SUPERCritical WATER OXIDATION
Benchscale Testing
Metallurgical Analysis Report

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February 1993

Idaho National Engineering Laboratory
EG&G Idaho, Inc.
Idaho Falls, Idaho

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Prepared for the
U.S. Department of Energy
Office of Technology Development
Under DOE Idaho Field Office
Contract DE-AC07-76ID01570

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ABSTRACT

This report describes metallurgical evaluation of witness wires from a series of tests using supercritical water oxidation (SCWO) to process cutting oil containing a simulated radionuclide. The goal of the tests was to evaluate the technology's ability to process a highly chlorinated waste representative of many mixed waste streams generated in the DOE complex. The testing was conducted with a bench-scale SCWO system developed by the Model Development Corporation. Significant test objectives included process optimization for adequate destruction efficiency, tracking the radionuclide simulant and certain metals in the effluent streams, and assessment of reactor material degradation resulting from processing a highly chlorinated waste. The metallurgical evaluation described herein includes results of metallographic analysis and Scanning Electron Microscopy analysis of witness wires exposed to the SCWO environment for one test series.
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Introduction  
Metallographic analysis and Scanning Electron Microscopy of Inconel 625 weld wires have been completed. These wires were exposed to the corrosive environment of the Supercritical Water Oxidation (SCWO) reactor.

Three weld wires were examined. All three wires were placed in the reactor and sensitized. The sensitizing heat treatment was 800°C for 24 hours. This sensitization heat treatment was inaccurately considered a passivation treatment. After the sensitizing heat treatment, one wire was removed from the reactor and the other two were exposed to the reactor environment for 20 and 40 hours respectively.

After exposure the wires were removed from the reactor. Between 68 and 80 samples were cut from each wire at various locations along the wire length and then prepared for metallographic examination.

The objective of the study was to get an estimate of the corrosion rate at various locations in the reactor, to determine the type of corrosive attack, to determine the effect of the sensitization heat treatment, and to determine the composition and effect of deposits that formed on the wires.

Nominal Corrosion Rate  
Method for Determining the Corrosion Rate  
Gravimetric (weight loss) methods were not used to determine the corrosion rate because it was not possible to place small corrosion coupons throughout the reactor. As an alternative, the diameters of the weld wires placed in the reactor were measured using the metallograph. The three weld wires ran the entire length of the reactor and as such allowed for an estimate of the corrosion rate in all areas of the reactor.
Determining corrosion rates using thickness measurements are not as accurate as gravimetric methods. However, it is possible to get an estimate of corrosion rate if the rates are high.

**Statistical Error of the Corrosion Rates**

The nominal thickness of the original weld wires before heat treating was 0.0625 inch. Measurements of the diameter of the sensitized wire (the wire that was not exposed to the corrosive fluids) at 68 locations along the length of the wire, gave a mean value of 0.0612 inches. The standard deviation for the diameter was 0.0003 inches. Therefore, the 95% confidence interval for the true mean diameter was from 0.0606 inches to 0.0618 inches.

The units used for the calculated corrosion rates were mpy, or 0.001"/year. The calculated confidence interval is equivalent to +/- 70 mpy. Therefore, the reported corrosion rates could vary by as much as 70 mpy. Reported corrosion rates less than 70 mpy could easily be attributed to measurement errors and variations in the wire diameter, not actual corrosion loss.

**Corrosion Rate Calculations**

The estimated corrosion rates along with the estimated metal loss for each wire are listed in Tables 1 and 2. The corrosion rates are also plotted in Figures 1 and 2. In several areas of the reactor the corrosion rates were high enough that the metal loss could be attributed to corrosion and not just variations in the weld wire diameter.

The corrosion rates were extremely high from about the 12 foot area of the reactor to the 26 foot area. Figures 3 through 5 show the progressive attack of the wires during operation of the reactor. The areas of active corrosion were between about the 10 and 37 foot area of the reactor and at the end of the reactor. At the 54 foot area the wire exposed for 20 hours experienced rates in excess of 2000 mpy (Figure 6). A similar event occurred at the 53.5 foot area and the 54 foot area on the wire that was exposed for 40 hours.

