-adjusting knob (B) is in the “AUX” setting, no oxygen is supplied to the outlet port (A).

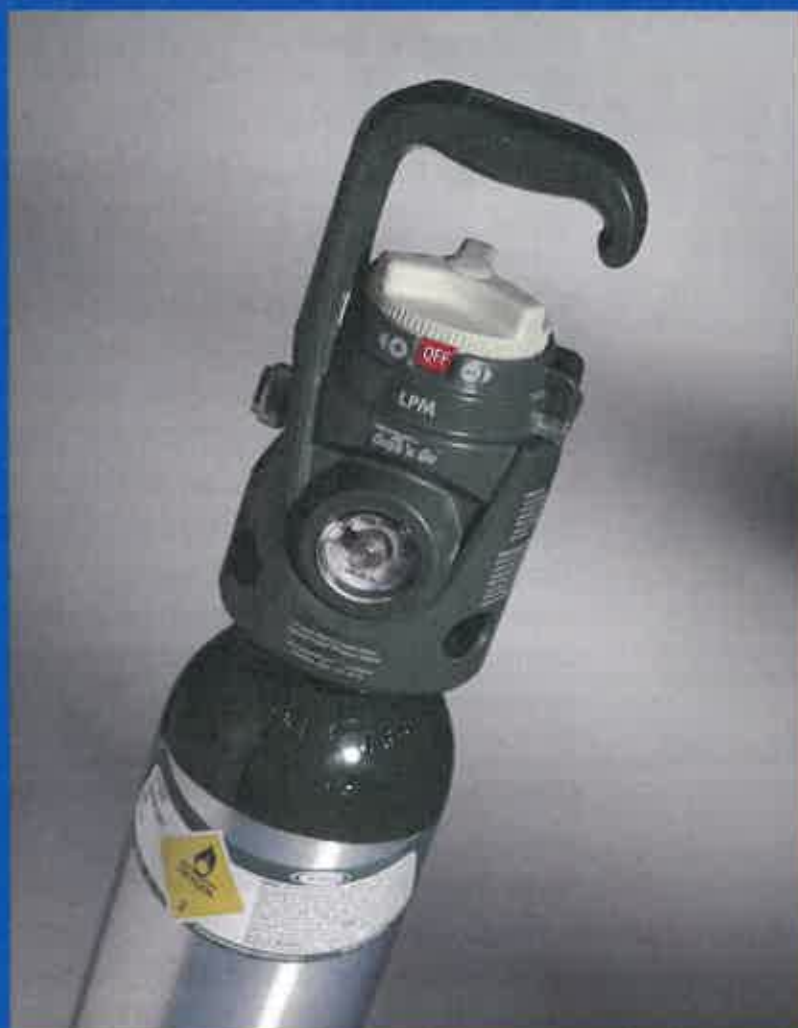
Caution: Never attempt to repair any part of the regulator. Any needed system repairs must be performed by an authorized Praxair representative.

Unit replacement and refill

The high-pressure gauge visible through the plastic housing gives a continuous reading of cylinder pressure, regardless of whether oxygen is flowing or not. To ensure proper functioning and an adequate supply of oxygen, replace the system whenever the gauge indicates 500 pounds per square inch (psi) (3,500 kPa) or less.

The Praxair *Grab 'n Go III* and *Grab 'n Go Vantage* systems must be refilled with USP grade medical oxygen by Praxair or an authorized Praxair dealer.

Caution: Never attempt to remove the large cap (D) from the regulator’s fill port.



All in one... the one for all your portable medical oxygen needs.

**How to setup and use the *Grab 'n Go*
Advanced Respiratory System.**

Recommended Reference Publications

Publication Title	Publication No.	Source
Safety Precautions and Emergency Response Planning Awareness: Oxygen, Nitrogen, Argon, Helium, Carbon Dioxide, Hydrogen, Fuel Gases	P-3499	Praxair, Inc. P.O. Box 44 Tonawanda, NY 14150-7891 Phone: 1-800-PRAXAIR
Condensed Safety Information, Compressed Gases and Cryogenic Liquids	P-12-237	
Guidelines for Handling Compressed Gas Cylinders and Cryogenic Liquid Containers	P-14-153	
Material Safety Data Sheet - Oxygen	P-4638	Praxair supplier, sales representative, the Call Center (1-800-PRAXAIR), our Web site (http://www.praxair.com), or write the Praxair Tonawanda, NY address above.
Additional copies of this publication	P-15-228 B	
Safe Handling of Compressed Gases in Containers	CGA P-1	Compressed Gas Association 1725 Jefferson Davis Highway Suite 1004 Arlington, VA 22202-4102 Phone: (703) 412-0900
Accident Prevention in Oxygen-Rich and Oxygen-Deficient Atmospheres	CGA P-14	



All information referred to and/or included in the manual is current as of the original issue date of this manual. Praxair, Inc. makes no warranty or representation with respect to the accuracy of the information outside Praxair, Inc., nor does Praxair, Inc. assume responsibility for any injury or damage that may result, directly or indirectly, from the use of such information.

Praxair, the *Flowing Airstream* design, *Grab 'n Go* and *Grab 'n Go Vantage* are trademarks or registered trademarks of Praxair Technology, Inc. in the United States and other countries.

Manufactured for Praxair by Western Medica, Westlake, OH 44145.

Praxair Healthcare Services
39 Old Ridgebury Road
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P-15-228 C

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U.S. Food and Drug Administration
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Archived Content

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Praxair Inc., Grab 'n Go Vantage Portable Oxygen Cylinder Units

Recall Class: Class I

Date Recall Initiated: November 16, 2012

Product(s): Grab 'n Go Vantage Portable Oxygen Cylinder unit

Catalog Number:

OX-MAEGNGVNTG (US and Canada)

WESPRX-9500 (Global)

Range of manufacturing and distribution dates: Products were manufactured and distributed from June 17, 2009 to November 16, 2012.

Use: The Grab 'n Go Vantage Portable Oxygen Cylinder unit consists of an oxygen container and pressure regulator (valve). The pressure regulator (valve) is intended for medical purposes and is used to convert a medical gas pressure from a high variable pressure to a lower, more constant working pressure.

Recalling Firm:

Praxair Inc.
39 Old Ridgebury Road
Danbury, CT 06817-0001

Reason for Recall: Praxair Inc. has initiated a voluntary recall of its Grab 'n Go Vantage Portable Oxygen Cylinder unit because fires may occur inside the cylinder unit causing the wall of the unit to break and release oxygen. The fires are self extinguishing and may be caused by physical impact such as dropping or knocking over the cylinder unit. If a fire occurs, users may experience burns or a lack of oxygen (hypoxia). This product may cause serious adverse health consequences, including death.

Public Contact: Customers with questions can call Praxair, Inc. at 877-772-9247, Monday through Friday from 8am-5pm EST.

FDA District: New England District Office

FDA Comments: On November 26, 2012, Praxair, Inc. sent "Medical Device Correction" letters to customers and distributors describing the product, problem, and actions to be taken. Customers were also reminded of the following operating instructions:

- Store the Grab'n Go systems indoors in a safe area,
- Never store a system in an elevated location, and
- Handle systems safely and avoid jarring or dropping the system.

Customers were also instructed to send Grab'n Go Vantage Portable Oxygen Cylinder units to Praxair, Inc. for servicing and refilling after use in accordance with their normal processes.

Class I recalls are the most serious type of recall and involve situations in which there is a reasonable probability that use of these products will cause serious adverse health consequences or death.

Health care professionals and consumers may report adverse reactions or quality problems they experienced using these products to **MedWatch: The FDA Safety Information and Adverse Event Reporting Program** (<http://www.fda.gov/Safety/MedWatch/HowToReport/default.htm>) either online, by regular mail or by FAX.

Additional Links:

1. **Praxair Inc. Press Release** (<http://www.praxair.com/praxair.nsf/AllContent/C020A08D01705E8485257ADA0061CA3A?OpenDocument>) 
(<http://www.fda.gov/AboutFDA/AboutThisWebsite/WebsitePolicies/Disclaimers/default.htm>)
2. **Grab'n Go Operating Instructions** (<http://www.praxair.com/GNGInstructions>) 
(<http://www.fda.gov/AboutFDA/AboutThisWebsite/WebsitePolicies/Disclaimers/default.htm>)

More in Medical Device Safety
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[Device Postmarket Surveillance \(/MedicalDevices/Safety/CDRHPostmarketSurveillance/default.htm\)](/MedicalDevices/Safety/CDRHPostmarketSurveillance/default.htm)



[Medical Device Reporting \(MDR\) \(/MedicalDevices/Safety/ReportaProblem/default.htm\)](/MedicalDevices/Safety/ReportaProblem/default.htm)



[MedSun: Medical Product Safety Network \(/MedicalDevices/Safety/MedSunMedicalProductSafetyNetwork/default.htm\)](/MedicalDevices/Safety/MedSunMedicalProductSafetyNetwork/default.htm)



[Emergency Situations \(Medical Devices\) \(/MedicalDevices/Safety/EmergencySituations/default.htm\)](/MedicalDevices/Safety/EmergencySituations/default.htm)



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Safety

Recall -- Firm Press Release

FDA posts press releases and other notices of recalls and market withdrawals from the firms involved as a service to consumers, the media, and other interested parties. FDA does not endorse either the product or the company.

Praxair, Inc. has Initiated a Voluntary Recall Affecting Grab 'n Go® Vantage Portable Oxygen Cylinder Units

Contact:

Consumer:
630-320-4452

Media:
Sue Neumann
203-837-2609
sue_neumann@praxair.com

FOR IMMEDIATE RELEASE - December 20, 2012 - Praxair, Inc. (NYSE: PX) today announced that it is in the process of conducting a voluntary recall involving *Grab 'n Go* Vantage portable oxygen cylinder units. Praxair is making a minor modification to the affected units as they come in for routine servicing and filling. Praxair has already communicated with its customers and the U.S. Food and Drug Administration.

Praxair initiated this field correction after becoming aware of a few isolated incidents of ignition inside *Grab 'n Go* oxygen cylinders that had been knocked over and subject to significant physical impact. Based on the preliminary results of Praxair's review of these few incidents, it appears that *Grab 'n Go* units fitted with certain equipment (a type of o-ring) may be more prone to this type of incident.

Out of an abundance of caution to protect patient safety, Praxair has been making a minor modification to affected units. As units come in for routine servicing and filling, the unit is being inspected and, if necessary, the o-ring that sits between the valve and the cylinder is being replaced. To date, over 50% of the units in the United States already have been modified. Modified units have a decal with a "T" affixed to their green shroud.

Praxair believes that the affected *Grab 'n Go* units may be used safely if proper handling procedures are followed. It is important to practice safe storage and handling practices for the *Grab 'n Go* cylinders to protect the cylinder from dropping, knocking over, or other significant physical impact.

Customers with questions may contact their sales representative or Thelma Brantley (e-mail: Thelma_Brantley@Praxair.com; phone 630-320-4452).

Praxair, Inc. is the largest industrial gases company in North and South America, and one of the largest worldwide, with 2011 sales of \$11 billion. The company produces, sells and distributes atmospheric, process and specialty gases, and high-performance surface coatings. Praxair products, services and technologies are making our planet more productive by bringing efficiency and environmental benefits to a wide variety of industries, including aerospace, chemicals, food and beverage, electronics, energy, healthcare, manufacturing, metals and others. More information on Praxair is available on the Internet at www.praxair.com²⁰.

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[Photo: Product Labels](#)²³

Recalled Product Photos Are Also Available on FDA's [Flickr Photostream](#).²⁴

Page Last Updated: 12/21/2012

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20. <http://www.praxair.com>
21. [/AboutFDA/ContactFDA/StayInformed/RSSFeeds/Recalls/rss.xml](#)
22. [/AboutFDA/ContactFDA/StayInformed/RSSFeeds/default.htm](#)
23. [/Safety/Recalls/ucm333199.htm](#)
24. <http://www.flickr.com/photos/fdaphotos/sets/72157624901041809/>



Praxair, Inc. Has Initiated a Voluntary Recall Affecting *Grab'n Go*[®] Vantage Portable Oxygen Cylinder Units

PRAXAIR, INC. HAS INITIATED A VOLUNTARY RECALL AFFECTING *GRAB'N GO*[®] VANTAGE PORTABLE OXYGEN CYLINDER UNITS

December 20, 2012

DANBURY, Conn., December 20, 2012 — Praxair, Inc. (NYSE: PX) today announced that it is in the process of conducting a voluntary recall involving *Grab 'n Go* Vantage portable oxygen cylinder units. Praxair is making a minor modification to the affected units as they come in for routine servicing and filling. Praxair has already communicated with its customers and the U.S. Food and Drug Administration.

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Customers with questions may contact their sales representative or Thelma Brantley (e-mail: Thelma_Brantley@Praxair.com; phone 630-320-4452).

Praxair, Inc. is the largest industrial gases company in North and South America, and one of the largest worldwide, with 2011 sales of \$11 billion. The company produces, sells and distributes atmospheric, process and specialty gases, and high-performance surface coatings. Praxair products, services and technologies are making our planet more productive by bringing efficiency and environmental benefits to a wide variety of industries, including aerospace, chemicals, food and beverage, electronics, energy, healthcare, manufacturing, metals and others. More information on Praxair is available on the Internet at www.praxair.com.

Contact

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Contact Us

1-800-PRAXAIR

(1-800-772-9247)

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Appendix C - FTIR Sampling Description and Log

Each FTIR spectrum was obtained using a PIKE Technologies MIRacle™ attenuated total reflectance (ATR) sampling accessory and a Bruker Tensor 27 Fourier Transform Infrared Spectrometer. The spectral data was collected with a Germanium sampling crystal with 32 scans at 4cm^{-1} resolution. The data was plotted in ATR units versus frequency (cm^{-1}) format.

Sampling methods included dry swab, solvent swabbing, direct sampling, and solvent extraction. Any samples taken using solvent were done so with Asahiklin 225 (AK225), also known as HCFC 225, because of its ability to dissolve perfluoropolyether lubricants and hydrocarbon oils off of surfaces with minimal exposure time. AK 225 is known to have good compatibility with many materials and is considered to be 100% volatile according to the material safety data sheet.

Dry swabs were applied to the sample surface and moved around the desired area under slight pressure in order to help lift residues off the surface and onto the dry swab. The dry swab was placed into a clean sample vial. The dry swab was then used to transfer the sample from the swab onto the ATR FTIR crystal to obtain a spectrum. The same technique was used when a solvent swab was applied except that the swab was immersed in solvent. The solvent swab was placed in a vial with approximately 5 ml of solvent. The vial was then placed into a sonication bath and sonicated for 180 seconds to remove insoluble solids from the swab as well as encourage residues collected on the swab to dissolve into the solvent. The solvent was then allowed to evaporate to approximately 1 ml to concentrate the sample. A small aliquot was then taken by clean pipette and placed on the ATR FTIR crystal where the rest of the remaining AK225 solvent was allowed to evaporate. After the solvent was completely evaporated a spectrum was obtained. Direct samples were placed directly onto the ATR FTIR crystal and a spectrum was obtained.

Solvent extraction samples were collected by directly placing solvent on an area or rinsing an area that was being sampled with solvent transferring it by clean pipette. The solvent samples were collected into clean sample jars. The samples were then allowed to evaporate to approximately 1 ml of solvent in order to concentrate the sample. A small aliquot was then taken by clean pipette and placed on the ATR FTIR crystal where the rest of the remaining AK225 solvent was allowed to evaporate. After the solvent was completely evaporated a spectrum was obtained.

WHA performed library searches to match the functionality observed in the FTIR spectra and has attempted to identify the best match of the spectra to both the standard libraries and WHA's internal libraries. The "WHA Interpretation" provided in the table below indicates an interpretation consistent with the functionality of the material indicated based on the best match obtained and based on unique identifying stretches that are characteristic of specific bonds in molecules.

Sample ID	Sample Type	Solvent	Description / location	WHA Interpretation
S1	Solvent Swab	AK225	Solvent swab of O-ring surface valve flange thread interface	No Signal
S2	Solvent Rinse	AK225	Wash of threads nearest the valve flange (half thread length)	Weak HC in 2700-3000 cm^{-1} range /Weak Carbonyl at 1742 cm^{-1} / Krytox GPL
S3	Solvent Rinse	AK225	Rinse of threads farthest from valve flange (half thread length) bottom face included in rinse	HC in 2700-3000 cm^{-1} range /Carbonyl at 1734 cm^{-1} /Weak Krytox GPL
S4	Solvent Swab	AK225	Solvent swab of threaded area valve threads	No Signal



Sample ID	Sample Type	Solvent	Description / location	WHA Interpretation
S5	Scrape	None	Scrape of purple residue in internal surface of filter	No Signal
S6	Solvent Rinse	AK225	Solvent rinse of O-ring	HC in 2700-3000 cm^{-1} range /Carbonyl at 1733 cm^{-1} /Weak Krytox GPL
S7	Direct Sample	None	Direct sample of O-ring	PTFE material
S8	Solvent Swab	AK225	Solvent swab of fill port conical area (non-wetted region)	HC in 2700-3000 cm^{-1} range /Weak Krytox GPL
S9	Solvent Swab	AK225	Solvent swab of fill port inlet (wetted)	HC in 2700-3000 cm^{-1} range /Weak Krytox GPL
S10	Solvent Rinse	AK225	Fill port filter rinse	Weak HC in 2700-3000 cm^{-1} rang / Krytox GPL
S11	Solvent Rinse	AK225	Fill port poppet O-ring rinse	HC in 2700-3000 cm^{-1} range/Krytox GPL
S12	Direct Sample	AK225	Direct sample of fill port poppet O-ring	FKM Elastomer
S13	Solvent Rinse	AK225	Solvent rinse of the fill port poppet and spring	HC in 2700-3000 cm^{-1} range/Carbonyl at 1732 cm^{-1} /Krytox GPL
S14	Solvent Rinse	AK225	Solvent rinse of fill port retainer O-ring	HC in 2700-3000 cm^{-1} range/Carbonyl at 1730 cm^{-1} /Krytox GPL
S15	Direct Sample	None	Direct sample of fill port O-ring	FKM Elastomer
S16	Solvent Swab	AK225	Solvent swab of external facing side of burst disc gasket	No Signal
S17	Solvent Swab	AK225	Solvent swab of internal facing side of burst disc gasket	No Signal
S18	Direct Sample	AK225	Direct sample of burst disk gasket material	Polyamide material
S19	Solvent Swab	AK225	Solvent swab of internal side of burst disc	No Signal
S20	Solvent Swab	AK225	Solvent swab of external side of burst disc	Weak Krytox GPL
S21	Solvent Swab	AK225	Solvent swab of pressure gauge up to snubber	HC in 2700-3000 cm^{-1} range Carbonyl at 1732 cm^{-1} /Weak Krytox GPL
S22	Solvent Swab	AK225	Solvent swab of pressure gauge port (VIPR side port) small diameter port	No Signal
S23	Solvent Swab	AK225	Solvent swab of regulator seat	Weak Krytox GPL
S24	Dry Swab	None	Dry swab of regulator piston O-ring (small)	Krytox GPL
S25	Dry Swab	None	Dry swab of regulator piston lubricant	Krytox GPL
S26	Dry Swab	Rinse	Dry swab of regulator piston O-ring (small)	Krytox GPL
S27	Solvent Swab	AK225	Solvent swab of black residue on large end of regulator	Krytox GPL
S28	Solvent Swab	AK225	Solvent swab of black residue on regulator piston housing	Krytox GPL
S29	Dry Swab	None	Dry swab of lubricant on large end of piston	Krytox GPL
S30	Dry Swab	None	Dry swab of lubricant on regulator piston housing	Krytox GPL
S31	Solvent Swab	AK225	Solvent swab of regulator piston retainer face	Krytox GPL/Carbonyl at 1727 cm^{-1}

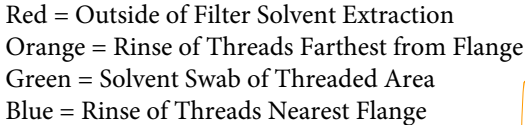


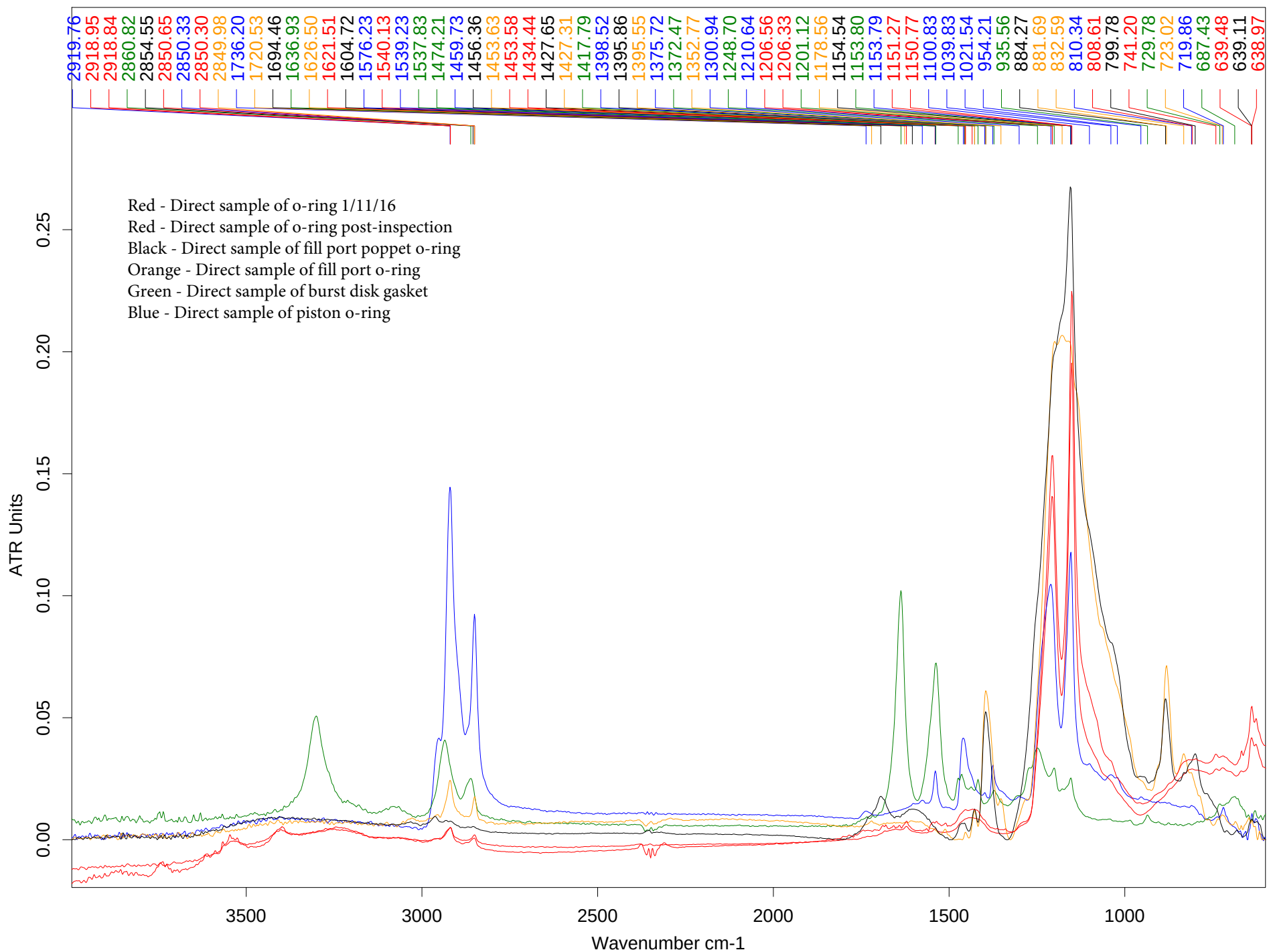
Sample ID	Sample Type	Solvent	Description / location	WHA Interpretation
S32	Dry Swab	None	Dry swab of regulator housing O-ring	Krytox GPL/Weak HC in 2700-3000 cm^{-1} range/Carbonyl at 1726 cm^{-1}
S33	Solvent Swab	AK225	Solvent swab of surface around the regulator orifice	Krytox GPL/HC in 2700-3000 cm^{-1} range/Carbonyl at 1726 cm^{-1}
S34	Solvent Rinse	AK225	Solvent rinse through of regulator filter	Krytox GPL
S35	Direct Sample	None	Direct sample of regulator filter lubricant	Krytox GPL
S36	Dry Swab	None	Dry swab of lubricant d/s of the regulator filter	Krytox GPL
S37	Dry Swab	None	Dry swab of pressure relief valve	Krytox GPL
S38	Solvent Swab	AK225	Solvent swab of pressure relief valve conical surface	Krytox GPL/Weak HC in 2700-3000 cm^{-1} range /Carbonyl at 1725 cm^{-1}
S39	Solvent Swab	AK225	Solvent swab of lower surface of flowhead body	Krytox GPL/Weak HC in 2700-3000 cm^{-1} range
S40	Solvent Swab	AK225	Solvent swab of upper surface of flowhead body	Weak Krytox GPL
S41	Solvent Swab	AK225	Solvent swab of cylinder filter port	Krytox GPL/Weak HC in 2700-3000 cm^{-1} range/Carbonyl at 1726 cm^{-1}
S42	Solvent Swab	AK225	Solvent swab the internal fill valve poppet bore	Krytox GPL/Weak HC in 2700-3000 cm^{-1} range/Carbonyl at 1728 cm^{-1}
S43	Dry Swab	None	Dry swab of lower end soft goods of high pressure valve	Krytox GPL/Weak HC in 2700-3000 cm^{-1} range/Carbonyl at 1726 cm^{-1}
S44	Dry Swab	None	Dry swab of upper end of high pressure valve	Krytox GPL / Weak HC at 2700-3000 cm^{-1} range
S45	Solvent Rinse	AK225	Outside of filter rinse	Krytox GPL /HC in 2700-3000 cm^{-1} range /Carbonyl at 1737 cm^{-1}
S46	Solvent Rinse	AK225	Inside of filter rinse	Krytox GPL/Weak HC in 2700-3000 cm^{-1} range
S47	Direct Sample	None	Direct sample of piston O-ring (small)	EPDM Elastomer/PTFE material NOTE: The PTFE observed is similar to that in the Krytox mixture. This area was sampled previously by dry swab and solvent swab. Those two samples would be expected to remove PFPE oil in Krytox and leave behind some of the PTFE filler.
S48	Dry Swab	None	Dry swab of regulator piston O-ring (large)	Krytox GPL
S49	Solvent Rinse	AK225	Solvent rinse sample of regulator piston O-ring (large)	Krytox GPL
S50	Direct Sample	None	Direct sample of regulator piston O-ring (large)	FKM Elastomer
S51	Direct Sample	None	Regulator Piston Seat Direct Sample Direct	PTFE Material
S52	Direct Sample	None	Shuttle Valve O-Ring 1 Black Direct Sample	FKM Elastomer/Krytox GPL
S53	Direct Sample	None	Shuttle Valve O-Ring 1 White Direct Sample	Krytox GPL/PTFE Material
S54	Direct Sample	None	Shuttle Valve O-Ring 2 Black Direct Sample	Krytox GPL/FKM Elastomer
S55	Direct Sample	None	Regulator Inlet O-Ring Direct Sample	Krytox GPL/FKM Elastomer