Materials with general corrosion rates less than 2 mpy are generally considered to have good corrosion resistance and considered compatible with the environment. As a common rule, corrosion rates in excess of 20 mpy are typically considered unacceptably high for typical engineering structures fabricated from corrosion resistant alloys.

The corrosion rates from the 55 to 60 foot area of the reactor could not be calculated because the wires were not available.
Effect of Deposit on Corrosion Rate

In some areas the corroded wires were irregularly shaped indicating localized corrosion (Figures 6 and 7). The corrosion rates seemed to be effected by deposits that formed on the wires. These deposits seemed to form a protective coating and hence slow down the corrosion. Apparently, corrosion would occur in a specific area then layers of scale would begin to form on the wires. The corrosion would slow (or stop) until the scale spalled off. This would explain why, in general, the corrosion rates for the wire exposed to the reactor for 20 hours had higher corrosion rates than the wire exposed for 40 hours. The appearance of the scale was similar to sedimentary rocks with distinct layers (Figure 8).

Type of Corrosive Attack

Most of the areas of the wires experienced general corrosion. However, some areas of the wires experienced intergranular attack (Figure 9). Intergranular attack is a form of localized corrosion where corrosion occurs more rapidly at the grain boundaries.

The degree of intergranular attack varied within short distances. Wire segments adjacent to each other experienced different degrees of intergranular attack (compare Figures 10, 11, and 12). Since the degree of sensitization would be relatively uniform, the variance in intergranular attack would indicate different chemical environments within short distances along the reactor.

Microstructure

The microstructure of the weld wires showed equiaxed grains with twinning, typical for Inconel 625 (Figure 13). This microstructure is typical of the solution annealed condition (that is, heated to about 2100°F and water quenched). There were no obvious indications of secondary precipitates caused by the sensitization heat treatment. However, there were some secondary phase precipitates (or maybe just oxide scale) concentrated near the edges of the weld wires (Figure 14). There was no indication of grain distortion indicative of cold working.

The grain size varied along the length of the weld wire. This difference in size occurred within inches. Figure 15 shows the difference in grain size between two samples that were taken one half inch apart from each other.

The microstructure of the weld wire was similar to any plate, pipe, or tubing that would have been hot worked, solution annealed, and sensitized. The nominal chemical composition of Inconel 625 weld wire is identical to the
nominal chemical composition of Inconel 625 plate. Similar composition and similar microstructure mean that the weld wires most likely behaved the same as the reactor walls.

Scanning Electron Microscopy

The chemical composition of the deposits were analyzed using Energy Dispersive X-ray Spectrometry (EDS) of the Scanning Electron Microscope (SEM). The composition was determined for the deposits at the 6.5 to 7.0 foot location, the 19.5 to 20 foot location, and the 53.5 to 54 foot location. Twenty six different spectra were run.

In general, the deposits on the wires included oxides of the elements that compose Inconel 625 (see spectra 1-4 and Figures 16-17). In addition to these oxides, cerium, zinc, lead, chloride, and sulfur were found in the deposits. Sulfur was only found in spectra 5.

Of the three wire locations analyzed, the cerium and lead were concentrated at the 6.5 to 7 foot level (see spectra 8, 12, and 13 and Figures 18-21). Small amounts of cerium and lead were also detected at the 19.5 to 20 foot location and the 53.5 to 54 foot location (see spectra 6, 7, 17, and 18 and Figures 22-25). Zinc was only detected at the 53.5 to 54 foot location (see spectra 17, 18, 20, and 21 and Figures 24-27). Chloride was detected in all three analyzed areas (Spectra 7-11 and 16-18). High chloride nodules were found imbedded in the deposit at the 6.5 to 7 foot area (Spectra 10, Figure 19).

Depending on the valence, small amounts of cerium in the waste would accelerate the corrosion. Cerium (+4) is a strong oxidizer. The oxidation potential of Ce$^{3+}$ to Ce$^{4+}$ is -1.61 volts. The presence of cerium could also enhance intergranular attack.