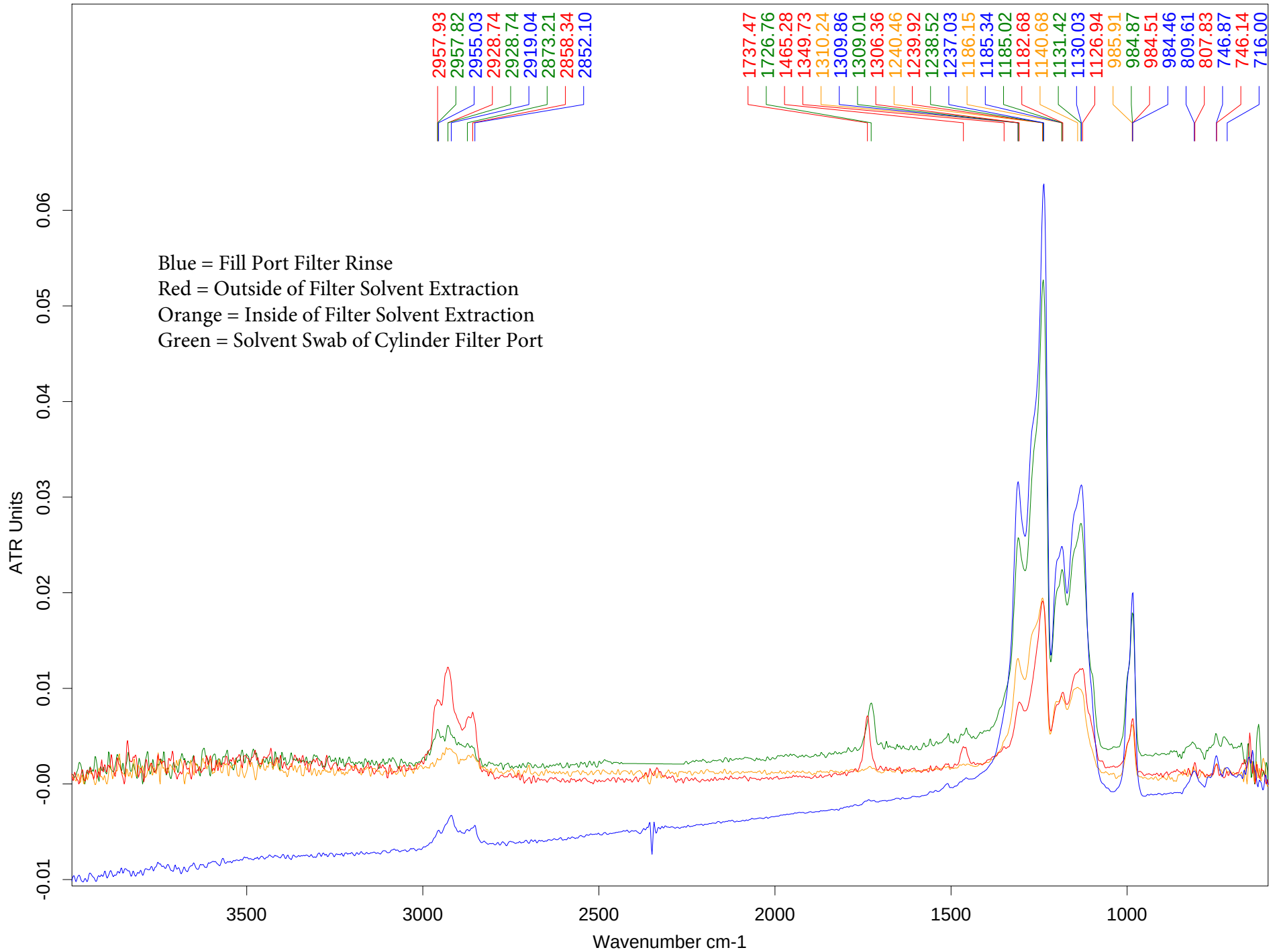


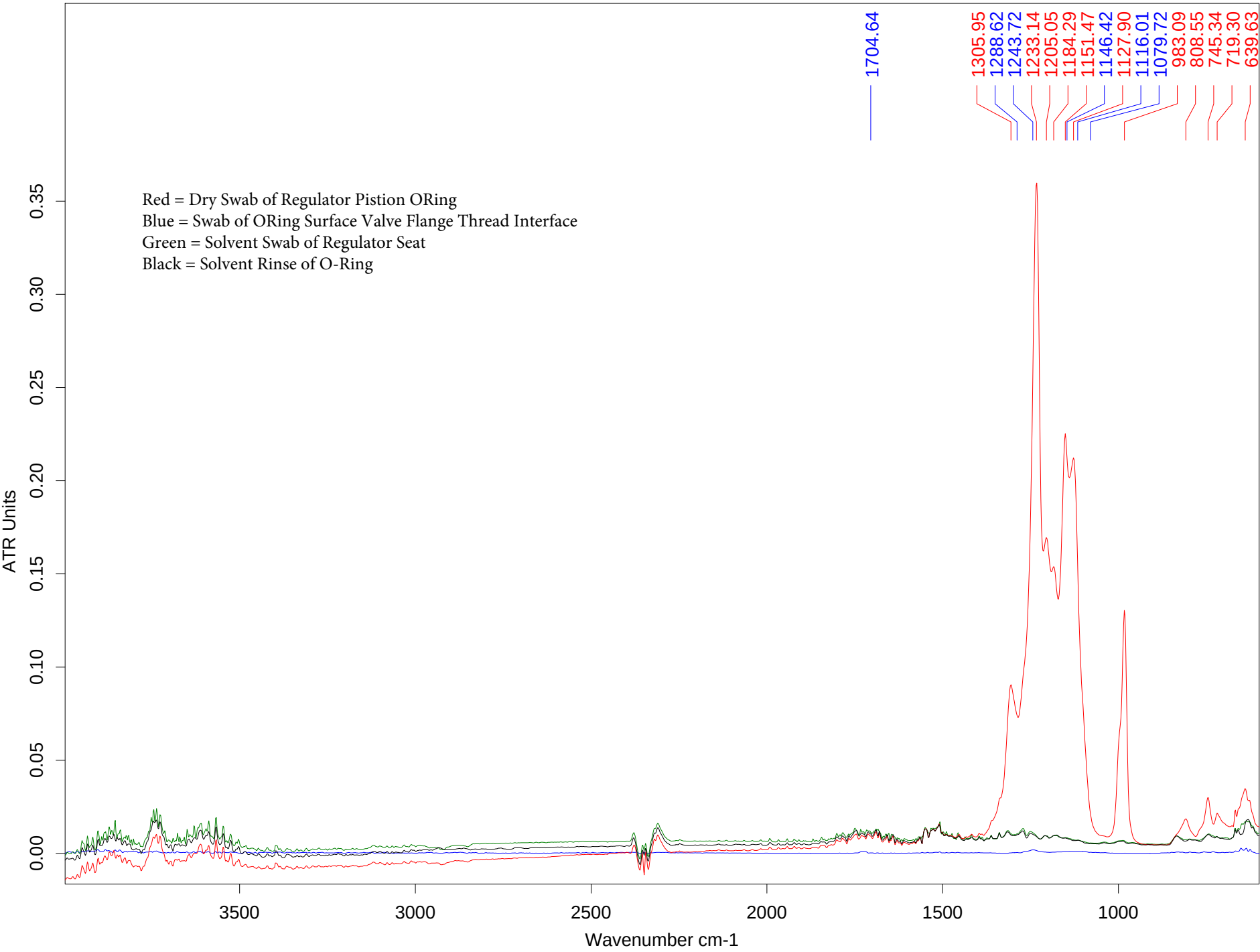
Sample ID	Sample Type	Solvent	Description / location	WHA Interpretation
S56	Direct Sample	None	Relief Seat Seal O-Ring Direct Sample	EPDM Elastomer

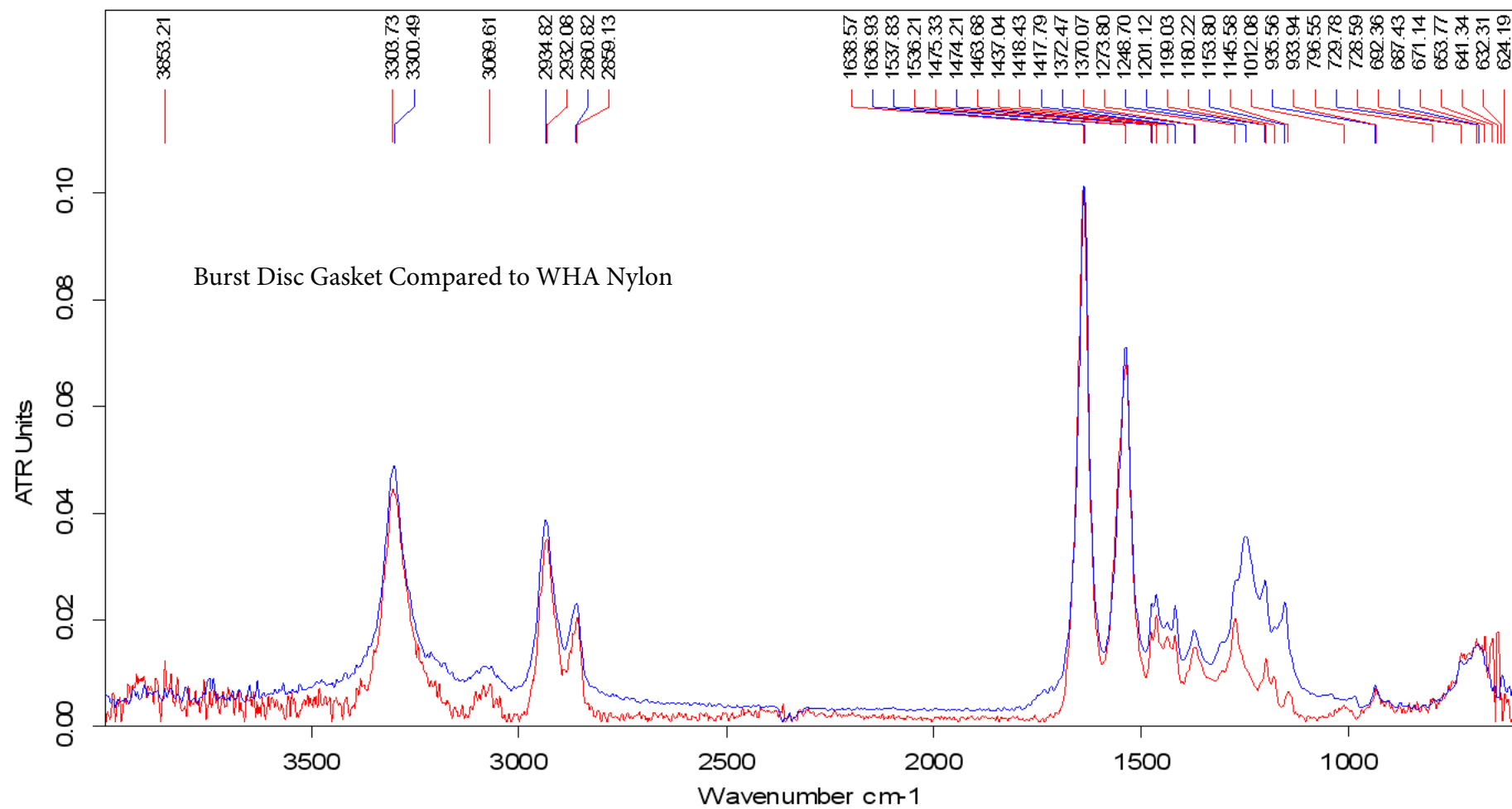




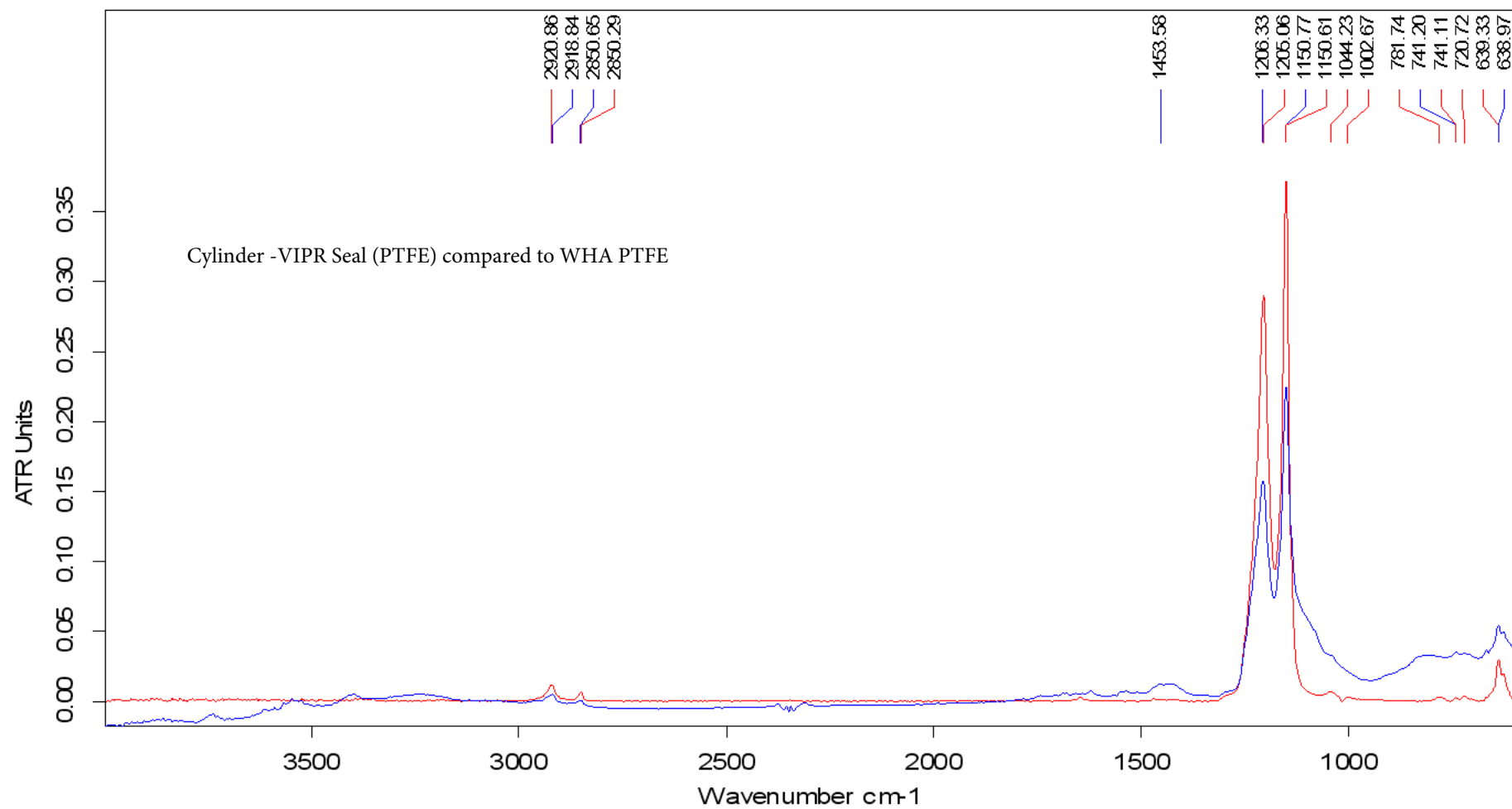




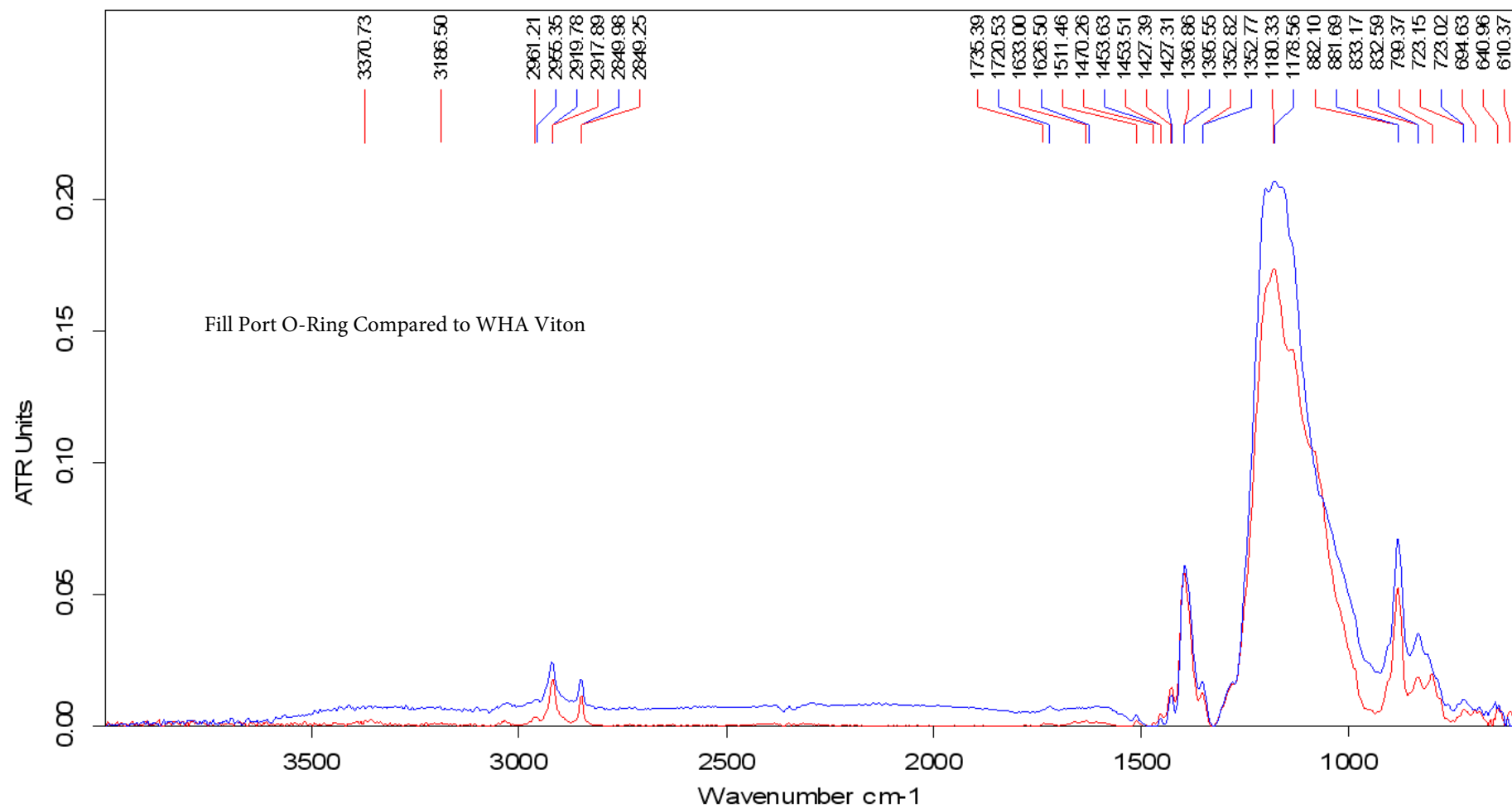




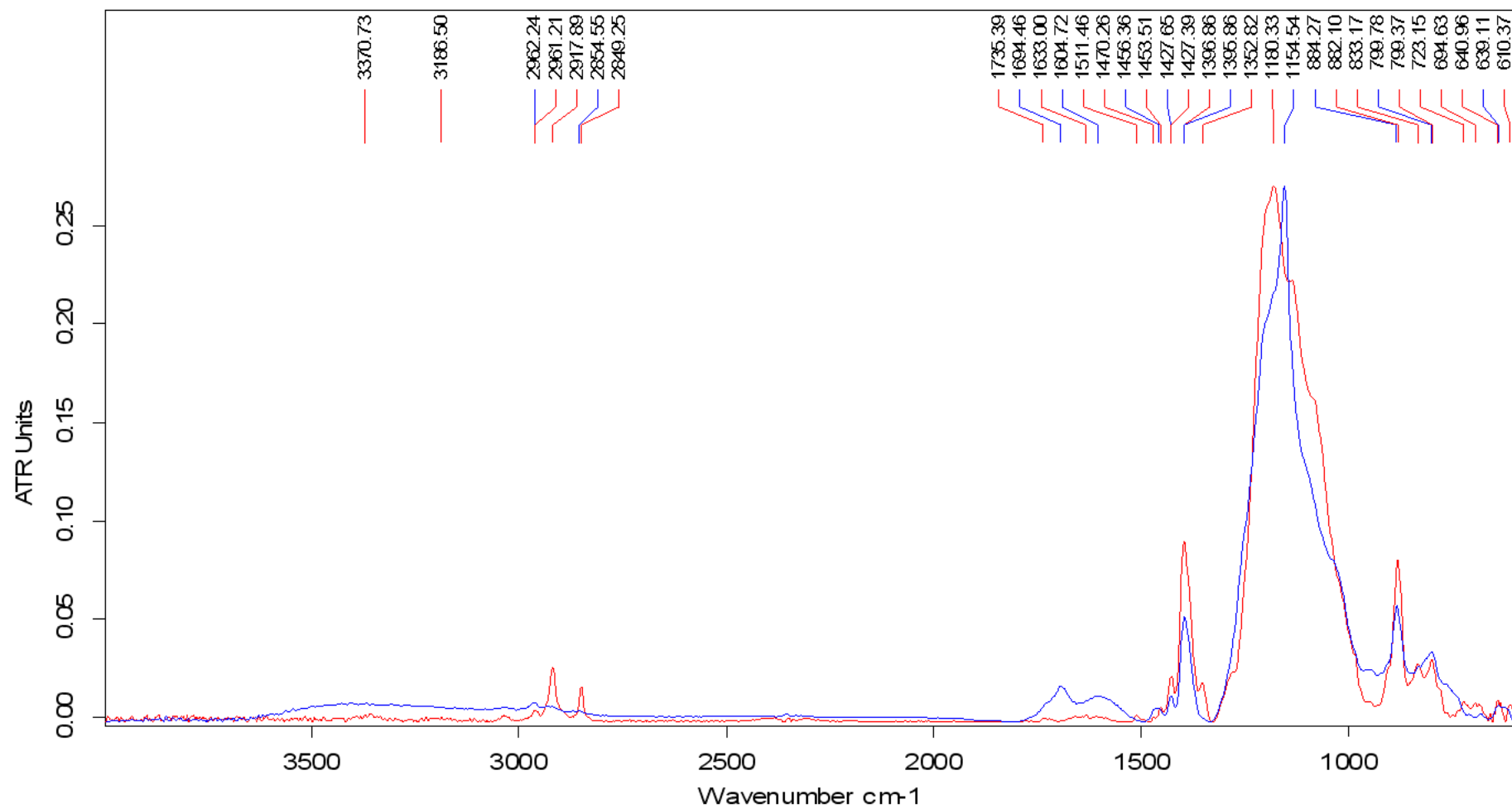
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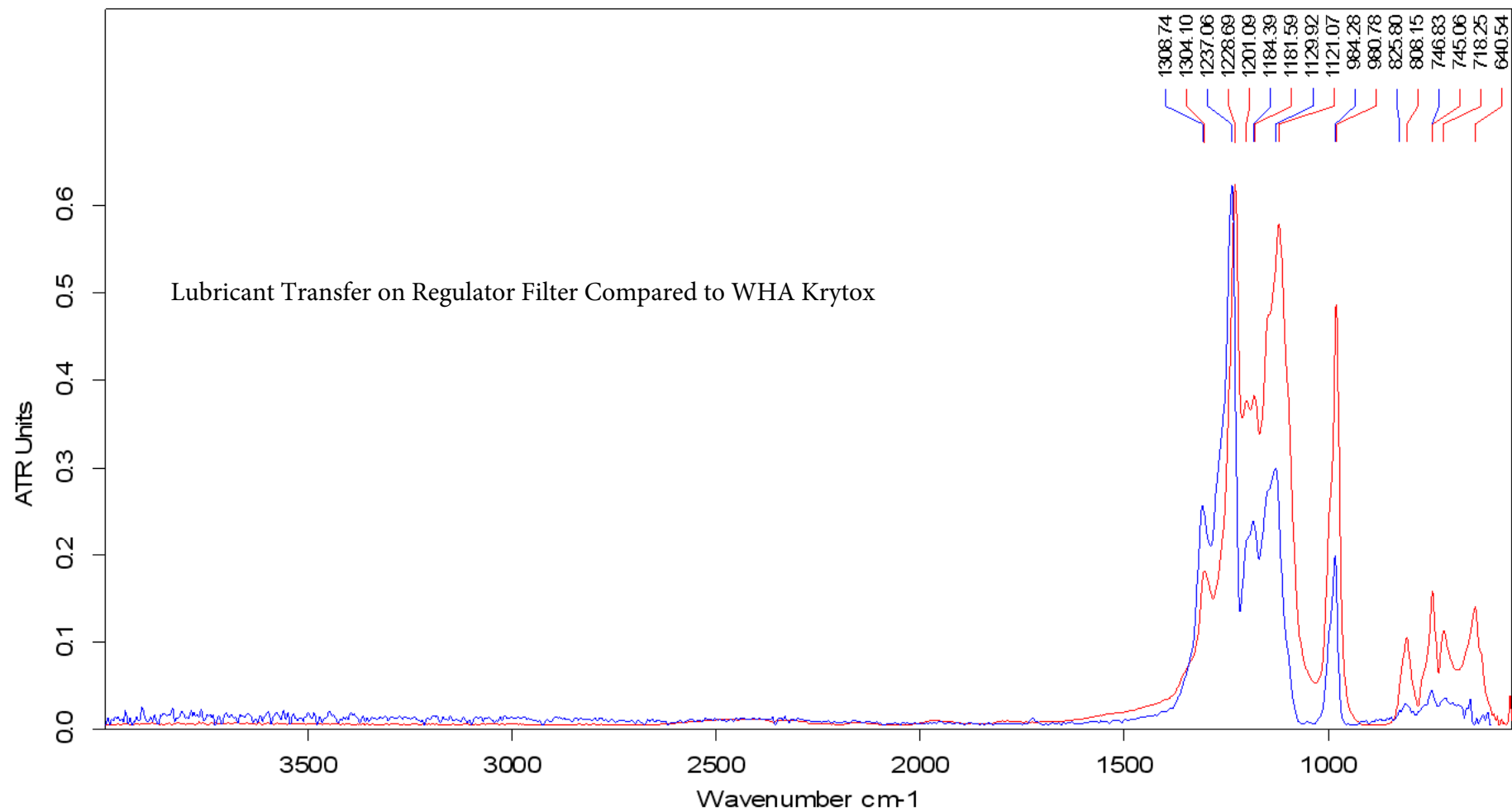
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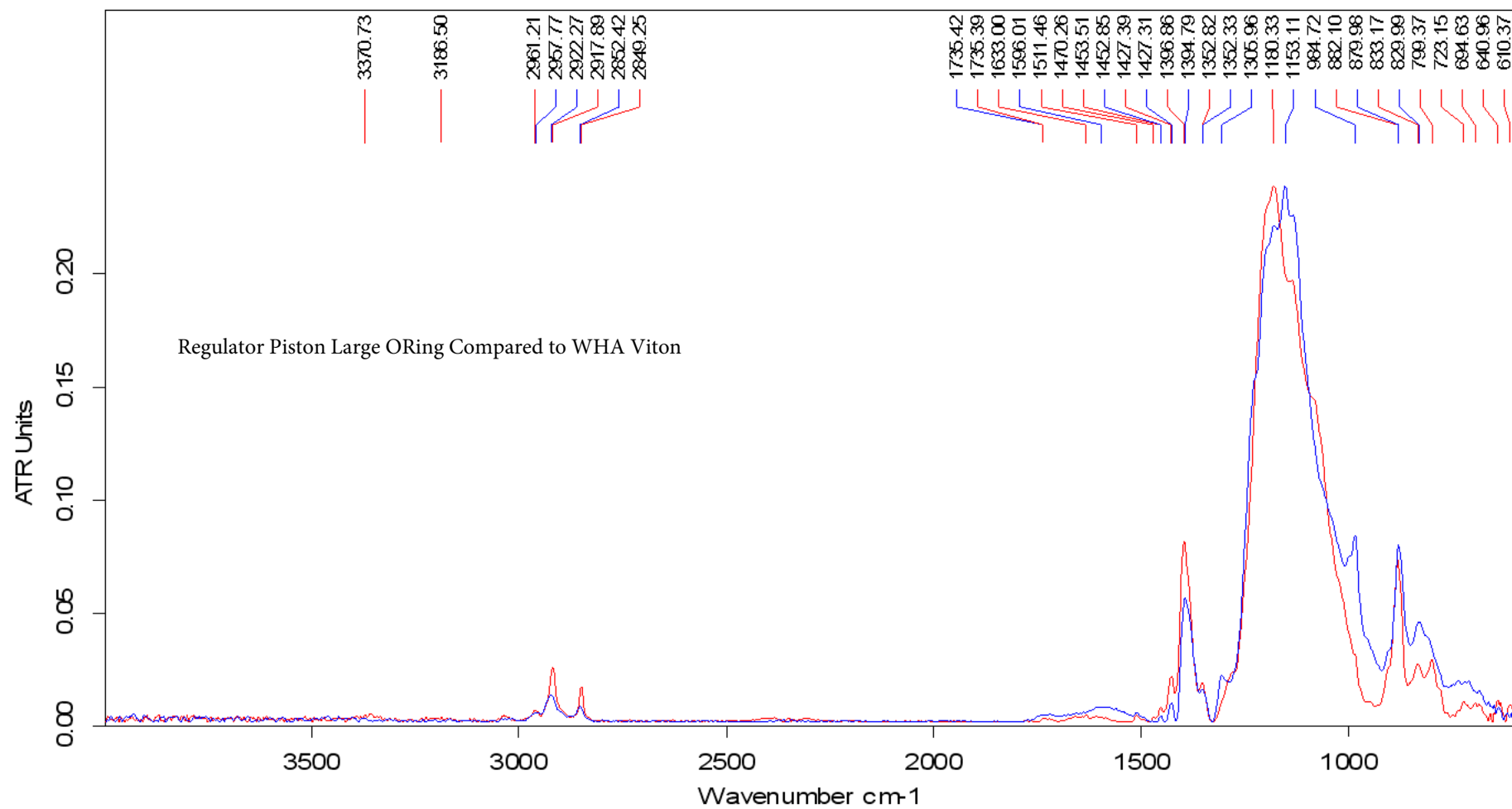
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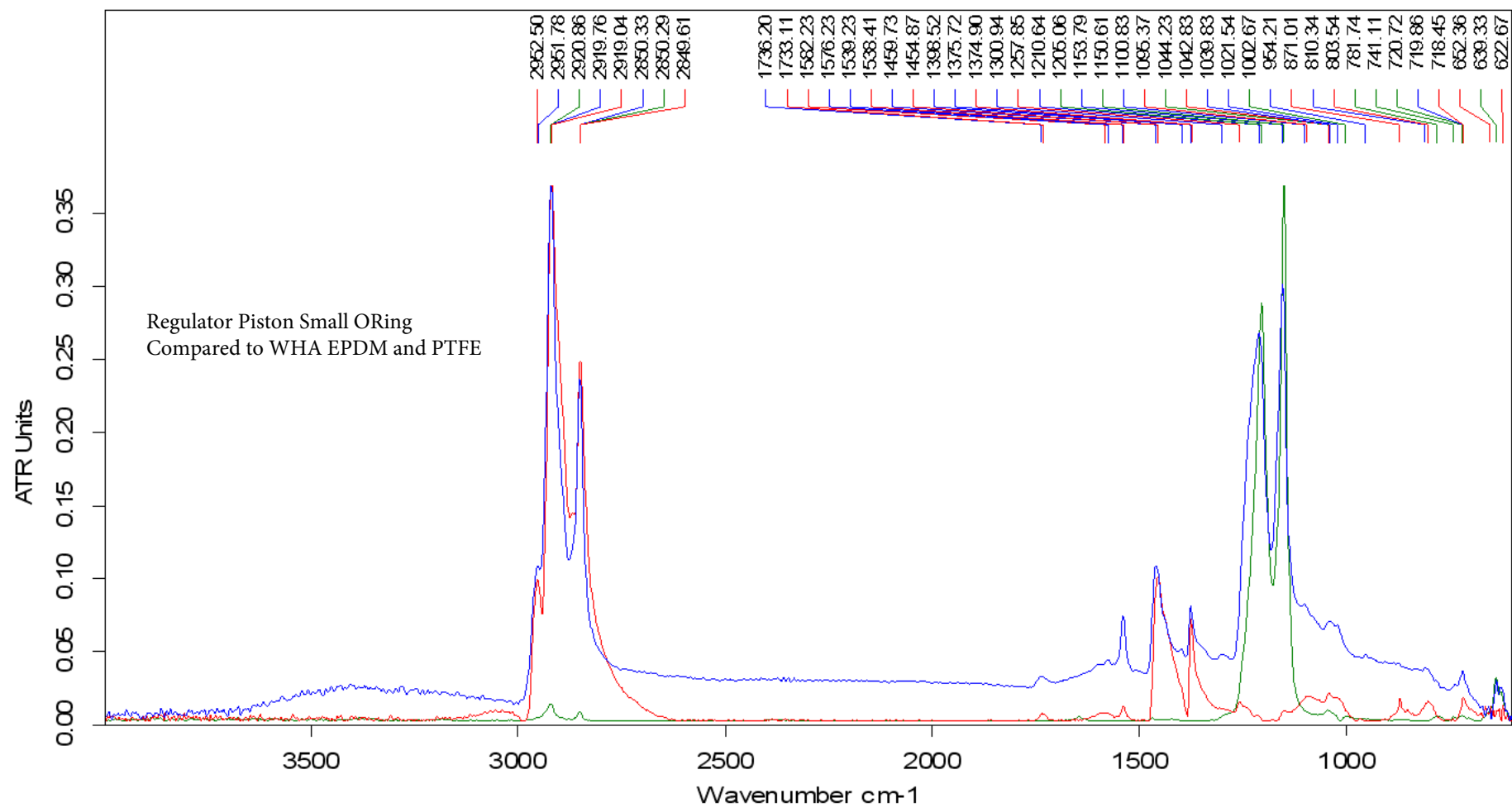
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Appendix D – SEM/EDS Analysis Summary



15113-0 DOT VIPR SEM/EDS Summary

(Detailed SEM/EDS analysis results included in the full WHA data package)



The VIPR was mounted in a Scanning Electron Microscope Chamber for elemental analysis using a specially fabricated holding fixture as shown in the figure to the left. The holding fixture allowed orientation of the VIPR into a variety of positions and was fixed securely to the SEM chamber stage. The full 360 degrees of the threads were accessible using the fixture and the tilt functions of the SEM chamber.

In general the threads were analyzed at the 12, 3, 6, and 9 O'clock positions and EDS elemental analysis was obtained from the base of the threads to the flange.

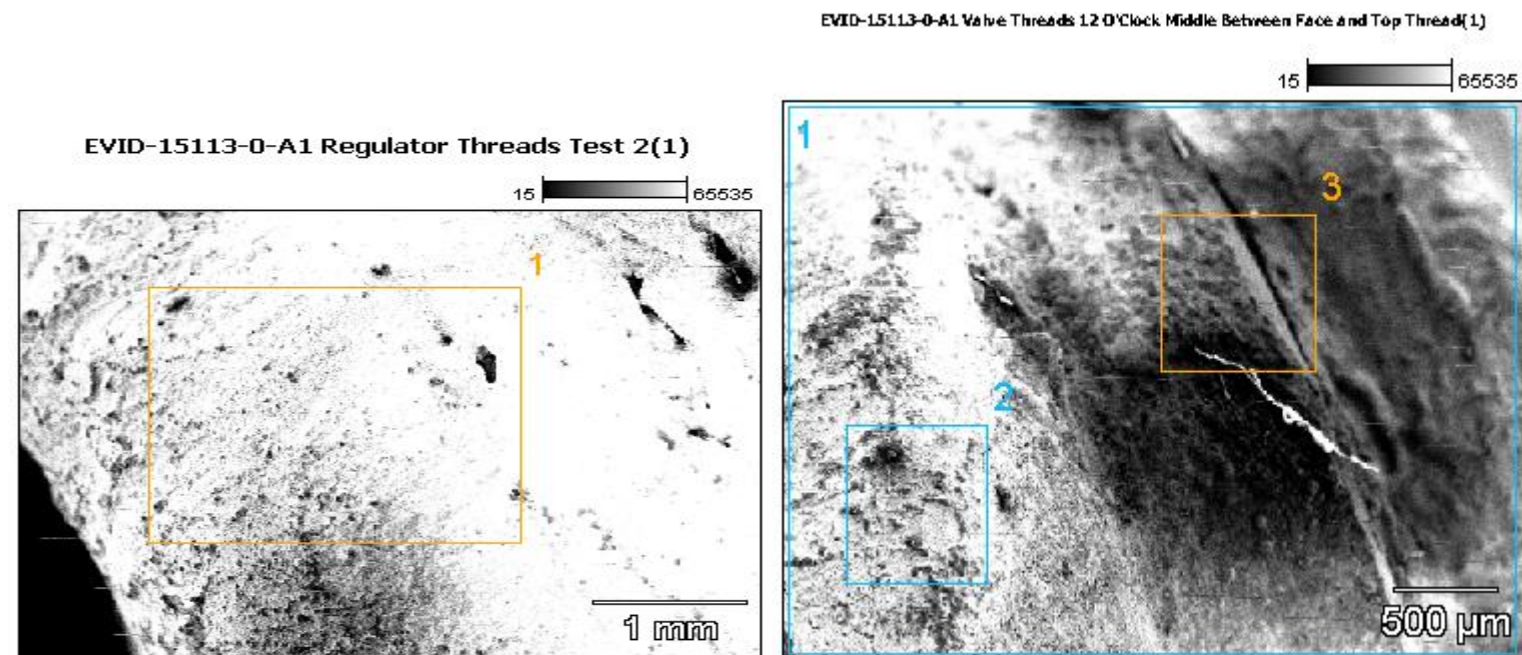




Flange bend
corresponds to
6 o'clock thread
region



Thread Position Region (O'Clock)	Location	Al	Cu	Zn	F	C	O	Mo	Pb	Other	Notes
12	Damaged End 1	5.36	11.17	78.01	1.06	-	4.16	-	-	Ca <1	Localized region
	Damaged End 2	13.98	14.23	45.70	0.81	8.89	13.91	1.51	-	Si, Ca <1	Localized region
	Damaged End 3	8.68	13.55	48.47	0.93	13.10	12.19	1.56	-	Si, P, Cl, Ca <1	Overall
	Damaged End 4	8.74	16.08	44.86	0.45	14.51	12.03	-	1.68	Si, P, Cl, Ca <1	Localized region
	Damaged End 5	9.04	14.53	48.44	0.80	11.68	12.60	-	1.90	Si, Ca <1	Localized region
	Middle Region 1	8.72	14.55	52.60	1.86	11.37	9.30	-	1.48	S <1	Overall
	Middle Region 2	7.82	11.30	52.39	0.55	15.23	11.10	-	-	Si, S, Cl, Ca <1	Localized region, thread crest nearest damaged end
	Middle Region 3	9.11	16.29	51.39	1.93	11.97	7.69	-	1.62	none	Localized region, thread crest nearest middle thread region
	Region near flange 1	10.03	14.30	56.05	4.63	7.32	7.67	-	-	none	Overall
	Region near flange 2	12.40	13.09	55.99	4.23	6.03	8.26	-	-	none	Localized region, top thread
	Region near flange 3	9.59	11.90	58.36	3.91	7.20	8.37	-	0.66	none	Localized region, flat surface near flange shoulder



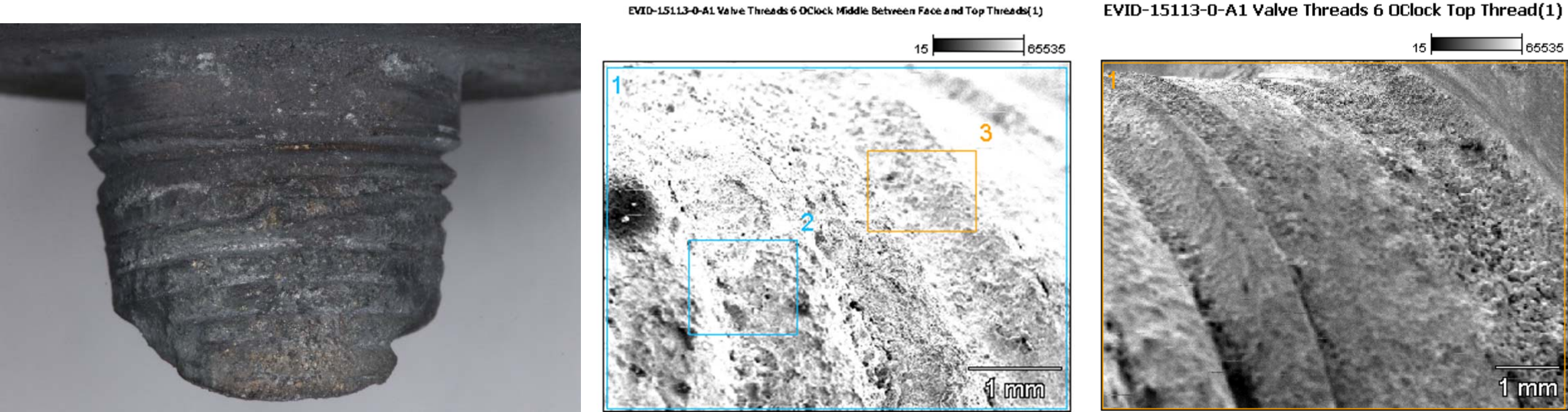
- Al (~10%) generally consistent throughout thread region from damaged end to flange
- F generally higher on middle and flange regions
- Mo and Pb seem to be present more toward the damaged end
- C and O generally higher on damaged and middle thread regions
- Cu (~13%) consistent throughout thread region from damaged end to flange
- Zn highest near flange and decreases moving toward damaged end, Zn content generally higher on this thread position than all other regions, note this end experienced most material loss during event

Thread Position Region (O’Clock)	Location	Al	Cu	Zn	F	C	O	Mo	Pb	Other	Notes
3	Damaged End 1	12.38	16.52	37.98	0.61	12.62	16.18	-	2.55	Si, P, Ca <1	Overall
	Damaged End 2	11.16	19.10	37.37	-	14.84	15.28	-	-	Si, P, S, Cl, Ca <1	Localized region
	Damaged End 3	13.63	16.51	36.58	0.67	10.41	18.17	-	2.98	Si, P, Ca <1	Localized region
	Middle Region 1	9.10	10.31	53.90	3.39	11.28	10.20	-	1.60	Ca <1	Overall
	Middle Region 2	9.32	9.23	54.50	2.96	10.89	11.33	1.16	-	Si, Ca <1	Localized region nearest damaged end
	Middle Region 3	7.81	10.41	57.36	3.11	11.86	7.91	-	1.38	S <1	Localized region further from damaged end
	Upper Thread Region 1	9.74	12.46	51.44	4.93	10.12	10.32	0.99	-	None	Overall
	Upper Thread Region 2	10.41	10.45	52.27	4.92	9.68	11.81	0.45	-	None	Localized, top thread root
	Upper Thread Region 3	12.70	13.01	46.61	5.08	9.56	13.04	-	-	None	Localized region between top thread and flange shoulder
	Flange 1	2.71	45.69	40.28	2.19	5.65	3.48	-	-	none	Localized region on flange
	Flange 2	5.15	33.76	47.29	2.62	6.01	5.17	-	-	none	Overall region of flange
	Flange 3	10.55	17.38	50.85	2.65	8.04	10.25	-	-	S <1	Anomaly on flange



- Al generally higher on damaged end and decreasing moving toward flange region
- F generally higher on middle, upper, and flange regions of threads
- Pb higher on damaged end, no Pb detected on flanged region
- C and O generally higher on near damaged end
- Cu generally higher near flanged region and less on damaged end (Cu content lowest in middle thread region)
- Zn content higher on middle, upper and flange regions and above base material percentage (~46%)

Thread Position Region (O’Clock)	Location	Al	Cu	Zn	F	C	O	Mo	Pb	Other	Notes
6	Damaged End 1	16.29	18.87	18.69	-	18.85	20.44	-	-	S 1.31, Ca 1.65, P 1.92, Si 0.99, Cl, Ti, Mg <1	Overall of region
	Damaged End 2	16.16	12.18	10.20	-	21.03	26.11	-	-	Mg 3.85, Si 2.39, P 3.08, S 2.21, Ca 1.99, Cl, Ti <1	Localized region on end
	Damaged End 3	20.43	19.05	19.25	-	15.04	20.72	2.40	-	P 1.37, Mg, Si, Ca <1	Localized region further from end
	Middle Region 1	24.93	19.94	19.91		12.66	18.56	1.52	-	Mg, Si, P, Cl, Ca <1	overall
	Middle Region 2	24.69	17.57	21.12	1.05	10.42	20.38	1.90	-	Mg, Si, P, Cl, Ca <1	Localized region, thread root
	Middle Region 3	26.68	21.35	19.66	0.84	10.02	18.94	0.94	-	Mg, Si, Cl, Ca <1	Localized region, thread root
	Top Thread Region 1	29.91	19.11	21.40	0.93	8.62	20.03	-	-	none	Overall of region
	Top Thread Region 2	27.02	19.02	20.19	1.26	11.60	20.15	0.76	-	none	Localized thread region further from flange
	Top Tread Region 3	33.59	20.22	15.07		7.91	22.78	-	-	Si <1	Localized region near flange shoulder
	Flange shoulder region	30.11	19.74	19.37	1.86	7.79	21.13	-	-	none	Localized region in flange/thread shoulder
	Flange surface region	28.32	21.69	18.73	1.09	8.72	20.50	-	-	P, Cl <1	Localized region of flange face



- Al content significantly higher on this side of threads, Al content generally increases moving from damaged end to flanged region
- No F detected on damaged end, detected F on middle and flanged regions
- O remains generally consistent throughout thread region
- C generally higher on damaged end, lesser C on top thread and flanged regions
- No Pb detected on this region of thread
- Mo generally higher on damaged end and decreases moving toward flange. No Mo detected on flange region.
- Zn (~20%) generally consistent throughout thread region
- Cu (~20%) generally consistent throughout thread region

Thread Position Region (O'Clock)	Location	Al	Cu	Zn	F	C	O	Mo	Pb	Other	Notes
9	Damaged End 1	11.73	9.19	43.98	3.59	12.19	15.83	1.77	-	Si, P, Cl, Ca <1	Overall analysis of region
	Damaged End 2	12.41	11.37	41.57	3.52	12.91	15.78	-	1.32	P, S, Ca, Ni <1	Overall, similar to Damaged End 1 analysis region
	Damaged End 3	12.71	11.30	40.72	0.81	14.24	16.93	-	1.95	Si, P, Ca, Fe <1	Localized region near damaged edge
	Damaged End 4	10.98	10.01	43.83	1.97	13.15	16.60	-	-	Mg, Si, P, S, Ca <1	Localized region, similar to Damaged End 3 analysis region
	Damaged End 5	13.88	7.31	41.75	5.84	12.90	16.54	-	-	Si, P, S, Cl, Ca <1	Localized region, further from damaged end
	Damaged End 6	12.84	13.84	41.91	1.25	10.30	15.84	-	2.43	Si, P, Ca <1	Different orientation, overall of region
	Damaged End 7	14.73	13.18	42.29	0.48	8.68	16.49	-	2.30	Si, P, S, Ca <1	Different orientation, localized region damaged end
	Damaged End 8	12.63	11.07	46.17	1.14	11.51	13.38	-	2.79	Si, P, Ca <1	Different orientation, localized region damaged end
	Damaged End 9	9.55	7.22	46.48	2.13	13.19	16.10	-	1.98	P 1.13, Si, S, Ca <1	Different orientation, localized region damaged end
	Middle Region 1	13.27	7.62	44.61	5.46	11.22	14.81	-	1.19	Si, P, S, Ca <1	Overall analysis region
	Middle Region 2	12.85	6.67	43.51	6.43	9.25	17.29	2.45	-	Zr 1.04, Ca <1	Localized thread root
	Middle Region 3	11.65	8.72	40.58	5.14	15.24	14.61	1.63	-	P, Cl, K, Ca<1	Localized thread crest
	Middle Region 4	14.29	11.57	44.68	4.44	9.85	12.52	-	2.06	P, Ca <1	Overall analysis region, closer to flange
	Middle Region 5	15.21	12.44	47.75	3.83	6.52	11.46	-	2.35	P <1	Localized thread root
	Top Thread Region 1	13.68	18.34	43.44	4.01	8.33	11.05	0.99	-	Ca <1	Overall analysis region including flange shoulder
	Top Thread Region 2	17.72	9.65	41.80	4.61	10.37	14.53	1.07	-	Ca <1	Localized region , top thread crest
	Top Thread Region 3	5.42	33.88	43.05	4.14	7.54	5.60	0.37	-	none	Localized region, flat face near flange shoulder



- Aluminum (~12%) consistent throughout thread region from damaged end to flange, highest amount of Al (~18%) on thread region near flange
- F generally higher on middle and top thread regions
- Zn (~40-45%) generally consistent throughout threaded region
- Cu generally consistent through threaded region, Cu content higher at top thread region
- C and O generally higher on damaged end
- Generally more Pb on damaged and middle thread regions
- Generally more Mo on middle and top threads regions

Valve Base Metal	Location	Al	Cu	Zn	F	C	O	Other	Notes
	Fill Valve Face 1	0.34	48.94	30.25	1.22	12.12	6.41	Cl, K, Ca, Fe <1	Overall
	Fill Valve Face 2	-	61.61	38.00	-	-	-	Fe <1	Overall

49 CFR 178.46 - Specification 3AL seamless aluminum cylinders.

CFR

eCFR

Authorities (U.S. Code)

Rulemaking

[prev](#) | [next](#)

§ 178.46 Specification 3AL seamless aluminum cylinders.

(a) *Size and service pressure.* A DOT 3AL cylinder is a seamless aluminum cylinder with a maximum water capacity of 1000 pounds and minimum service pressure of 150 psig.

(b) *Authorized material and identification of material.* The material of construction must meet the following conditions:

- (1) Starting stock must be cast stock or traceable to cast stock.
- (2) Material with seams, cracks, laminations, or other defects likely to weaken the finished cylinder may not be used.
- (3) Material must be identified by a suitable method that will identify the alloy, the aluminum producer's cast number, the solution heat treat batch number and the lot number.
- (4) The material must be of uniform quality. Only the following heat treatable aluminum alloys in table 1 and 2 are permitted as follows:

Table 1—Heat or Cast Analysis for Aluminum; Similar to “Aluminum Association”¹ Alloy 6061
[CHEMICAL ANALYSIS IN WEIGHT PERCENT²]

Simin/max	Femax	Cumin/max	Mnmax	Mgmin/max	Crmin/max	Znmax	Timax	Pbmax	Bimax	Other		A1
										eachmax	totalmax	
0.4/0.8	0.7	0.15/0.4	0.15	0.8/1.2	0.04/0.35	0.25	0.15	0.005	0.005	0.05	0.15	Bal.

¹ The “Aluminum Association” refers to “Aluminum Standards and Data 1993”, published by the Aluminum Association Inc.

² Except for “Pb” and “Bi”, the chemical composition corresponds with that of Table 1 of ASTM B 221 (IBR, see § 171.7 of this subchapter) for Aluminum Association alloy 6061.

Table 2—Mechanical Property Limits

Alloy and temper	Tensile strength—PSI		Elongation—percent minimum for 2” or 4D ¹ size specimen	
	Ultimate—minimum	Yield—minimum		
6061-T6	38,000	35,000	² 14	

¹ “D” represents specimen diameters. When the cylinder wall is greater than 3/16 inch thick, a retest without reheat treatment using the 4D size specimen is authorized if the test using the 2 inch size specimen fails to meet elongation requirements.

² When cylinder wall is not over 3/16-inch thick, 10 percent elongation is authorized when using a 24t×6t size test specimen.

Overall

Zinc

- *Zn content was greater for region of the thread that experienced more material loss (12, 3, and 9 o'clock positions)*

Fluorine

- *Generally see more F near flange area, lesser near damaged areas (exception 9 o'clock region)*

Aluminum

- *Al content was greatest for the 6 o'clock thread position (region of thread that experienced less material loss)*
 - o 12 o'clock position: Al content (~10%) generally consistent throughout thread position from damaged end to flange
 - o 3 o'clock position: Al content generally higher on damaged end (~12%) than flanged end (~6%)
 - o 6 o'clock position: Al content generally increasing from damaged end (~18%) to flanged end (~30%)
 - o 9 o'clock position: Al content (~12%) consistent throughout thread region from damaged end to flange, highest amount of Al (~18%) on thread region near flange

Lead and Molybdenum

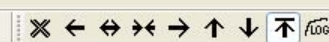
- *Generally more Pb and Mo observed at damaged end of thread positions*
 - o 6 o'clock position observed this trend for Mo but there was no Pb (region of lesser material consumption)

Carbon and Oxygen

- *C and O typically higher on damaged end of thread (12, 3, 9 o'clock positions)*
 - o Exception was 6 o'clock position where O remains consistent throughout threaded region and C generally higher on damaged end (region of lesser material consumption).

Copper

- *Cu content generally consistent throughout each thread region*
 - o Exception positions 3 and 9 where Cu content is generally higher at flanged end (lesser at damaged end)



EDS Calibration

Microanalysis

Service



Instrument Configuration



EDS Calibration

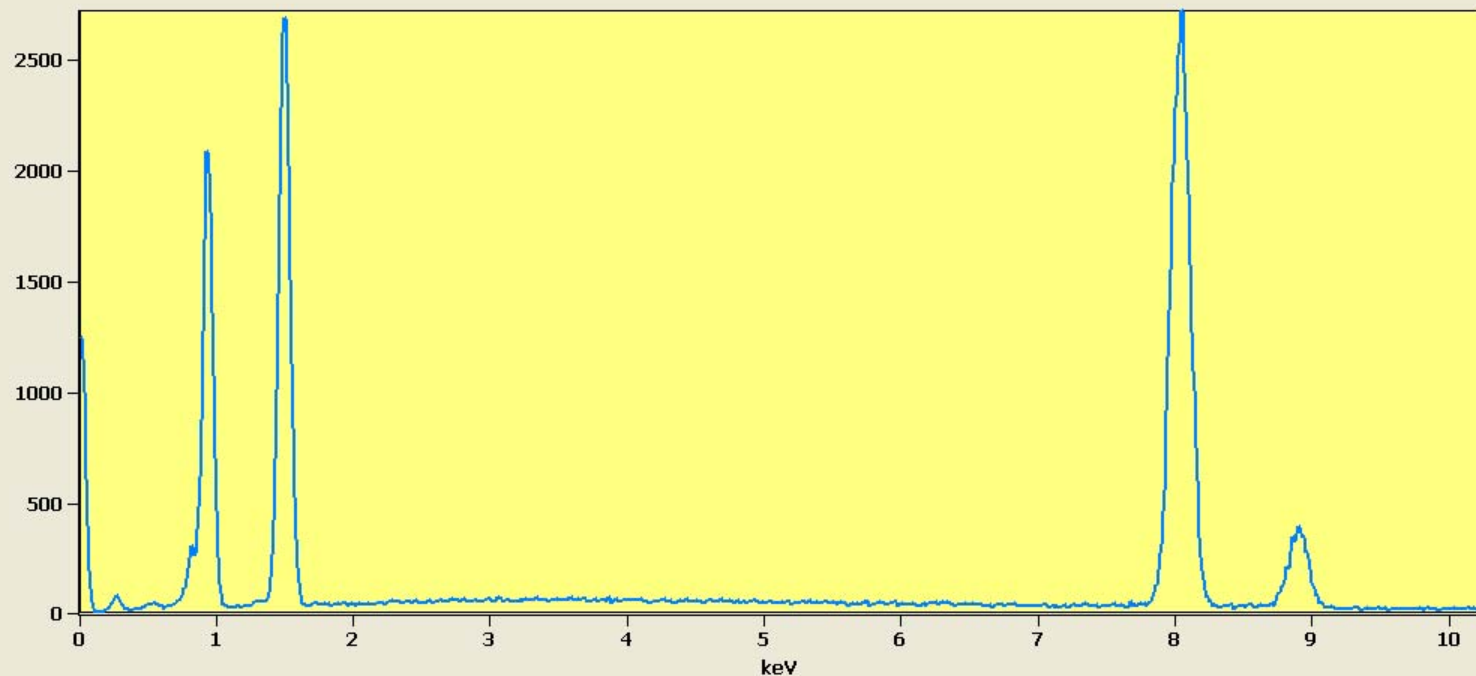


Image Calibration

Base name:

NASA Al Cu Calibration Standard

Full scale counts: 2719



Fine Gain Calibration Manual

Setup

Atomic Symbol: Cu Line: K

Maximum Iterations: 5

Time Constant: [dropdown]

Fine Gain

Current Setting: 45012

Name	Time Constant (nS)	Fine Gain	Calibration Date
Rate 1	90000	44895	8/4/2015
Rate 2	50000	45012	1/20/2016
Rate 4	20000	44866	8/4/2015
Rate 5	12000	44921	8/4/2015
Rate 6	7000	44904	8/4/2015
Rate 7	4000	44979	8/4/2015
Rate 9	2000	46042	8/4/2015
Rate 10	1000	46037	8/4/2015

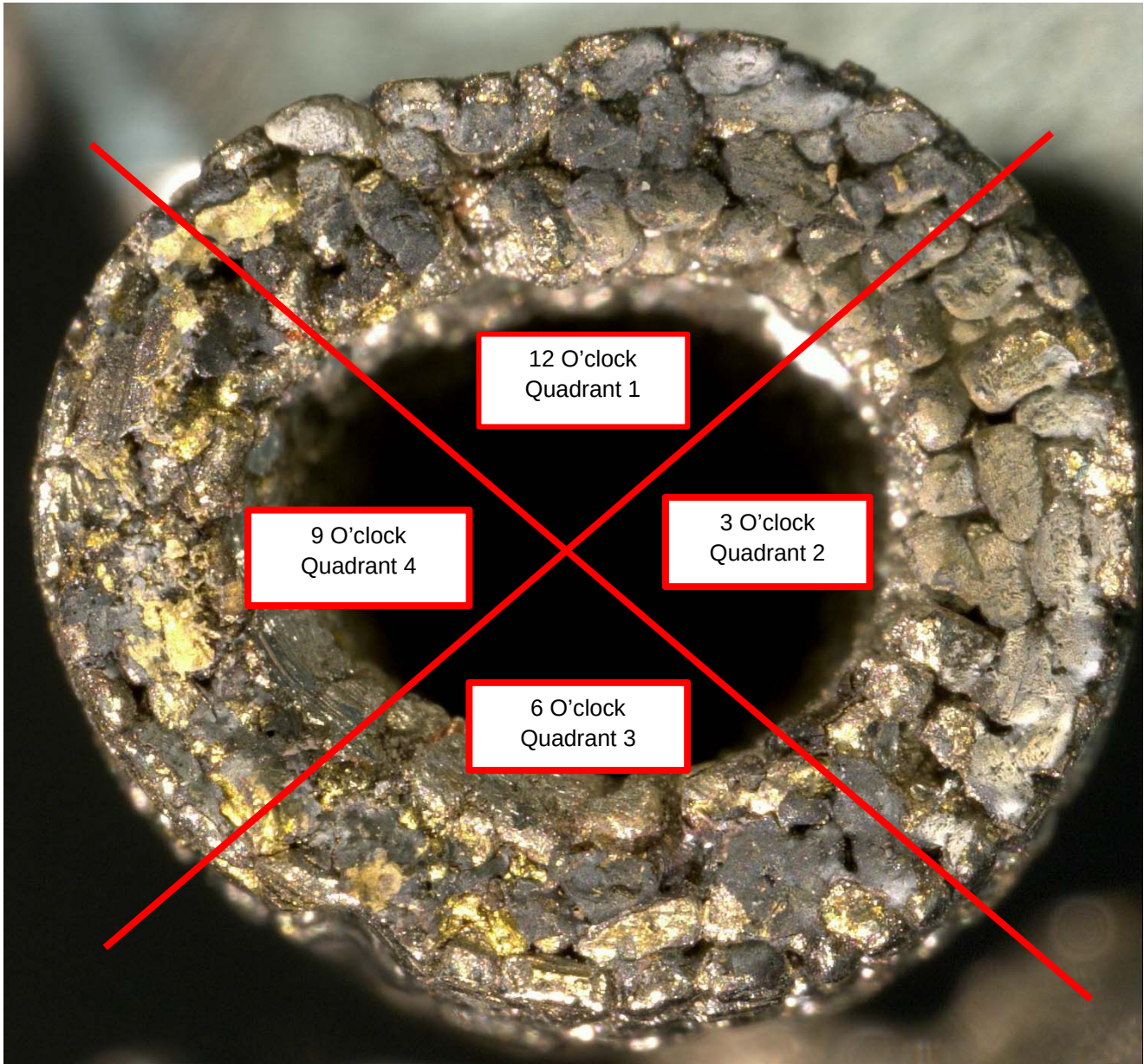


Figure 1 –EVID-15113-0-A1 Filter Approximate Quadrant Labels

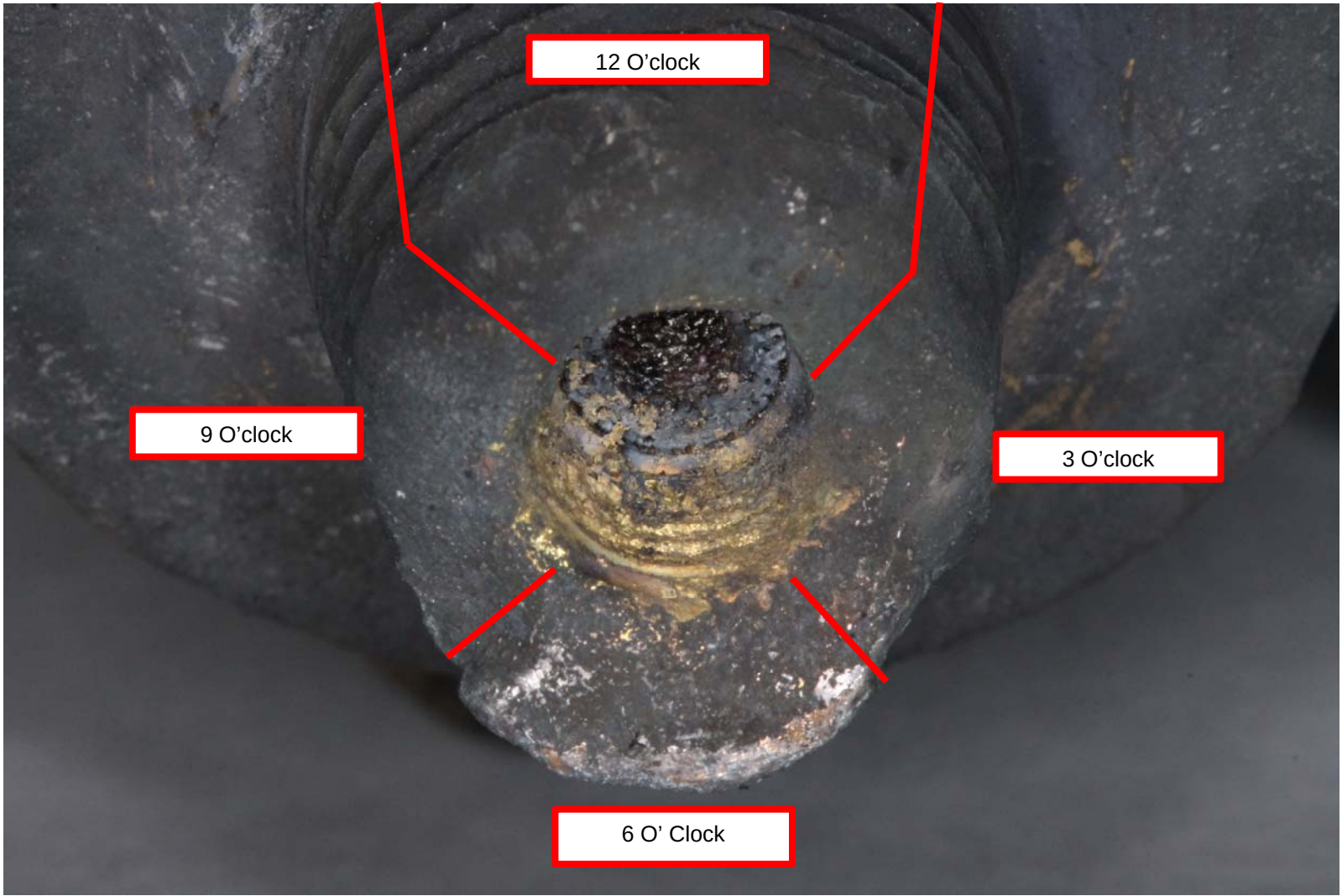
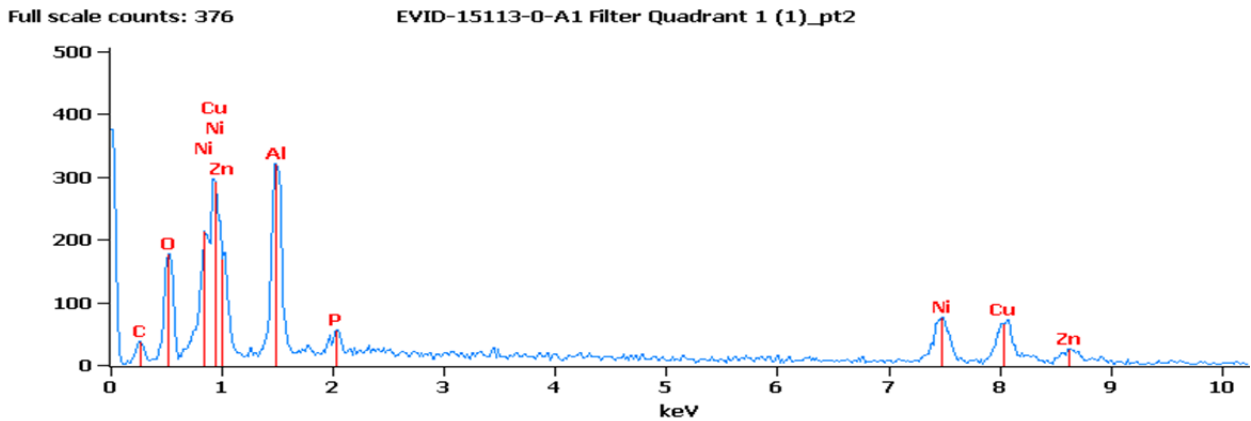
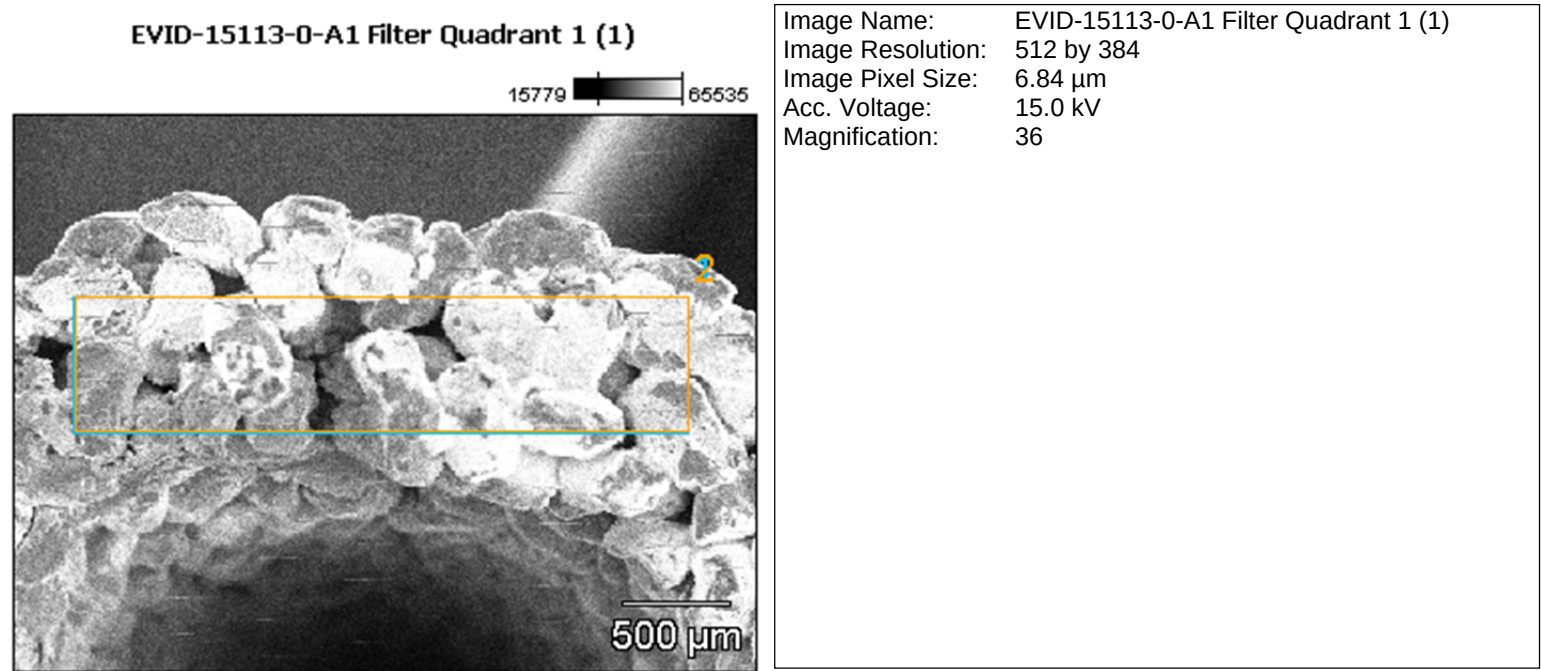


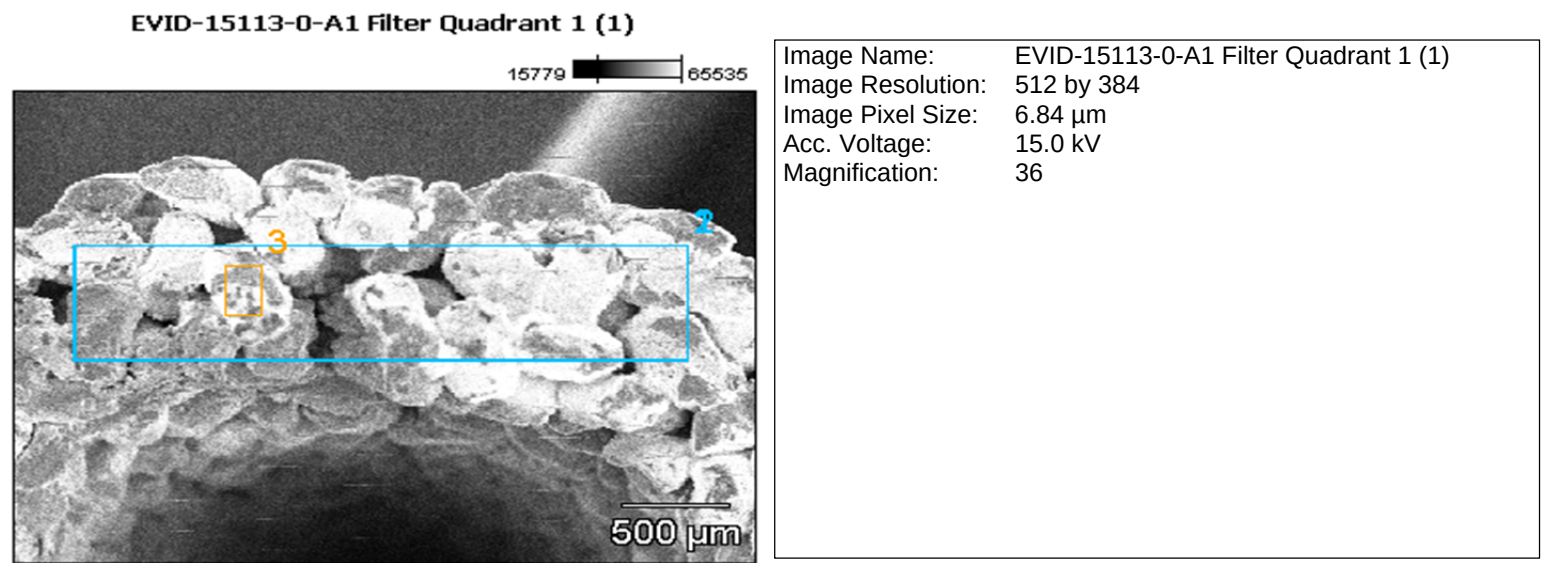
Figure 2 – EVID-15113-0-A1 Valve Threads (Including Flange) Approximate Area of Analysis (Excludes Face Area

EVID 15113-0-A1 Valve Stem Filter Face Facing Cylinder 12 OClock Region Quadrant 1: First and Second Scan



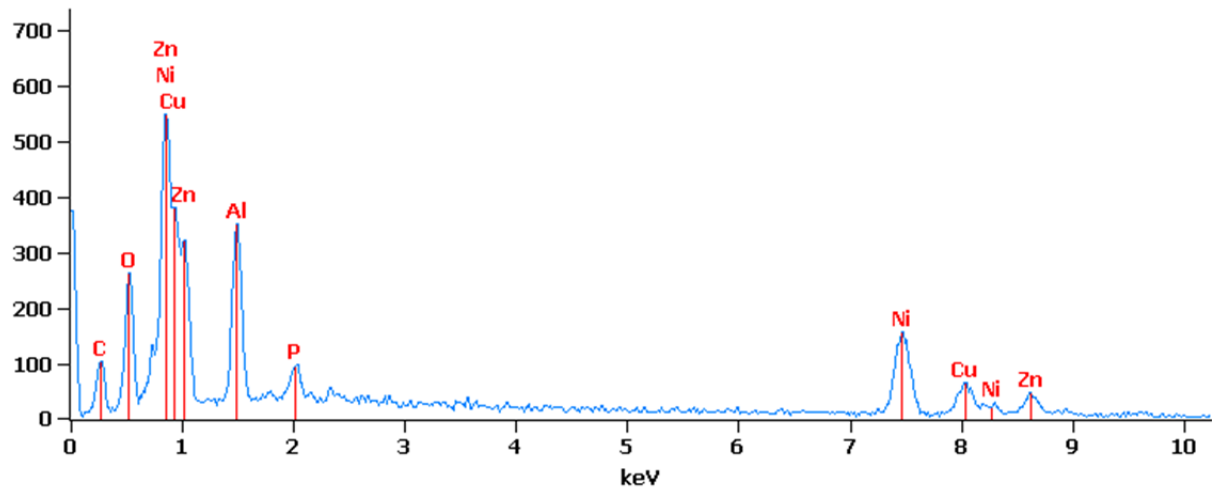
Filter Fit				
Chi² value: 1.092				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 15.0 kV Take Off Angle: 34.9°				
Element Line	Element Weight %	Element Weight % Error ±	Atom Weight %	Atom Weight % Error ±
C K	7.01	0.44	19.38	1.23
O K	13.74	0.45	28.50	0.92
Al K	12.97	0.28	15.95	0.35
P K	1.47	0.17	1.58	0.18
Ni K	22.76	1.61	12.86	0.91
Cu K	26.58	2.46	13.88	1.28
Zn K	15.47	1.57	7.85	0.80
Total	100	---	100	---

EVID 15113-0-A1 Valve Stem Filter Face Facing Cylinder 12 OClock Quadrant 1: Third Scan



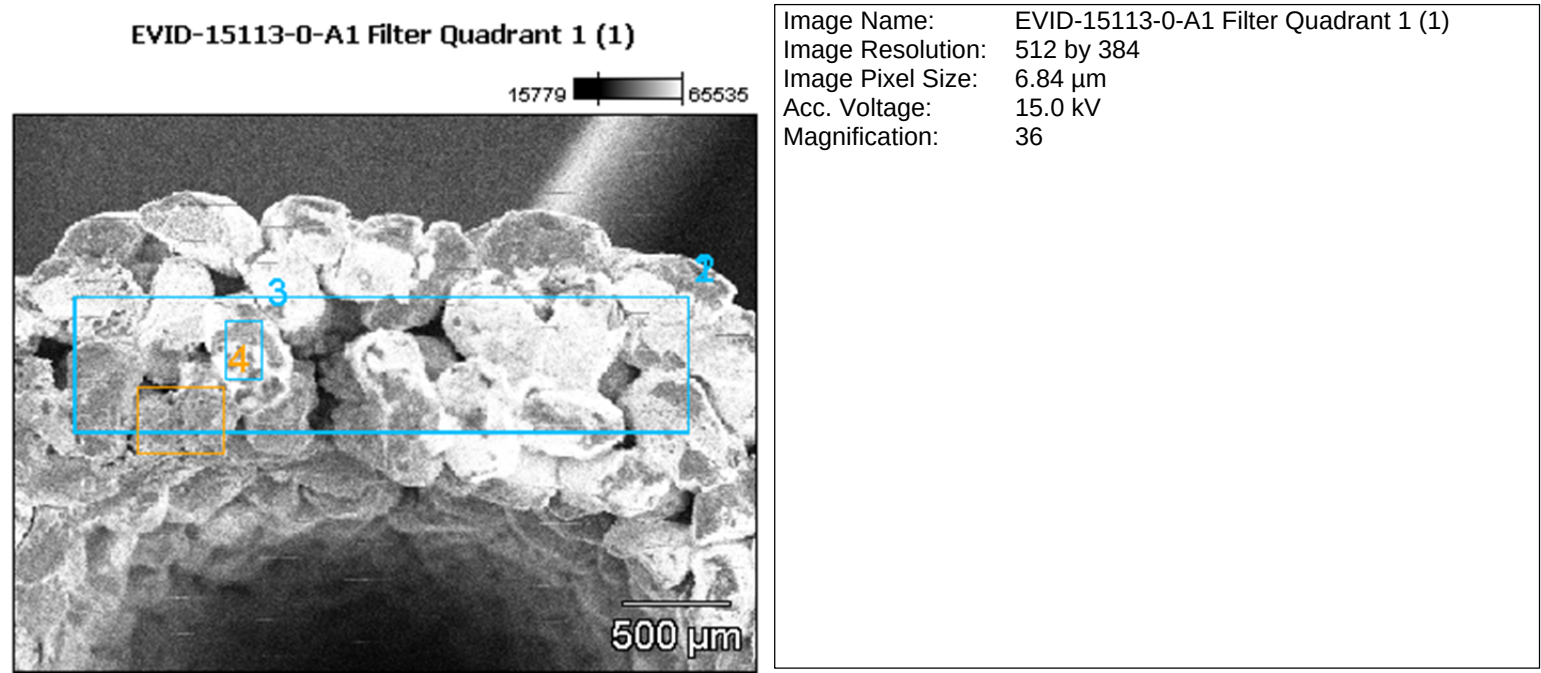
Full scale counts: 549

EVID-15113-0-A1 Filter Quadrant 1 (1)_pt3



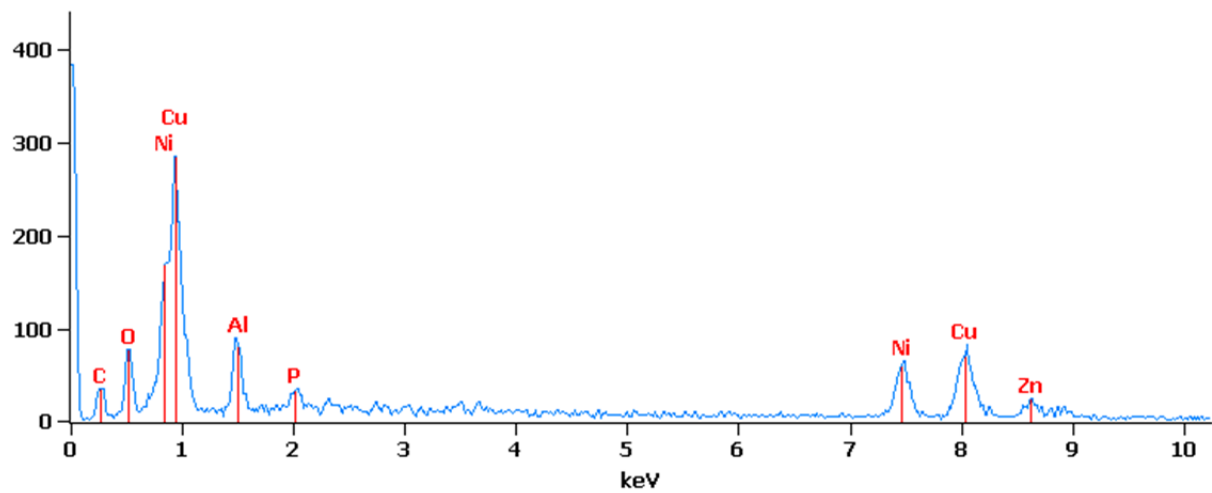
Filter Fit				
Chi² value: 0.880				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 15.0 kV Take Off Angle: 34.9°				
Element Line	Element Weight %	Element Weight % Error ±	Atom Weight %	Atom Weight % Error ±
C K	12.78	0.63	32.85	1.60
O K	11.83	0.27	22.82	0.53
Al K	8.71	0.21	9.97	0.24
P K	1.75	0.15	1.74	0.15
Ni K	31.99	1.42	16.82	0.74
Cu K	17.89	1.83	8.69	0.89
Zn K	15.06	1.25	7.11	0.60
Total	100	---	100	---

EVID 15113-0-A1 Valve Stem Filter Face Facing Cylinder 12 OClock Quadrant 1: Fourth Scan

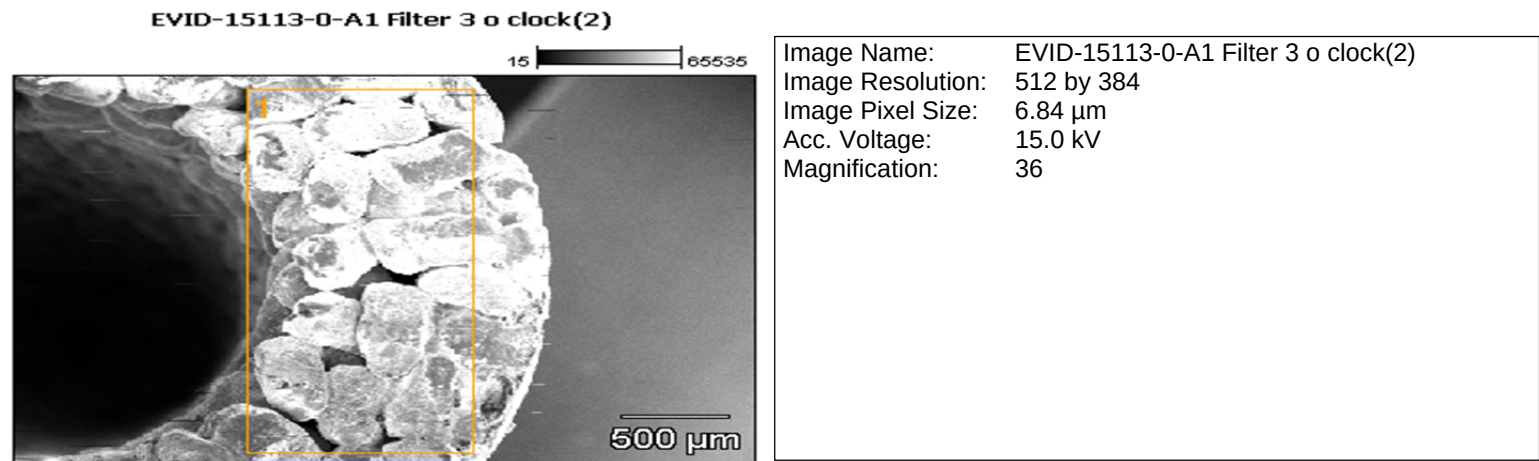


Full scale counts: 383

EVID-15113-0-A1 Filter Quadrant 1 (1)_pt4

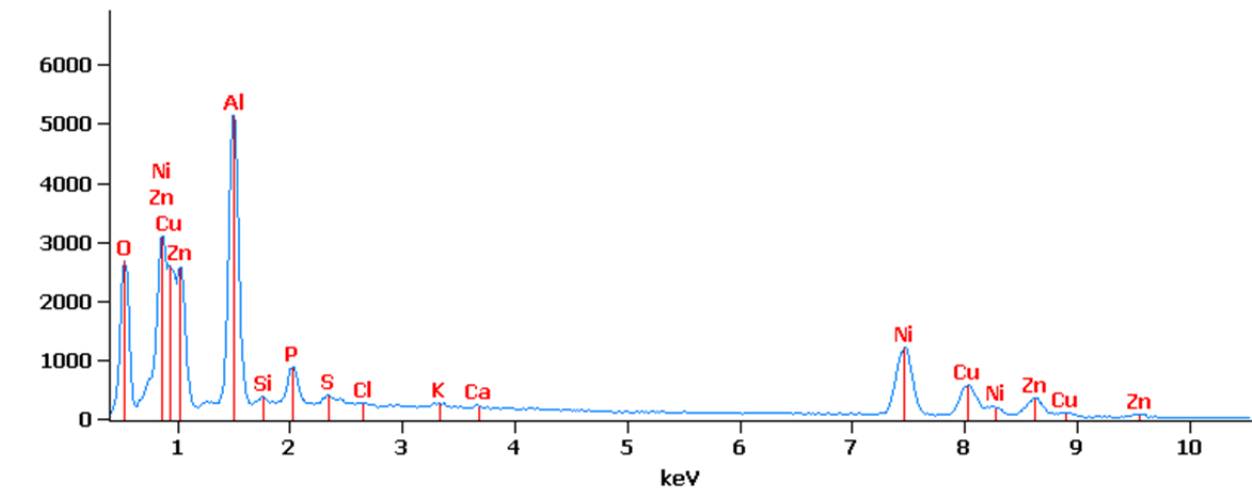


EVID 15113-0-A1 Valve Stem Filter Face Facing Cylinder 3 OClock Quadrant 2: First Scan



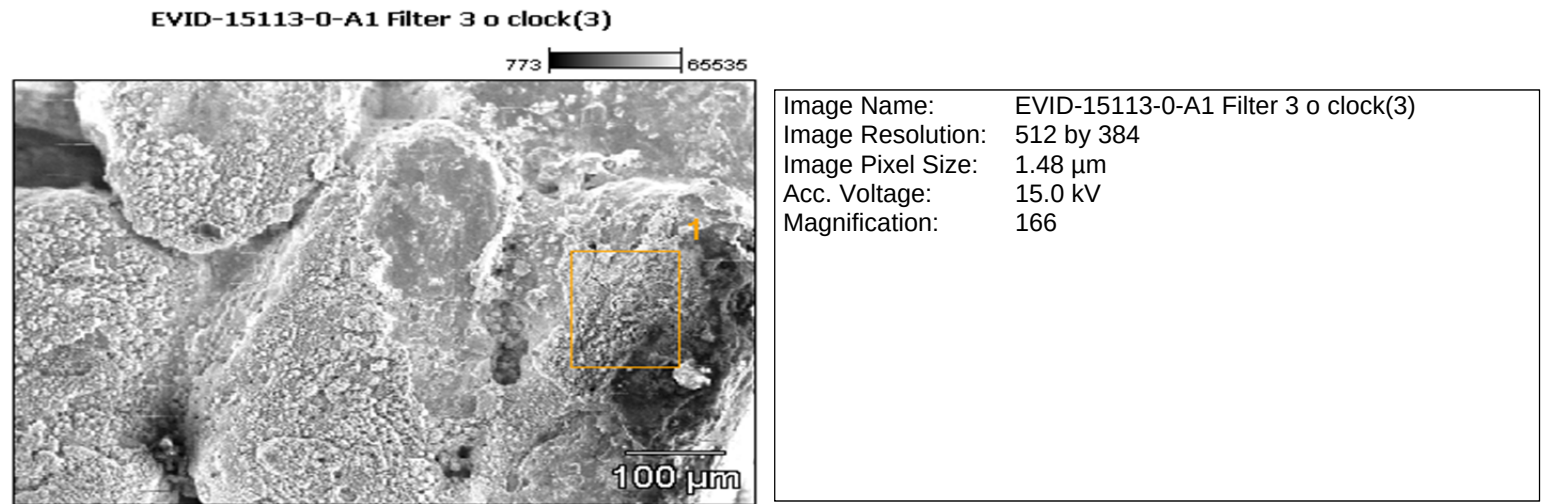
Full scale counts: 5136

EVID-15113-0-A1 Filter 3 o clock(2)_pt1



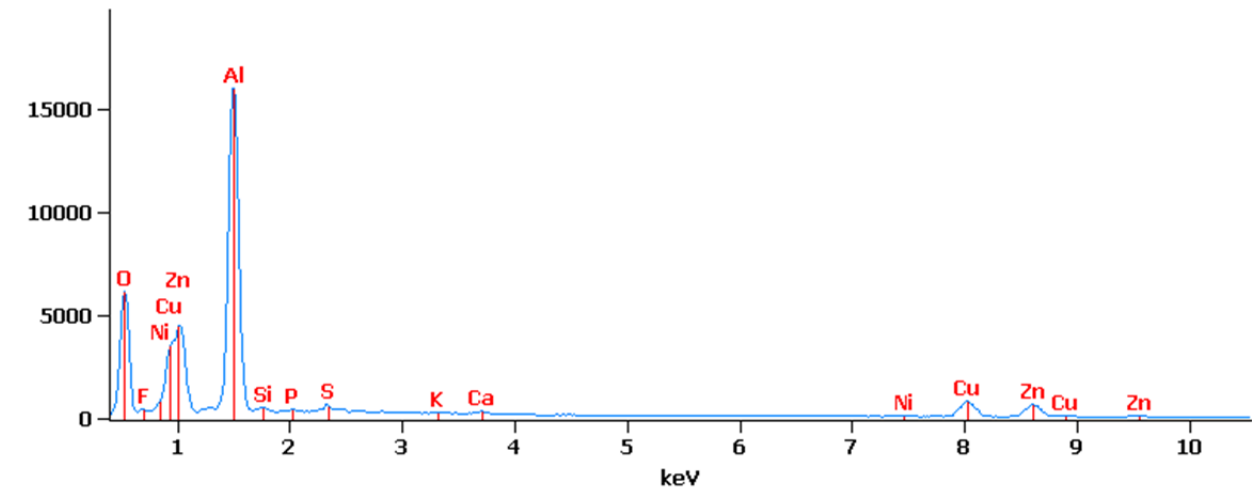
Filter Fit				
Chi² value: 1.473				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 15.0 kV Take Off Angle: 34.9°				
Element Line	Element Weight %	Element Weight % Error ±	Atom Weight %	Atom Weight % Error ±
C K	6.84	± 0.16	18.45	0.43
O K	14.23	± 0.14	28.84	0.28
Al K	14.54	± 0.09	17.48	0.11
Si K	0.22	± 0.03	0.25	0.03
P K	1.81	± 0.05	1.89	0.06
S K	0.30	± 0.05	0.30	0.05
Cl K	0.10	± 0.03	0.09	0.02
K K	0.14	± 0.03	0.12	0.02
Ca K	0.15	± 0.03	0.12	0.02
Ni K	28.35	± 0.46	15.66	0.26
Cu K	18.06	± 0.62	9.21	0.32
Zn K	15.26	± 0.75	7.57	0.37
Total	100	---	100	---

EVID 15113-0-A1 Valve Stem Filter Face Facing Cylinder 3 OClock Quadrant 2: Second Scan Magnified



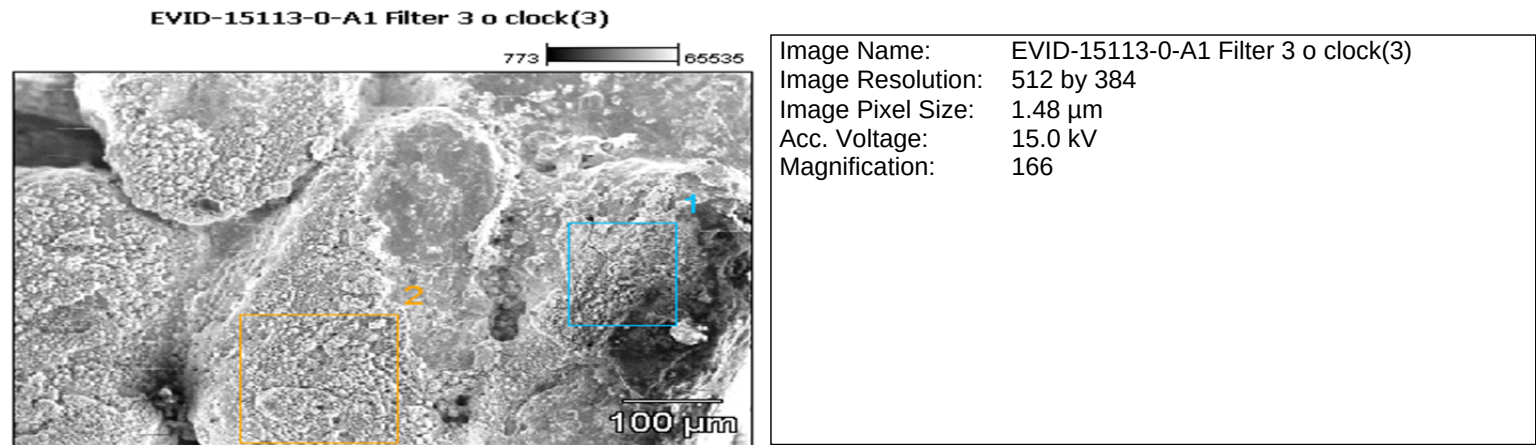
Full scale counts: 16008

EVID-15113-0-A1 Filter 3 o clock(3)_pt1



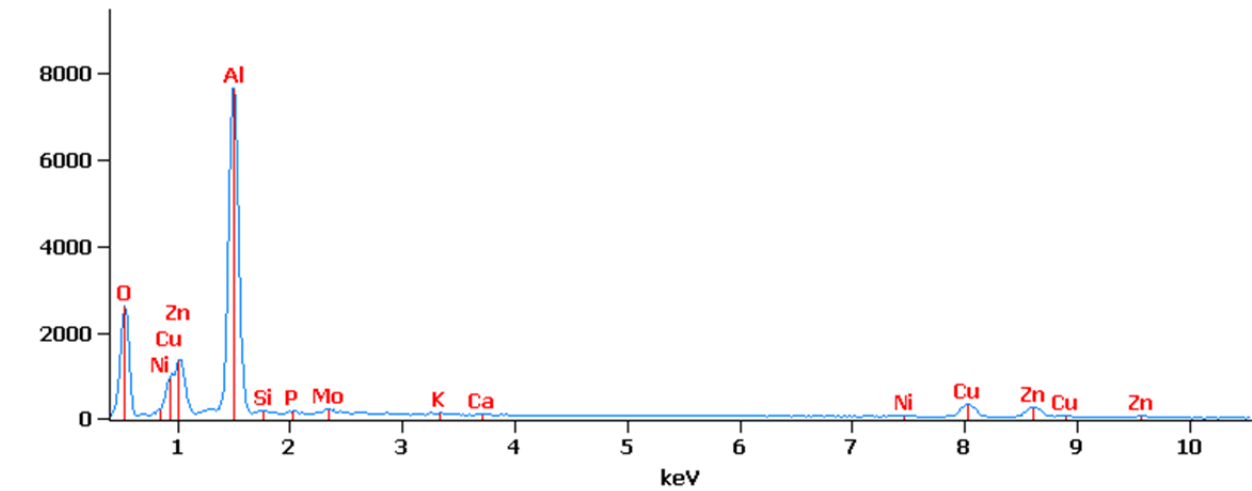
Filter Fit Chi² value: 2.664				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 15.0 kV Take Off Angle: 34.3°				
Element Line	Element Weight %	Element Weight % Error ±	Atom Weight %	Atom Weight % Error ±
C K	12.25	0.18	25.06	0.37
O K	23.49	0.18	36.08	0.28
F K	0.67	0.13	0.87	0.17
Al K	24.89	0.11	22.67	0.10
Si K	0.40	0.04	0.35	0.04
P K	0.27	0.04	0.21	0.03
S K	0.44	0.04	0.34	0.03
K K	0.10	0.02	0.07	0.01
Ca K	0.19	0.02	0.12	0.01
Ni K	1.10	0.10	0.46	0.04
Cu K	17.10	0.40	6.61	0.15
Zn K	19.08	0.34	7.17	0.13
Total	100	---	100	---

EVID 15113-0-A1 Valve Stem Filter Face Facing Cylinder 3 OClock Quadrant 2: Third Scan Magnified



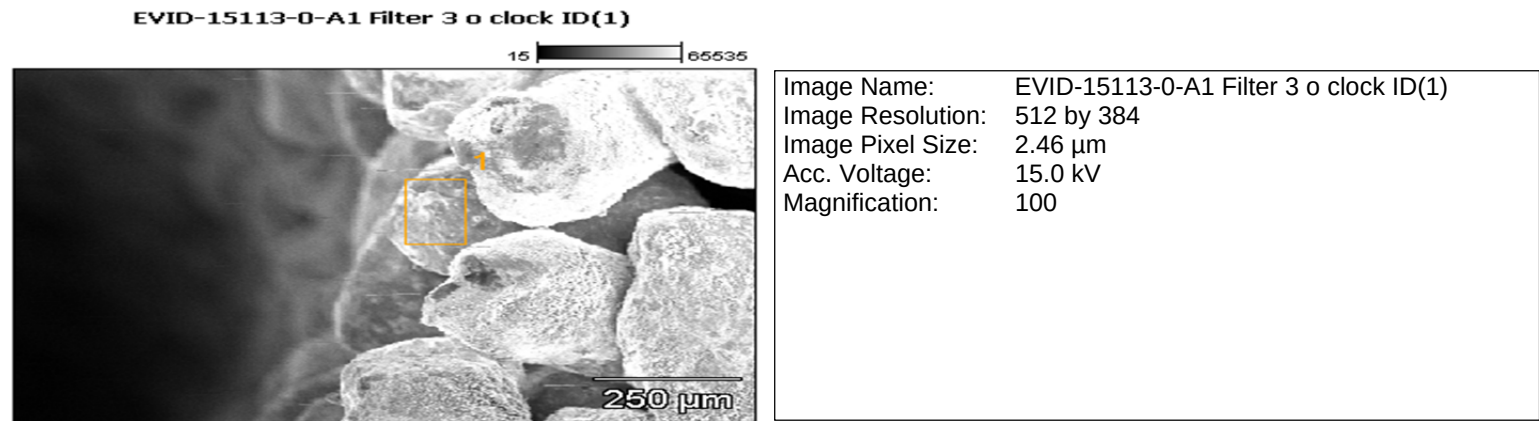
Full scale counts: 7659

EVID-15113-0-A1 Filter 3 o clock(3)_pt2



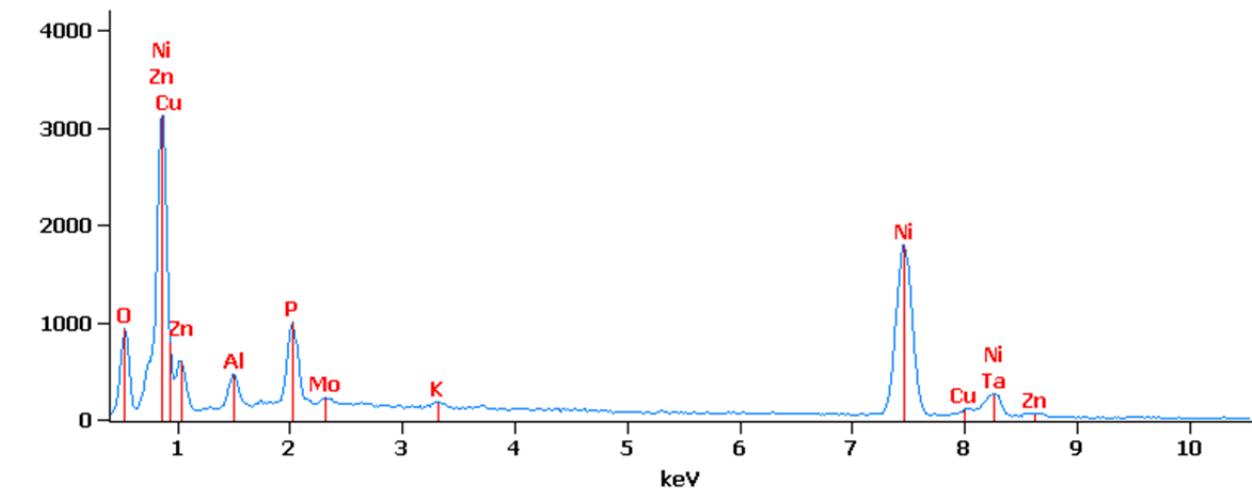
Filter Fit				
Chi² value: 1.197				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 15.0 kV Take Off Angle: 34.3°				
Element Line	Element Weight %	Element Weight % Error ±	Atom Weight %	Atom Weight % Error ±
C K	4.39	0.20	10.03	0.47
O K	23.69	0.20	40.64	0.34
Al K	31.23	0.16	31.77	0.17
Si K	0.30	0.04	0.29	0.03
P K	0.22	0.03	0.20	0.03
K K	0.13	0.03	0.09	0.02
Ca K	0.17	0.04	0.12	0.02
Ni K	1.34	0.17	0.63	0.08
Cu K	17.18	0.67	7.42	0.29
Zn K	20.30	0.91	8.52	0.38
Mo L	1.05	0.12	0.30	0.04
Total	100	---	100	---

EVID 15113-0-A1 Valve Stem Filter Interior Near Filter Face Facing Cylinder 3 OClock Quadrant 2: Third Scan Magnified



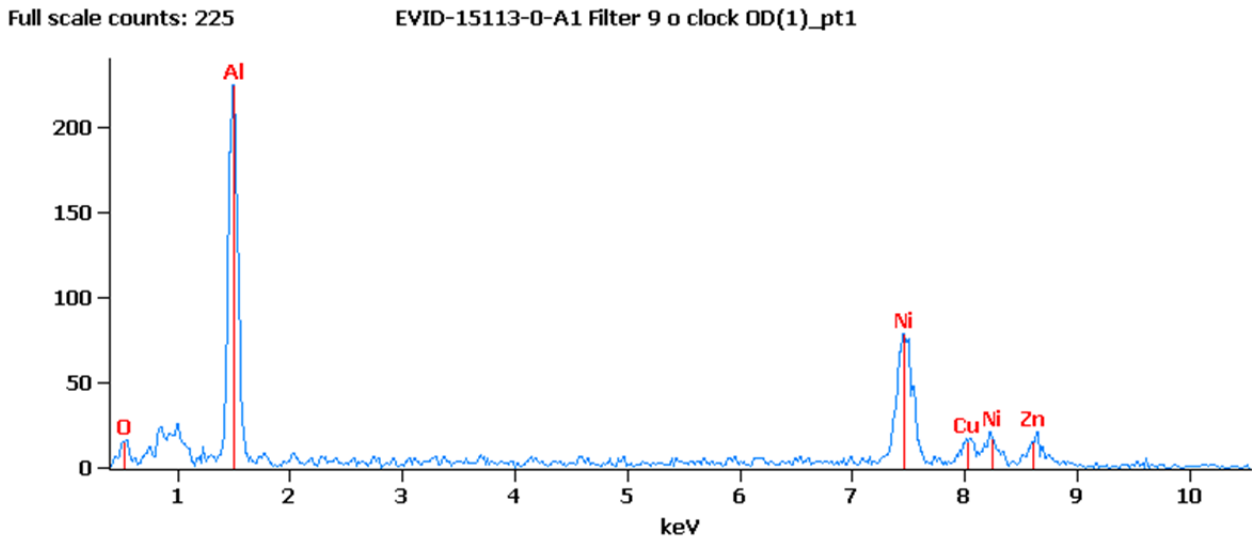
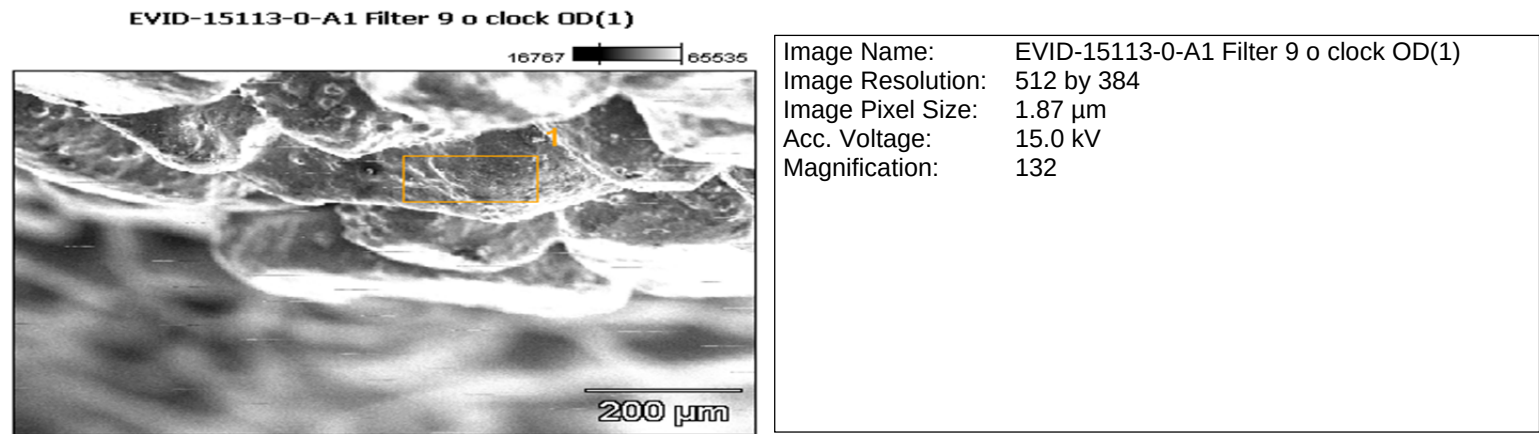
Full scale counts: 3125

EVID-15113-0-A1 Filter 3 o clock ID(1)_pt1



Filter Fit				
Chi² value: 1.728				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 15.0 kV Take Off Angle: 34.3°				
Element Line	Element Weight %	Element Weight % Error ±	Atom Weight %	Atom Weight % Error ±
C K	5.89	0.18	19.61	0.59
O K	7.22	0.13	18.03	0.32
Al K	1.86	0.06	2.76	0.09
P K	3.82	0.08	4.93	0.10
K K	0.27	0.03	0.28	0.04
Ni K	72.16	0.79	49.12	0.54
Cu K	3.55	0.36	2.23	0.23
Zn K	4.41	0.75	2.70	0.46
Mo L	0.82	0.10	0.34	0.04
Ta L	0.00	0.00	0.00	---
Total	100	---	100	---

EVID 15113-0-A1 Valve Stem Filter Exterior Near Filter Face Facing Cylinder 9 O'clock Quadrant 4: First Scan Magnified



Area of analysis uncertain- Analysis was shot on the outside of the filter at an angle that would have the beam hitting the sample clamp and reflecting into the detector. The sample clamp analysis is included below

Filter Fit Chi² value: 1.132 Correction Method: Proza (Phi-Rho-Z) Acc.Voltage: 15.0 kV Take Off Angle: 34.9°				
Element Line	Element Weight %	Element Weight % Error ±	Atom Weight %	Atom Weight % Error ±
C K	11.74	0.65	34.34	1.91
O K	1.85	0.20	4.06	0.44
Al K	16.06	0.36	20.91	0.46
Ni K	44.40	2.51	26.57	1.50
Cu K	11.44	1.58	6.32	0.87
Zn K	14.51	1.91	7.80	1.03
Total	100	---	100	---

Aluminum Clamp used to hold Valve Stem Filter

SED-EDS Clamp Background(1)

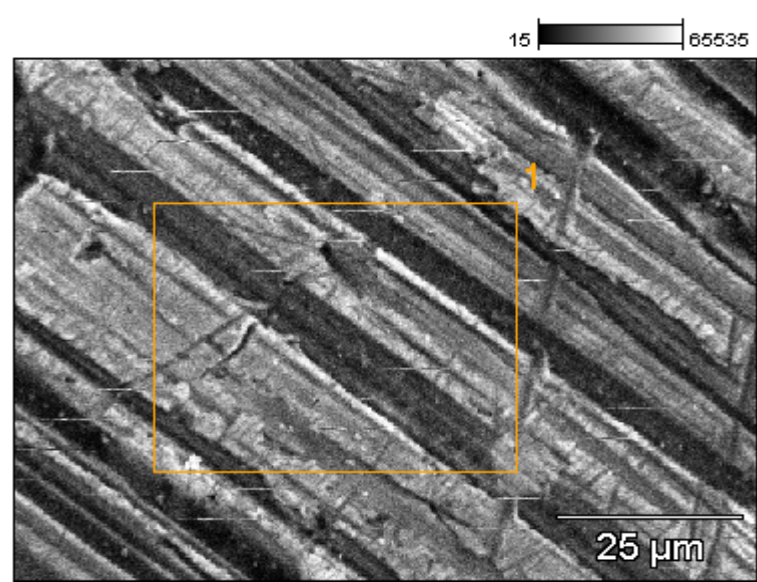
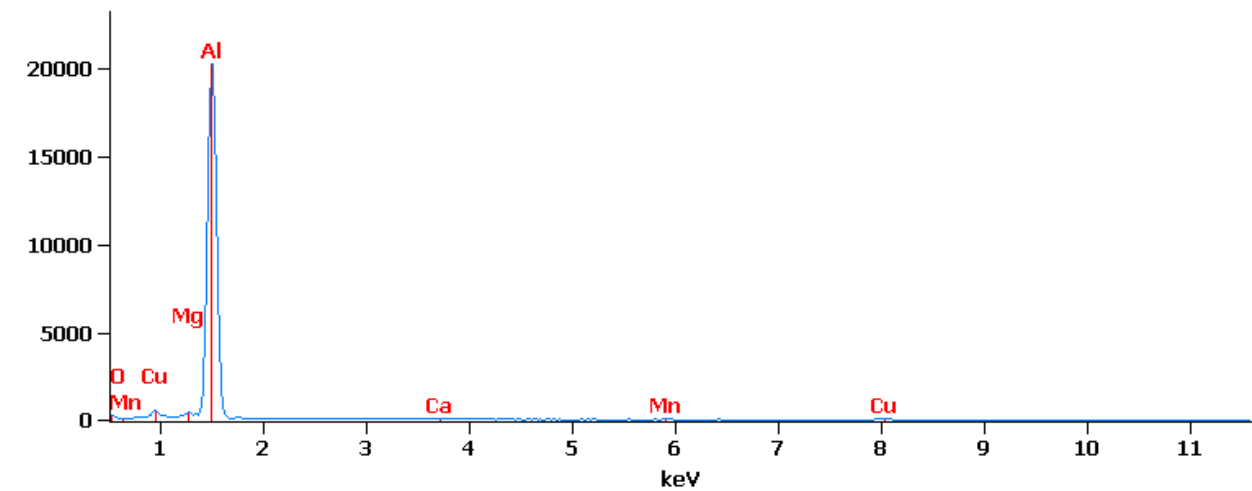


Image Name: SED-EDS Clamp Background(1)
Image Resolution: 512 by 384
Image Pixel Size: 0.20 µm
Acc. Voltage: 15.0 kV
Magnification: 1252

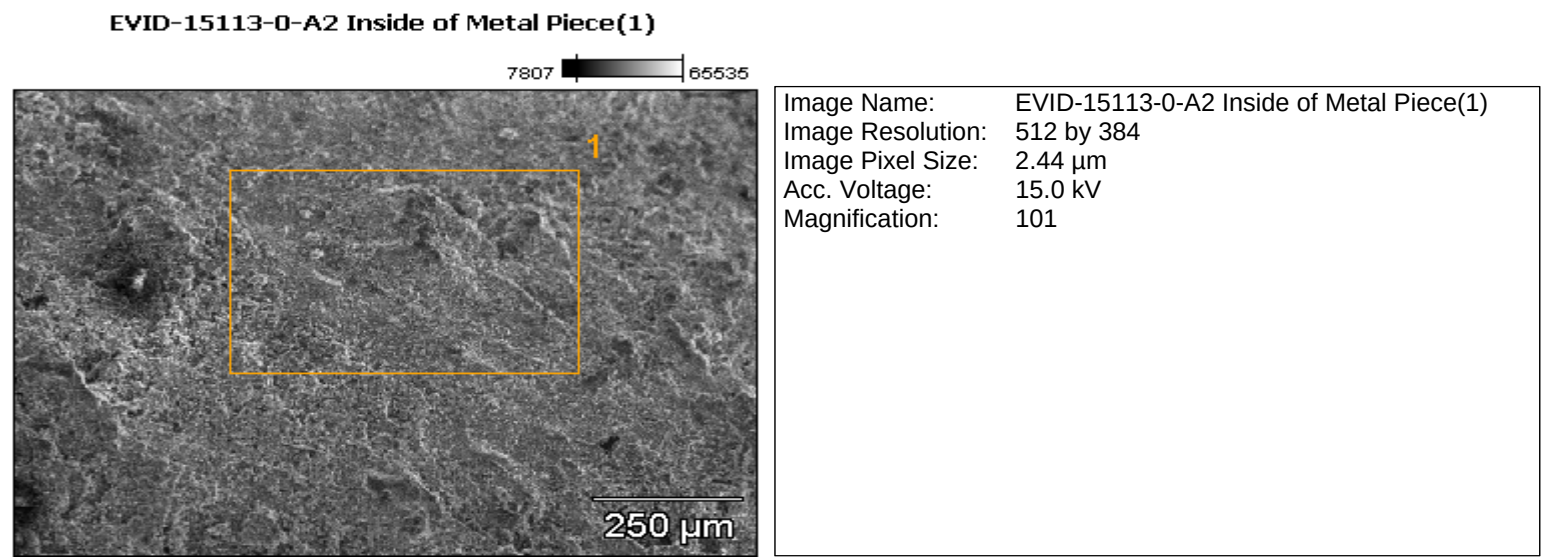
Full scale counts: 20245

SED-EDS Clamp Background(1)_pt1



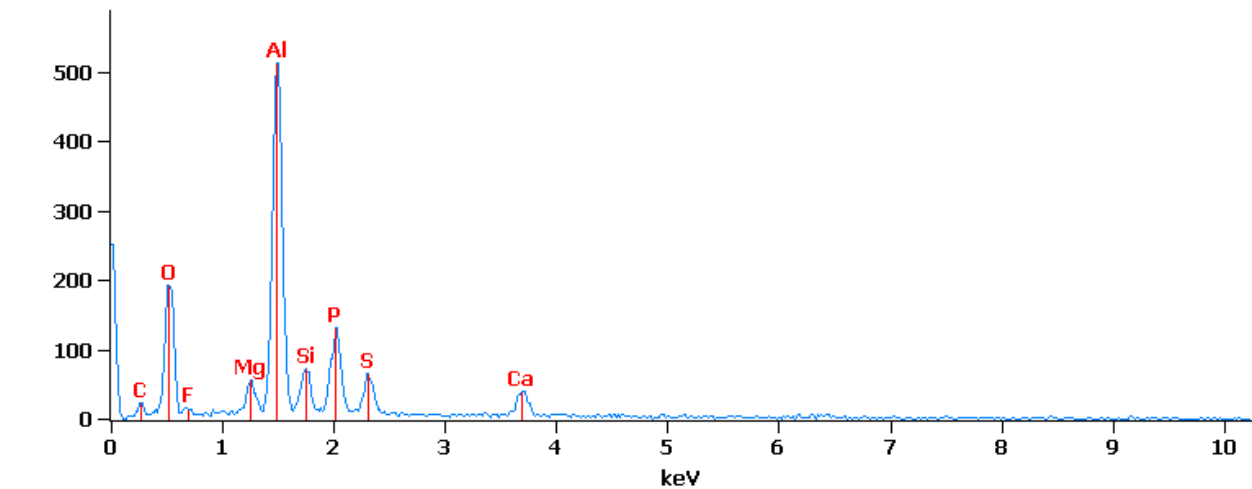
Filter Fit Chi² value: 1.813 Correction Method: Proza (Phi-Rho-Z) Acc.Voltage: 15.0 kV Take Off Angle: 37.4°				
Element Line	Element Weight %	Element Weight % Error ±	Atom Weight %	Atom Weight % Error ±
C K	27.10	0.42	45.82	0.72
O K	2.37	0.16	3.01	0.20
Mg K	0.73	0.04	0.61	0.03
Al K	65.06	0.27	48.97	0.20
Ca K	0.17	0.03	0.09	0.02
Mn K	0.77	0.10	0.28	0.04
Cu K	3.81	0.48	1.22	0.15
Total	100	---	100	---

EVID-15113-0-A2 Interior of Metal Piece (Side Opposite of Green Coloring)



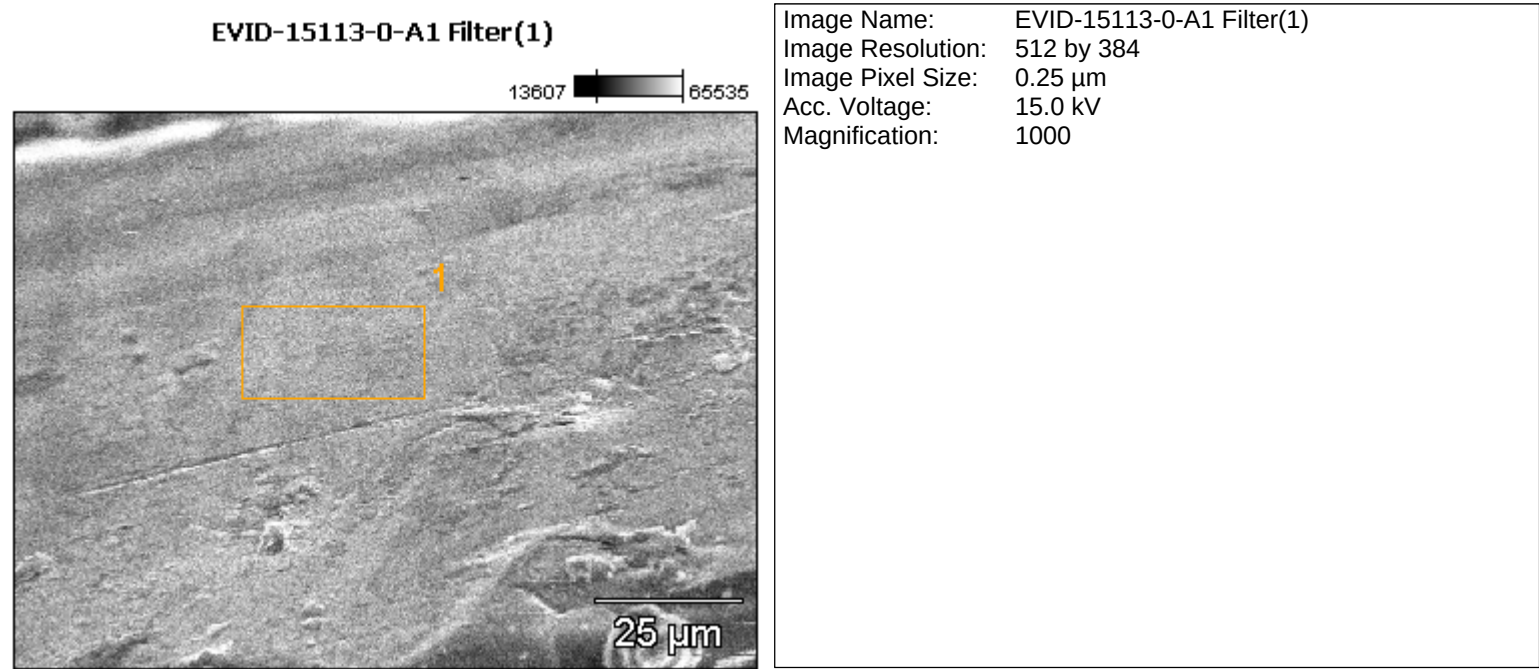
Full scale counts: 512

EVID-15113-0-A2 Inside of Metal Piece(1)_pt1



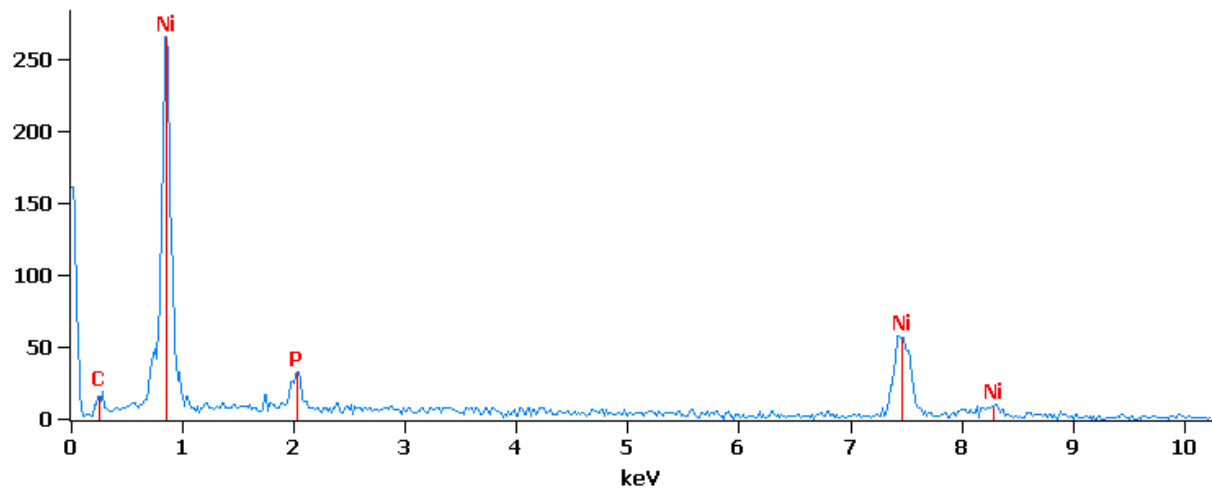
Filter Fit				
Chi² value: 0.765				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 15.0 kV Take Off Angle: 35.4°				
Element Line	Element Weight %	Element Weight % Error ±	Atom Weight %	Atom Weight % Error ±
C K	7.81	0.88	13.01	1.49
O K	39.53	1.12	49.44	1.39
F K	2.36	0.68	2.49	0.72
Mg K	2.13	0.16	1.75	0.13
Al K	27.37	0.45	20.30	0.33
Si K	3.83	0.21	2.73	0.15
P K	8.73	0.42	5.64	0.27
S K	4.24	0.21	2.65	0.13
Ca K	4.01	0.24	2.00	0.12
Total	100	---	100	---

EVID 15113-0-A1 Valve Stem Filter Exterior Near Filter Face Facing Cylinder 3 O'clock Quadrant 2: First Scan High Magnification



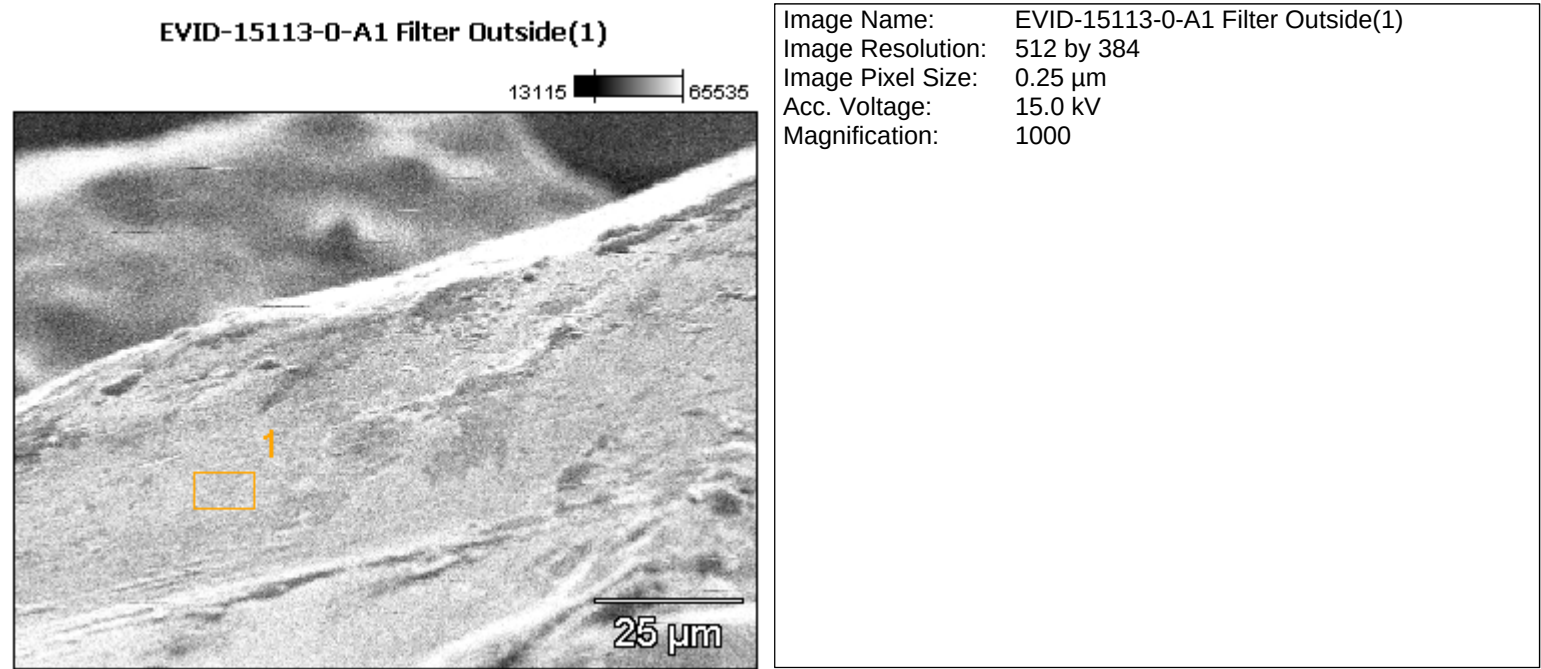
Full scale counts: 266

EVID-15113-0-A1 Filter(1)_pt1



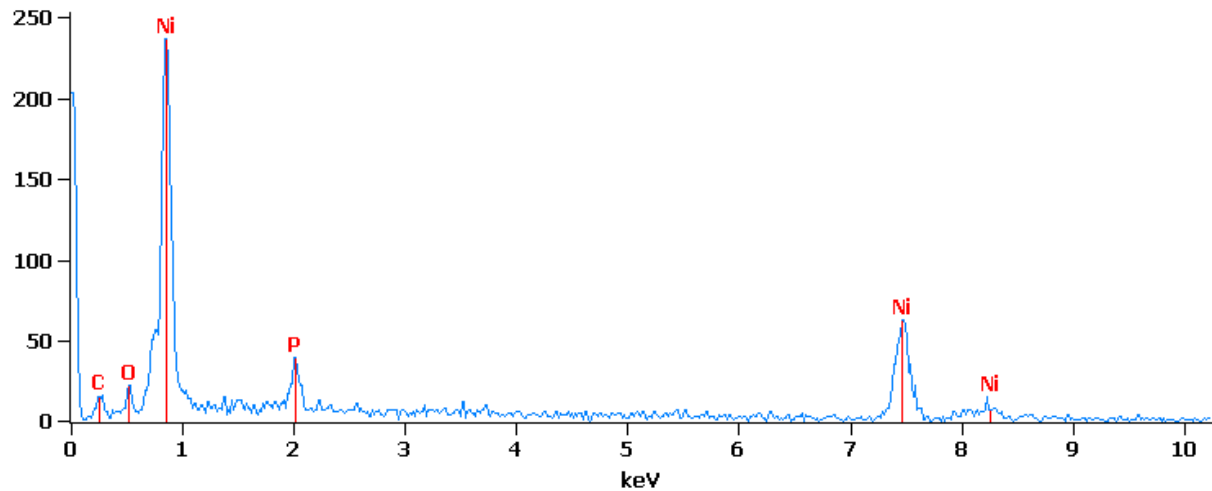
Filter Fit				
Chi² value: 1.369				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 15.0 kV Take Off Angle: 44.6°				
Element Line	Element Weight %	Element Weight % Error ±	Atom Weight %	Atom Weight % Error ±
C K	8.88	1.22	31.46	4.40
P K	3.89	0.44	5.34	0.61
Ni K	87.22	5.16	63.19	3.73
Total	100	---	100	---

EVID 15113-0-A1 Valve Stem Filter Exterior Near Filter Face Facing Cylinder 3 O'clock Quadrant 2: Second Scan High Magnification



Full scale counts: 237

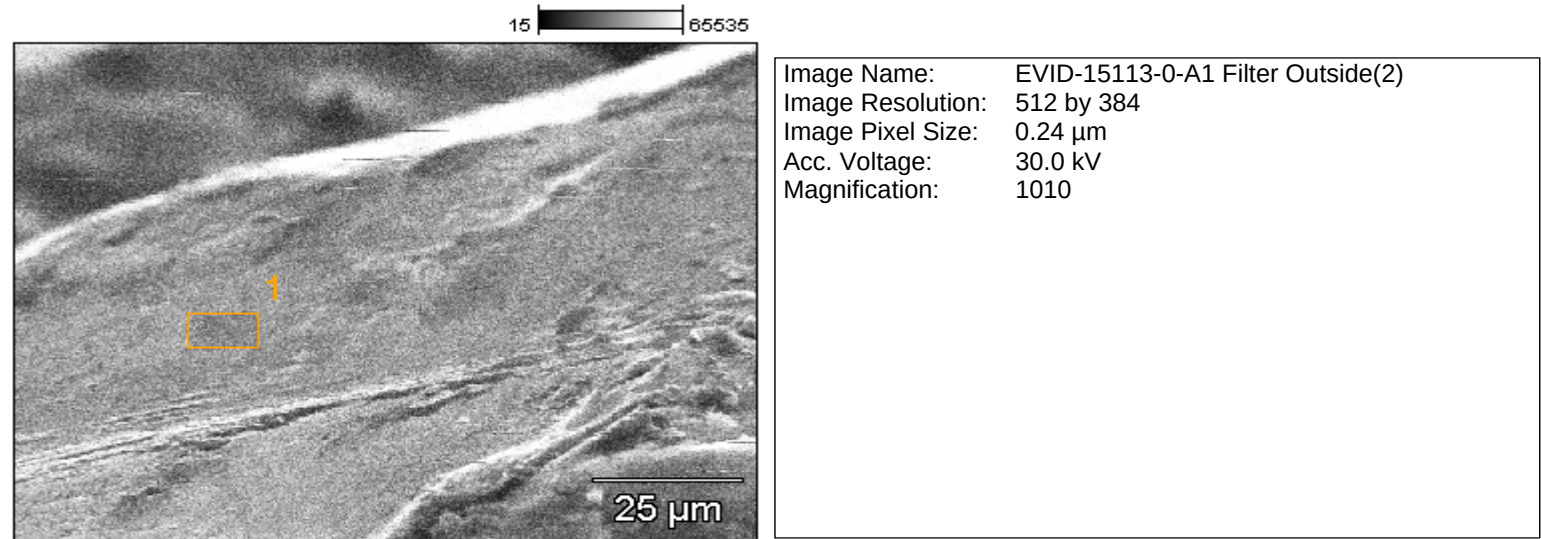
EVID-15113-0-A1 Filter Outside(1)_pt1



Filter Fit				
Chi² value: 1.611				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 15.0 kV Take Off Angle: 45.0°				
Element Line	Element Weight %	Element Weight % Error ±	Atom Weight %	Atom Weight % Error ±
C K	7.38	0.83	26.08	2.93
O K	2.32	0.42	6.15	1.14
P K	3.84	0.30	5.26	0.40
Ni K	86.46	5.16	62.50	3.71
Total	100	---	100	---

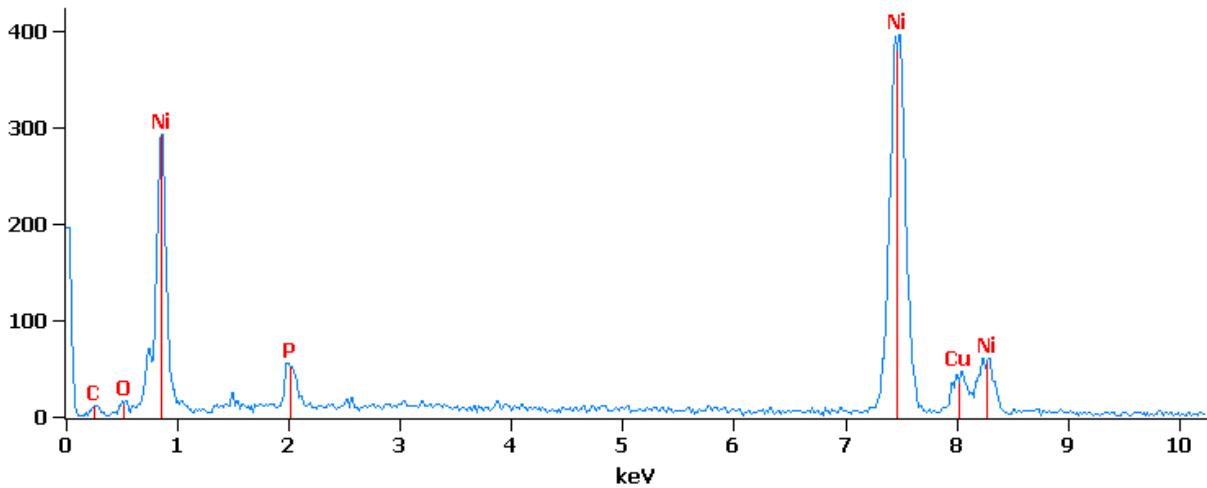
EVID 15113-0-A1 Valve Stem Filter Exterior Near Filter Face Facing Cylinder 3 O'clock Quadrant 2: Second High Magnification

EVID-15113-0-A1 Filter Outside(2)



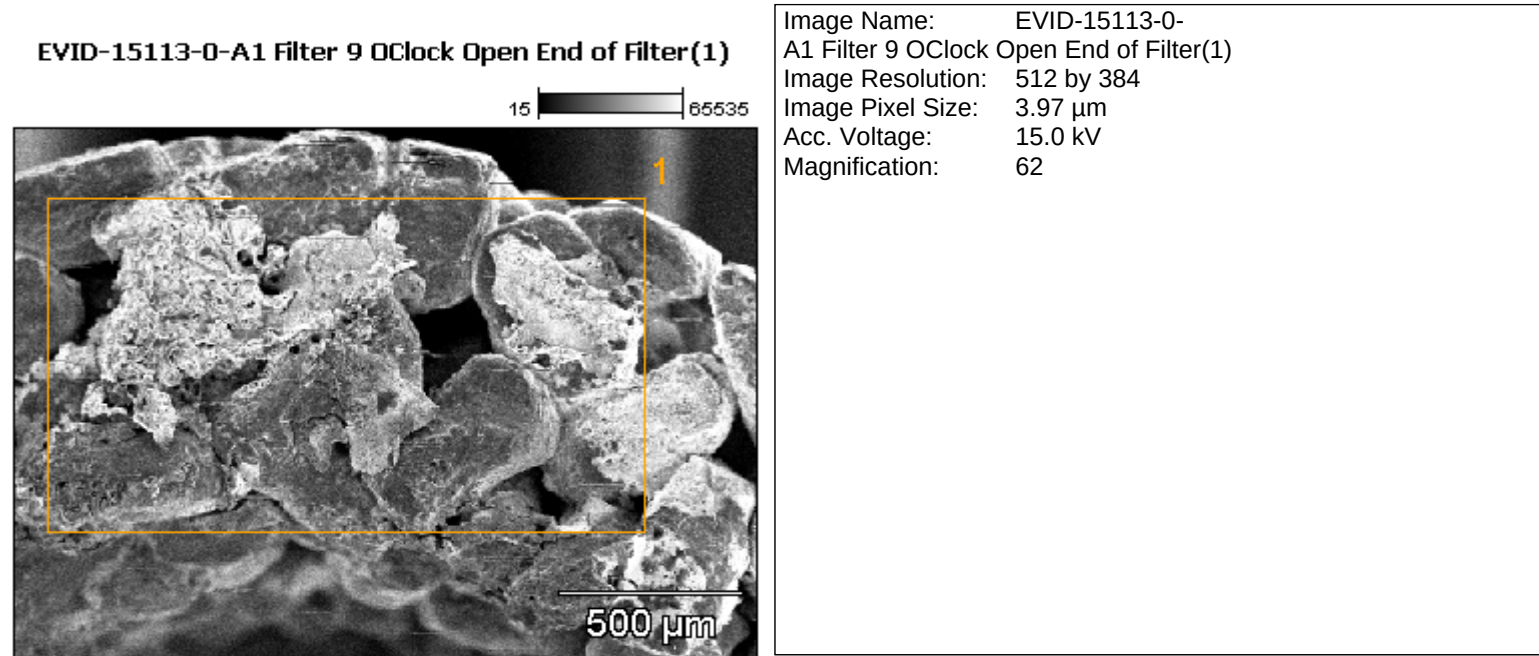
Full scale counts: 396

EVID-15113-0-A1 Filter Outside(2)_pt1

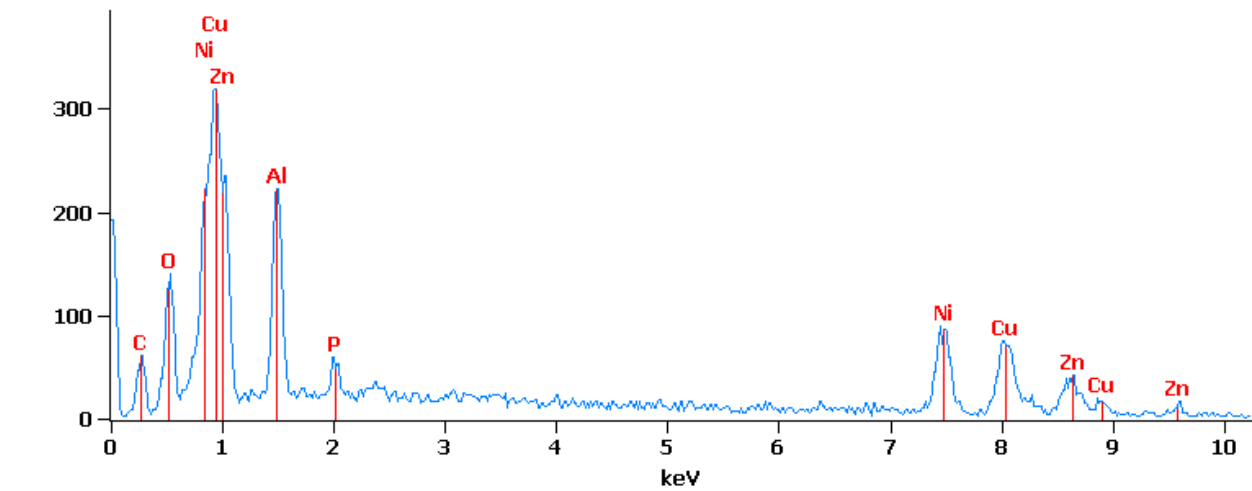


Filter Fit				
Chi² value: 1.236				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 30.0 kV Take Off Angle: 44.7°				
Element Line	Element Weight %	Element Weight % Error ±	Atom Weight %	Atom Weight % Error ±
C K	10.97	1.49	34.96	4.58
O K	2.89	0.57	6.91	1.38
P K	4.08	0.23	5.04	0.29
Ni K	73.72	1.52	48.07	0.99
Cu K	8.34	0.67	5.03	0.40
Total	100	---	100	---

EVID 15113-0-A1 Valve Stem Filter Face Facing Cylinder 9 O’Clock Quadrant 4: First Scan

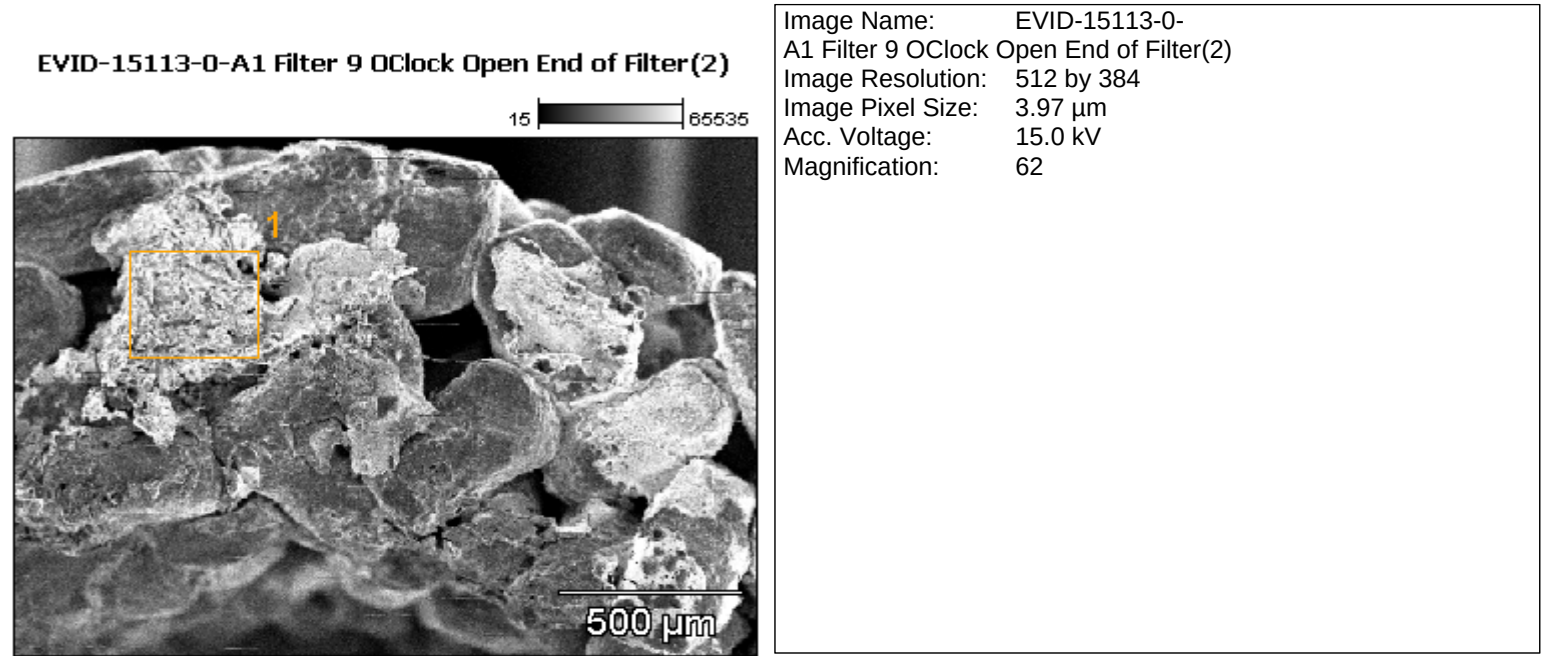


Full scale counts: 319 EVID-15113-0-A1 Filter 9 OClock Open End of Filter(1)_pt1

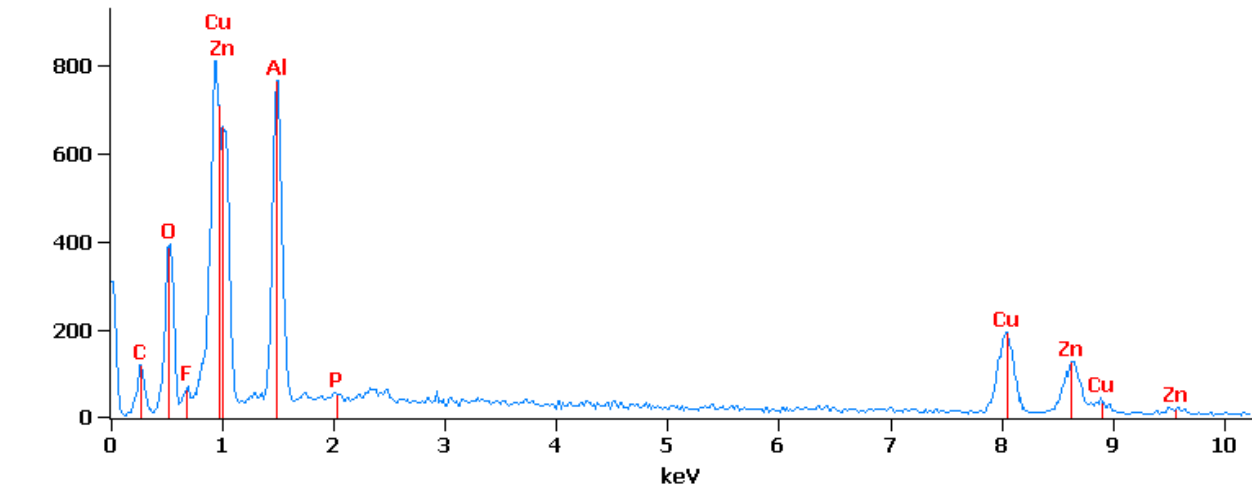


Filter Fit				
Chi² value: 1.196				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 15.0 kV Take Off Angle: 35.6°				
Element Line	Element Weight %	Element Weight % Error ±	Atom Weight %	Atom Weight % Error ±
C K	10.24	0.60	29.24	1.69
O K	9.33	0.40	20.00	0.85
Al K	8.13	0.24	10.34	0.30
P K	1.14	0.10	1.26	0.11
Ni K	22.59	1.52	13.20	0.89
Cu K	30.40	2.43	16.41	1.31
Zn K	18.18	2.91	9.54	1.53
Total	100	---	100	---

EVID 15113-0-A1 Valve Stem Filter Face Facing Cylinder 9 O’Clock Quadrant 4: First Scan

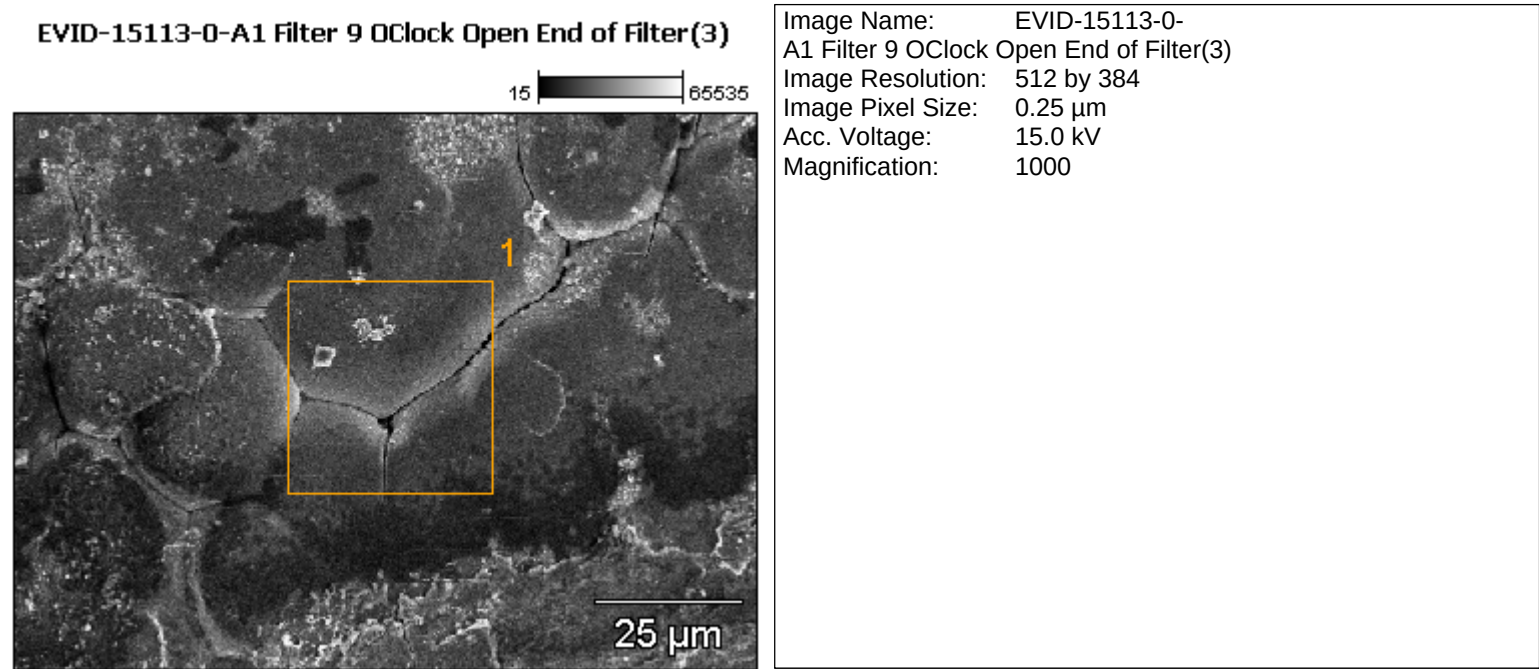


Full scale counts: 811 EVID-15113-0-A1 Filter 9 O’clock Open End of Filter(2)_pt1

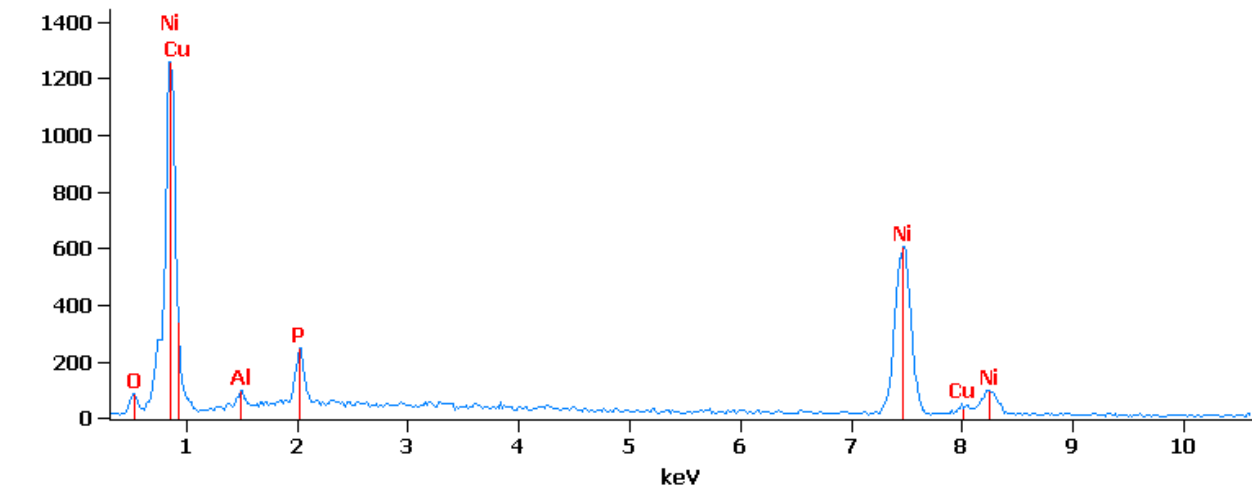


Filter Fit				
Chi² value: 1.196				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 15.0 kV Take Off Angle: 35.6°				
Element Line	Element Weight %	Element Weight % Error ±	Atom Weight %	Atom Weight % Error ±
C K	8.94	0.39	24.29	1.05
O K	12.86	0.33	26.24	0.68
F K	1.10	0.20	1.88	0.33
Al K	11.89	0.18	14.38	0.21
P K	0.26	0.05	0.28	0.06
Cu K	35.18	1.48	18.07	0.76
Zn K	29.77	1.99	14.86	1.00
Total	100	---	100	---

EVID 15113-0-A1 Valve Stem Filter Face Facing Cylinder 9 O’Clock Quadrant 4: Second Scan High Magnification

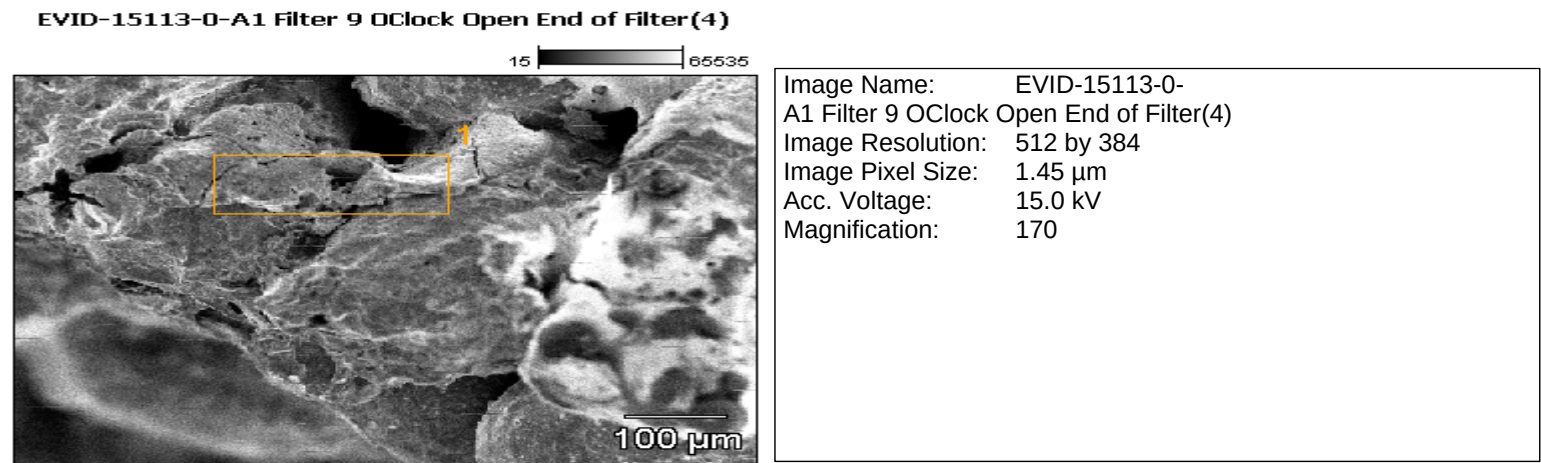


Full scale counts: 1258 EVID-15113-0-A1 Filter 9 OClock Open End of Filter(3)_pt1

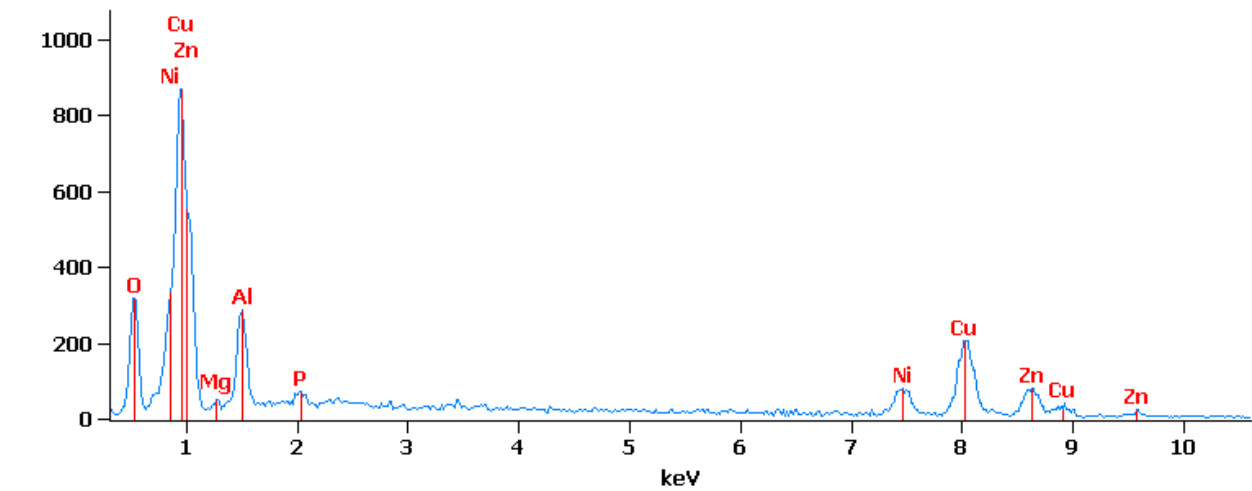


Filter Fit				
Chi² value: 1.436				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 15.0 kV Take Off Angle: 35.8°				
Element Line	Element Weight %	Element Weight % Error	Atom Weight %	Atom Weight % Error
C K	3.64	0.26	14.78	1.06
O K	1.25	0.16	3.80	0.48
Al K	0.73	0.07	1.32	0.12
P K	2.65	0.08	4.18	0.13
Ni K	87.26	1.58	72.50	1.31
Cu K	4.47	0.73	3.43	0.56
Total	100	---	100	---

EVID 15113-0-A1 Valve Stem Filter Face Facing Cylinder 9 O’Clock Quadrant 4: Second Scan Magnified

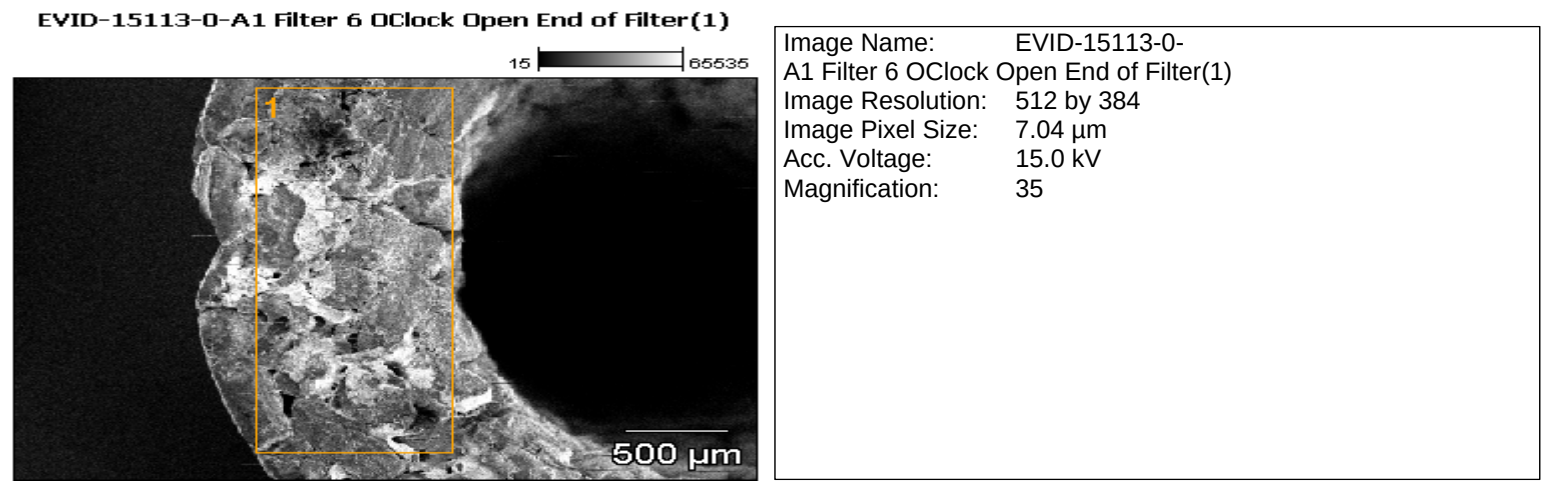


Full scale counts: 867 EVID-15113-0-A1 Filter 9 OClock Open End of Filter(4)_pt1

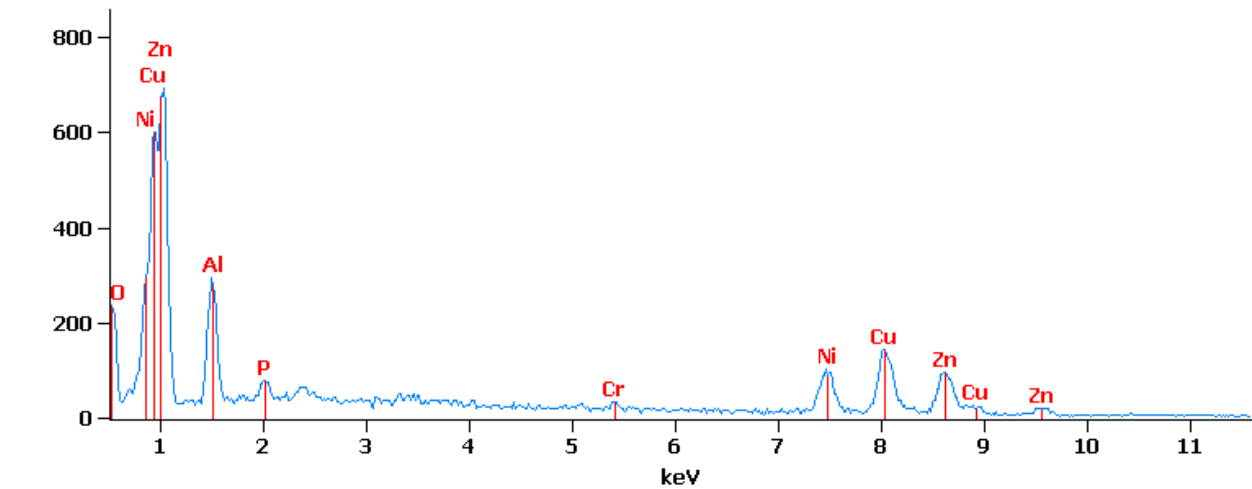


Filter Fit				
Chi² value: 0.771				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 15.0 kV Take Off Angle: 35.8°				
Element Line	Element Weight %	Element Weight % Error	Atom Weight %	Atom Weight % Error
C K	5.66	0.41	18.17	1.30
O K	10.95	0.29	26.36	0.69
Mg K	0.13	0.12	0.21	0.19
Al K	5.00	0.11	7.14	0.16
P K	0.74	0.11	0.92	0.14
Ni K	10.87	0.94	7.14	0.61
Cu K	45.45	1.87	27.56	1.13
Zn K	21.18	2.13	12.49	1.26
Total	100	---	100	---

EVID 15113-0-A1 Valve Stem Filter Face Facing Cylinder 6 O’Clock Quadrant 3: First Scan

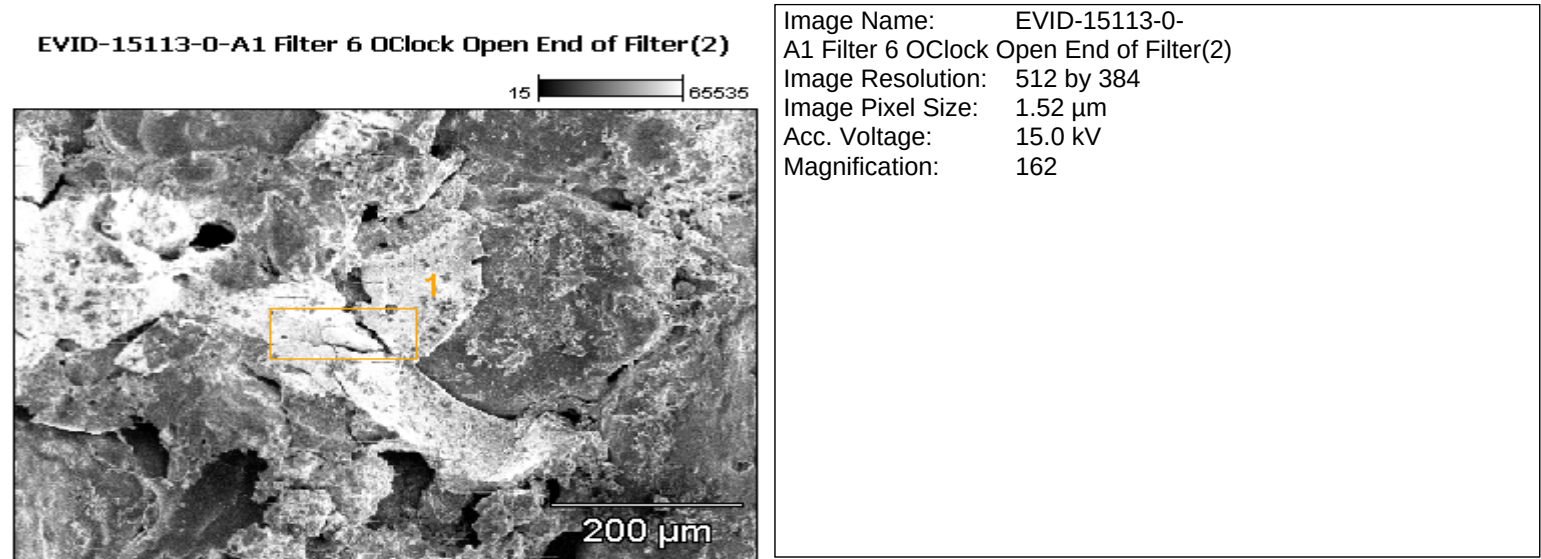


Full scale counts: 691 EVID-15113-0-A1 Filter 6 OClock Open End of Filter(1)_pt1

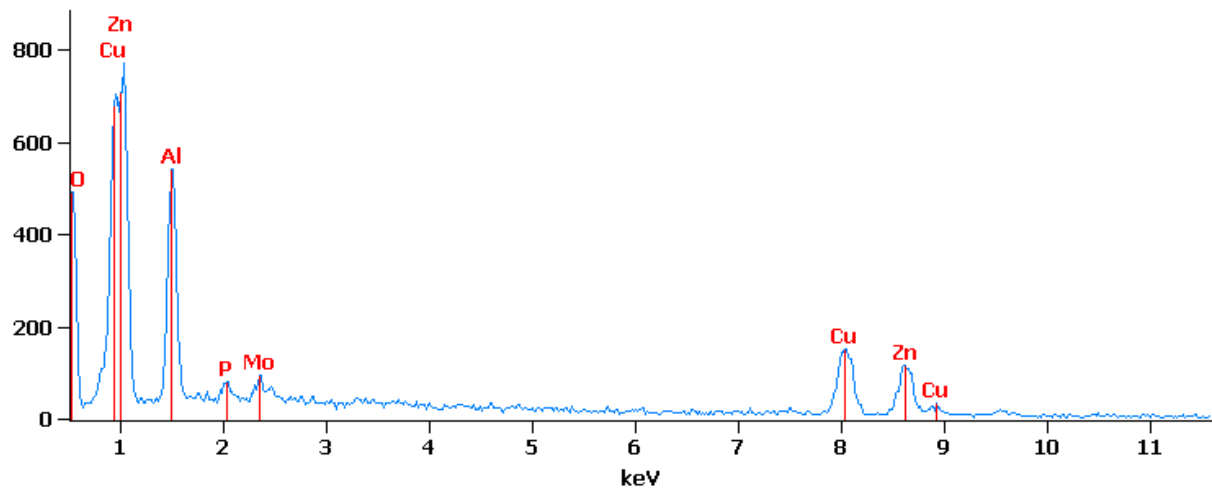


Filter Fit				
Chi² value: 0.845				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 15.0 kV Take Off Angle: 35.8°				
Element Line	Element Weight %	Element Weight % Error	Atom Weight %	Atom Weight % Error
C K	9.05	0.33	28.02	1.02
O K	7.62	0.37	17.72	0.85
Al K	6.00	0.16	8.27	0.22
P K	0.85	0.07	1.02	0.09
Cr K	0.64	0.15	0.46	0.11
Ni K	13.06	0.97	8.27	0.61
Cu K	31.39	0.75	18.37	1.03
Zn K	31.40	1.41	17.86	0.80
Total	100	---	100	---

EVID 15113-0-A1 Valve Stem Filter Face Facing Cylinder 6 O’Clock Quadrant 3: Second Scan Magnified

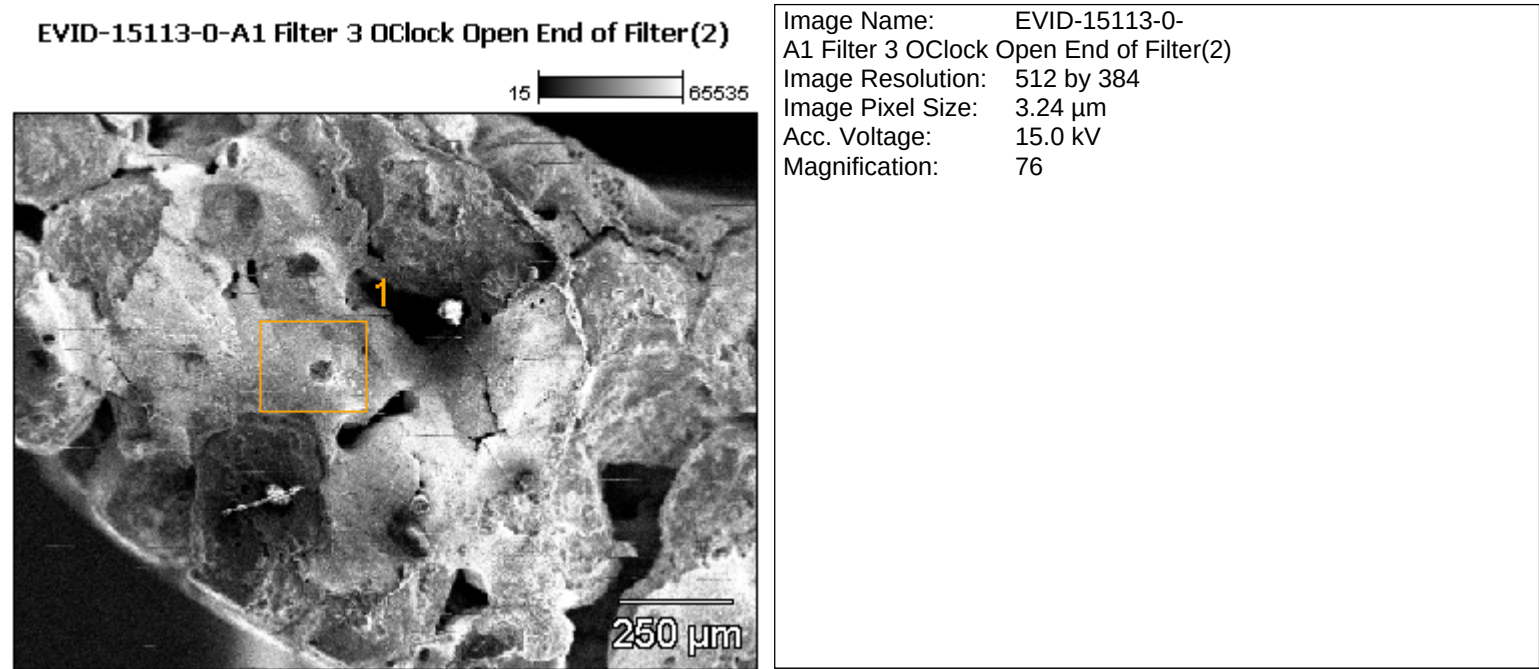


Full scale counts: 771 EVID-15113-0-A1 Filter 6 OClock Open End of Filter(2)_pt1

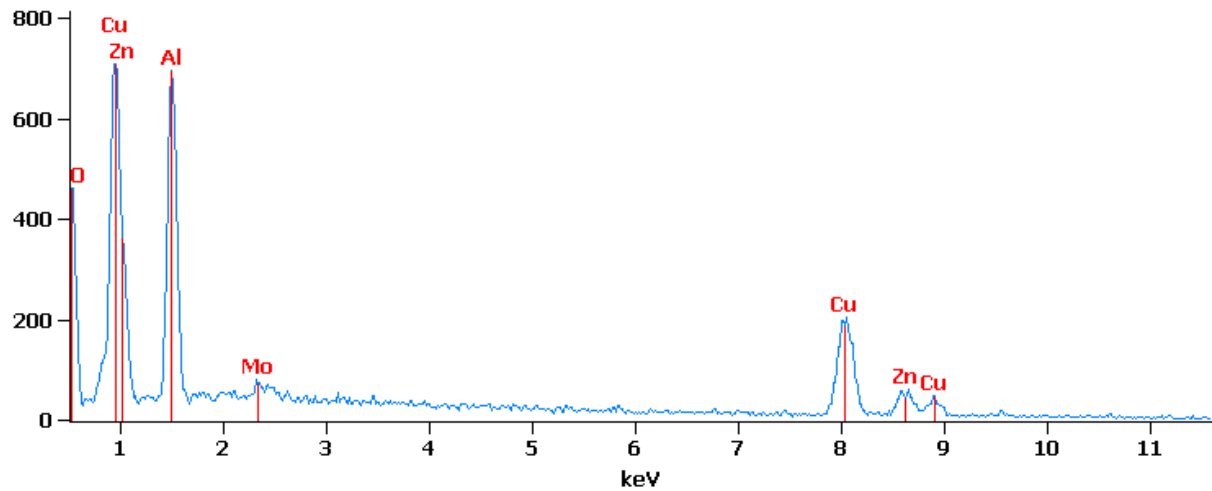


Filter Fit				
Chi² value: 0.949				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 15.0 kV Take Off Angle: 35.6°				
Element Line	Element Weight %	Element Weight % Error	Atom Weight %	Atom Weight % Error
C K	5.95	0.28	16.74	0.80
O K	16.80	0.31	35.46	0.66
Al K	9.95	0.18	12.45	0.23
P K	0.61	0.07	0.67	0.08
Cu K	33.78	1.64	17.95	0.87
Zn K	31.29	2.16	16.16	1.11
Mo L	1.61	0.30	0.57	0.11
Total	100	---	100	---

EVID 15113-0-A1 Valve Stem Filter Face Facing Cylinder 3 O’Clock Quadrant 2: First Scan

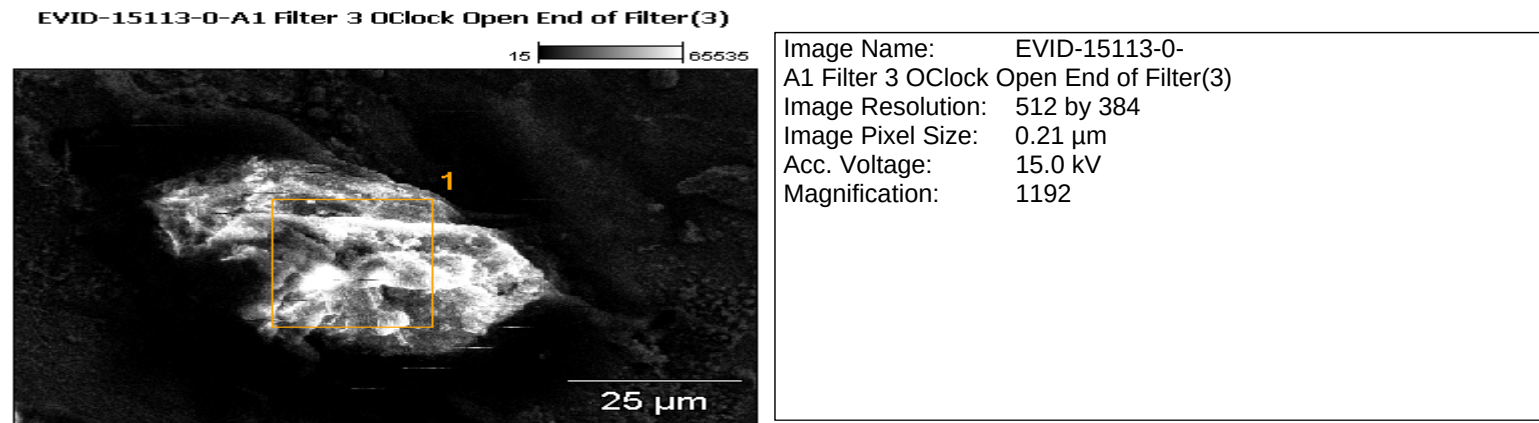


Full scale counts: 708 EVID-15113-0-A1 Filter 3 OClock Open End of Filter(2)_pt1

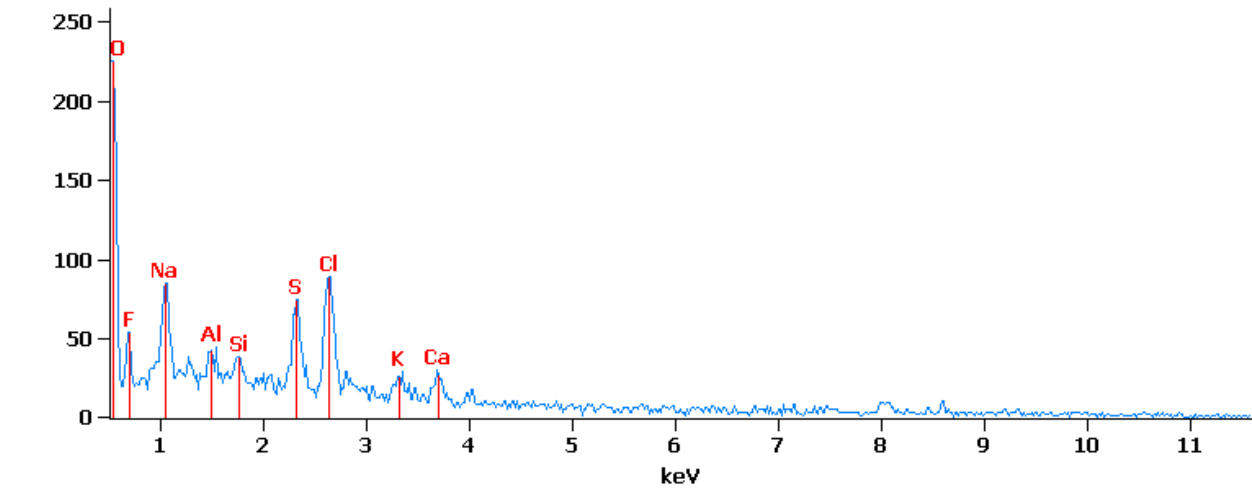


Filter Fit				
Chi² value: 1.204				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 15.0 kV Take Off Angle: 35.5°				
Element Line	Element Weight %	Element Weight % Error	Atom Weight %	Atom Weight % Error
C K	6.29	0.32	17.39	0.88
O K	15.56	0.31	32.31	0.65
Al K	13.89	0.21	17.10	0.26
Cu K	49.28	1.87	25.76	0.97
Zn K	13.89	2.00	7.06	1.01
Mo L	1.09	0.20	0.38	0.07
Total	100	---	100	---

EVID 15113-0-A1 Valve Stem Filter Face Facing Cylinder 3 O’Clock Quadrant 2: Second Scan High Magnification

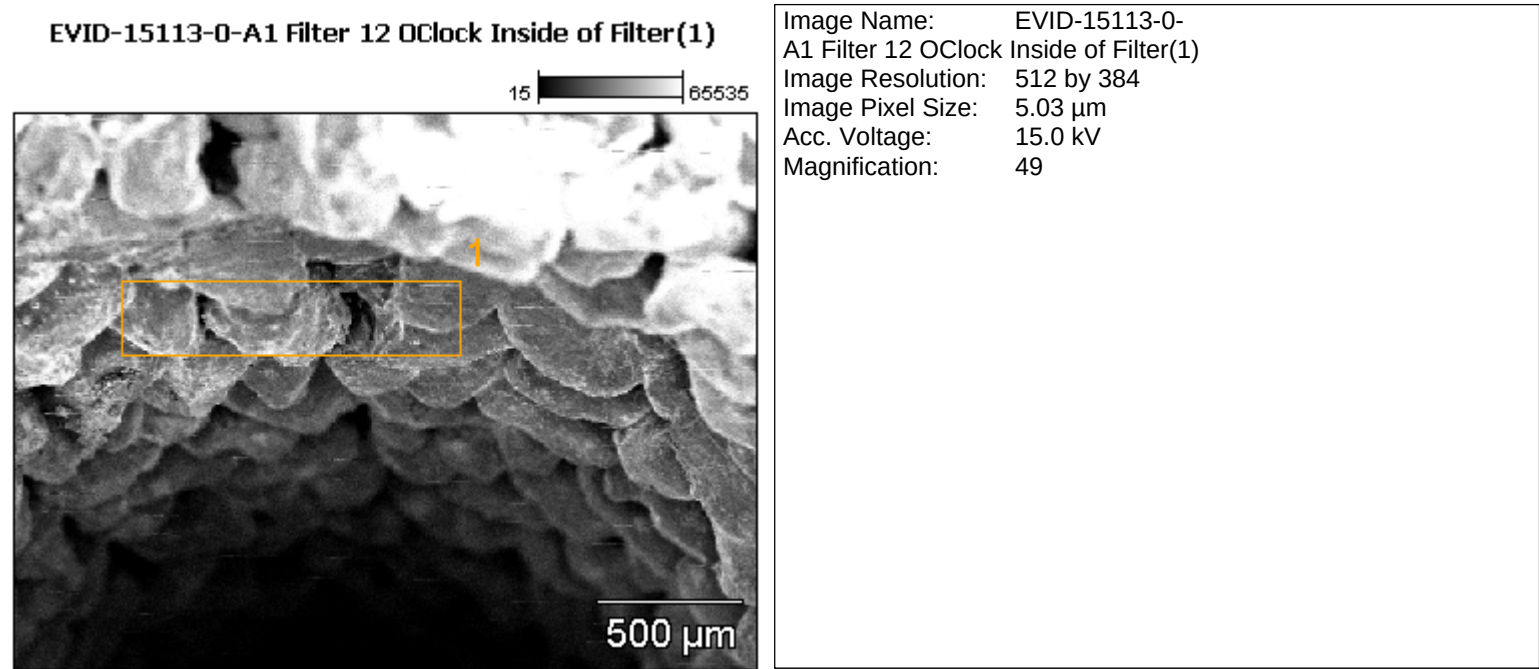


Full scale counts: 225 EVID-15113-0-A1 Filter 3 OClock Open End of Filter(3)_pt1

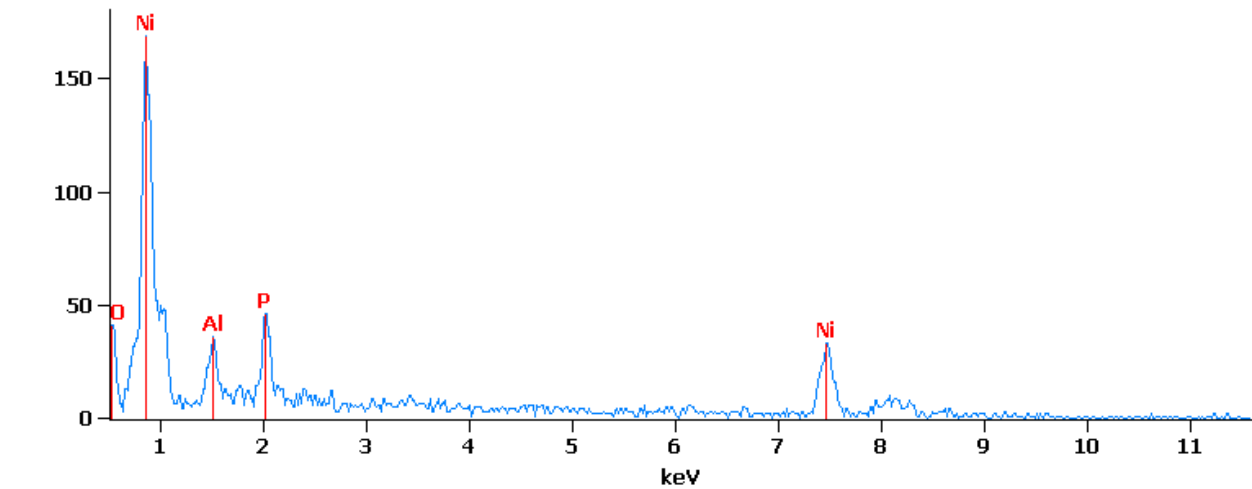


Filter Fit				
Chi² value: 0.823				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 15.0 kV Take Off Angle: 35.6°				
Element Line	Element Weight %	Element Weight % Error	Atom Weight %	Atom Weight % Error
C K	67.89	0.69	76.01	0.77
O K	23.17	0.69	19.48	0.57
F K	2.44	0.38	1.73	0.27
Na K	1.28	0.13	0.75	0.08
Al K	0.30	0.05	0.15	0.02
Si K	0.24	0.05	0.12	0.02
S K	1.24	0.12	0.52	0.05
Cl K	2.21	0.09	0.84	0.03
K K	0.43	0.07	0.15	0.02
Ca K	0.78	0.08	0.26	0.03
Total	100	---	100	---

EVID 15113-0-A1 Valve Stem Filter Interior 12 O’Clock Quadrant 1: First Scan Magnified

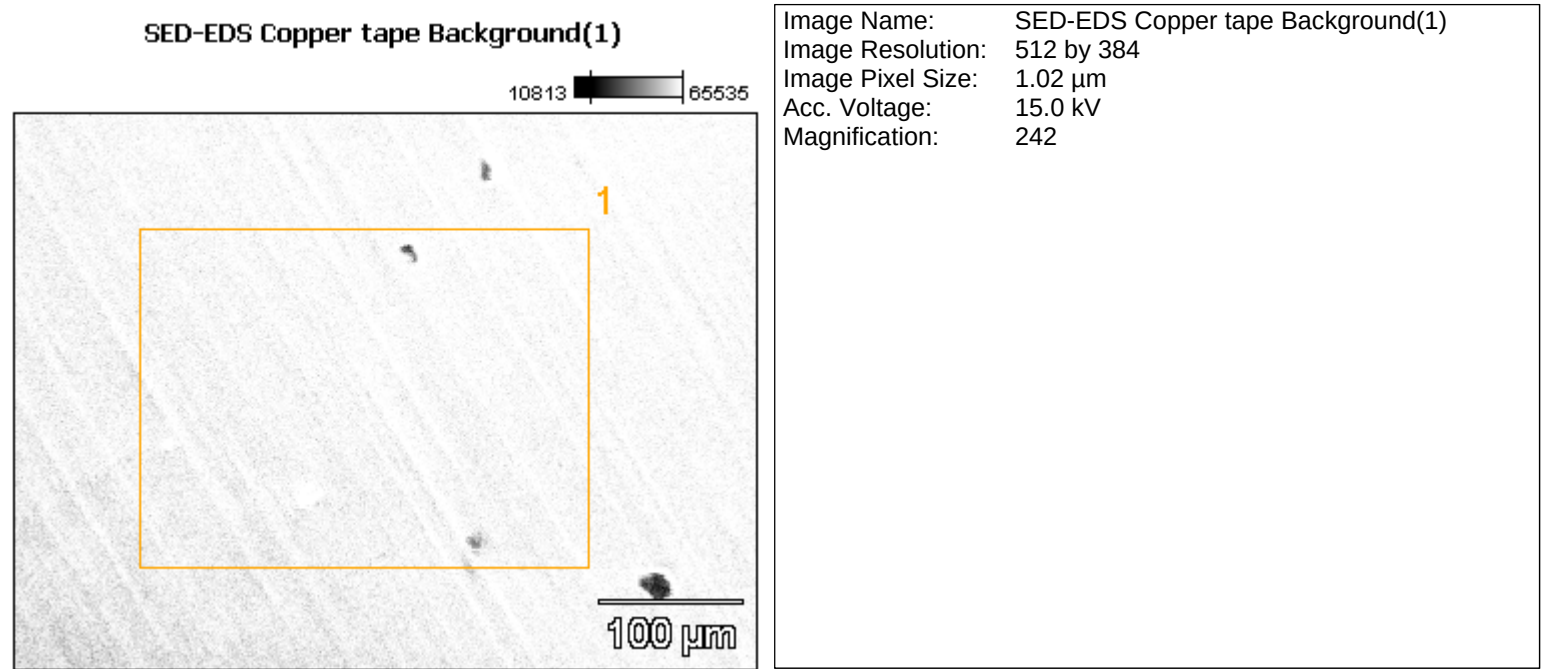


Full scale counts: 169 EVID-15113-0-A1 Filter 12 OClock Inside of Filter(1)_pt1



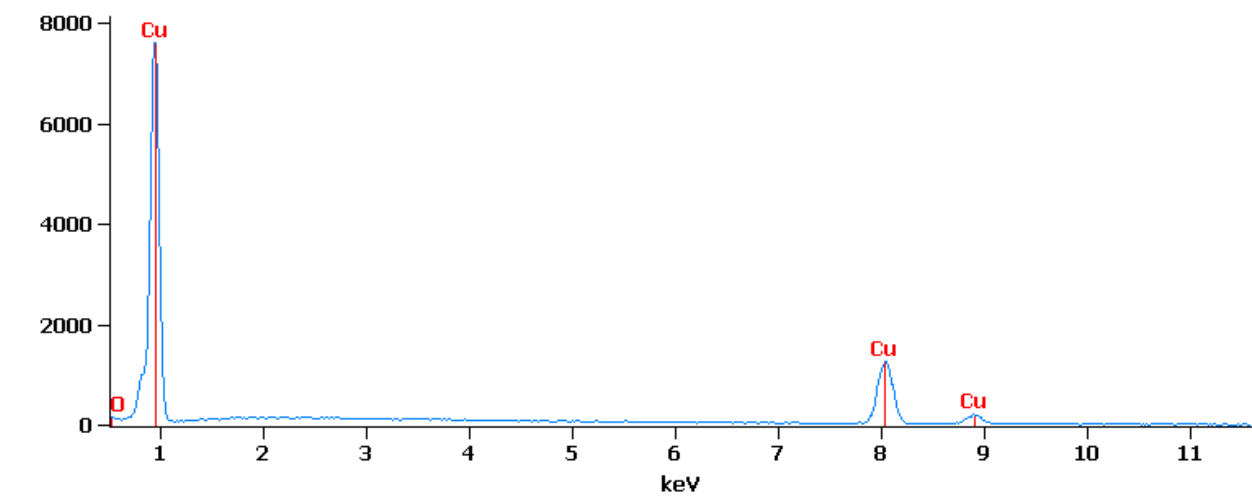
Filter Fit				
Chi² value: 3.303				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 15.0 kV Take Off Angle: 35.0°				
Element Line	Element Weight %	Element Weight % Error	Atom Weight %	Atom Weight % Error
C K	17.56	2.03	37.85	4.38
O K	17.49	1.56	28.30	2.48
Al K	5.37	0.60	5.15	0.56
P K	6.17	0.64	5.15	0.54
Ni K	53.41	5.25	23.55	2.30
Total	100	---	100	---

Copper Tape Background



Full scale counts: 7610

SED-EDS Copper tape Background(1)_pt1



Filter Fit				
Chi² value: 3.007				
Correction Method: Proza (Phi-Rho-Z)				
Acc.Voltage: 15.0 kV Take Off Angle: 36.5°				
Element Line	Element Weight %	Element Weight % Error	Atom Weight %	Atom Weight % Error
C K	4.54	0.18	19.88	0.82
O K	0.46	0.06	1.50	0.21
Cu K	95.00	1.25	78.62	1.03
Total	100	---	100	---