The composition of the oxide layer on the sensitized wire consisted of high concentrations of chromium oxide (Spectra 22-25, Figures 28-30). There was one area on the sensitized wire that was composed of nickel oxide (Spectrum 26).

The high concentrations of aluminum were most likely due to the polishing compound used for metallographic sample preparation.

Conclusions

1. Both intergranular and general attack occurs throughout the reactor. Intergranular attack seems to be inconsistent in its location. Two segments of wire 1/2 inch away from each other could experience general and
intergranular attack. The intergranular attack was most likely enhanced by the sensitization heat treatment and the presence of cerium in the waste.

2. The deposit that forms on the reactor provides partial protection from the corrosive atmosphere. However, this deposit is not tightly adhering and cannot be counted on to reduce corrosion.

3. Inconel 625 experiences heavy corrosion. In general the area of extensive corrosion is between the 12 foot and 26 feet area of the reactor and at the end of the reactor in the cool down section.

4. The sensitizing heat treatment that the reactor experienced was detrimental to the corrosion resistance of Inconel 625 (Figure 31).

5. The deposits on the wires consisted of oxides of the elements that compose Inconel 625. The deposits also included cerium, lead, zinc, chloride, and sulfur.

Recommendations

1. Inconel 625 is not a suitable alloy for the tested environment. A different alloy should be used. Depending on the reactor environment Hastelloy C-22 or Hastelloy G-30 could provide better corrosion resistance. Hastelloy C-22 has performed well in halide solutions both in reducing environments and those with low levels of oxidants. Hastelloy G-30 has performed well in oxidizing solutions with halide contaminants.

2. If Inconel 625 is used for continued pilot plant tests, do not sensitize the material prior to running the tests.

3. Replace the reactor prior to any further testing.

4. If possible, it would be advantageous to examine the reactor parts in the heavily corroded areas.

B. C. Norby, Senior Engineer
Materials Development and Technology

Attachment
/dj
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Table 1
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Variance 9.25x10^8
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Supercritical Water Oxidation Thickness Measurements
Inconel 625 Wire Exposed for 40 Hours

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SCWO Reactor Corrosion

Inconel 625  40 hour test

Figure 1
SCWO Reactor Corrosion

Inconel 625  20 hour test

Corrosion rate (mpy)

Reactor Location (feet)

Figure 2
Figure 3

Original diameter of sensitized wire between 19.5 and 20.0 ft. (50X as polished)

Figure 4

Wire diameter after 20 hours exposure in the reactor between 19.5 and 20 ft. (50X as polished)
Figure 5

Wire diameter after 40 hours exposure to the reactor between 19.5 and 20 ft. (50X as polished)

Figure 6

Irregular wire diameter after 20 hours exposure in the reactor between 54 and 54.5 ft. (50X as polished)
Figure 7

Irregular wire diameter after exposure in the reactor for 40 hours between 19.5 and 20 ft. (50X as polished)

Figure 8

Scale deposit on wire after exposure in the reactor for 40 hours between 19.5 and 20 ft. (400X as polished)
Figure 9

Intergranular attack of wire after exposure in the reactor for 20 hours between 54 and 54.5 ft. (400X as polished)

Figure 10

Intergranular attack of wires after exposure in the reactor for 40 hours between 18.5 and 19 ft. (100X as polished)
Figure 11

General attack of wires exposed in the reactor for 40 hours between 19 and 19.5 ft. (100X as polished)

Figure 12

Intergranular attack of wires after exposure in the reactor for 40 hours between 19.5 and 20 ft. (100X as polished)
the thin oxide layer and possible precipitates (400X as polished)

Edges of sensitized wires located between 19.5 to 20 h. showing

Figure 14

9.5 h (100X etched with 10% HCl, 10% C2H5OH, 10% HNO3)
Microstructure of sensitized wire between 9 and

Figure 13
Sensitized wires taken from between 9 and 9.5 ft. showing the difference in grain sizes (100X etched with 10% HCl, 10% C₆H₅O₂H, 10% HNO₃)
Figure 16

SEM photograph showing scale and intergranular attack

Figure 17

SEM photograph of the wire in Figure 16 at higher magnification, the numbers indicate the location of the spectra where the chemical analysis was taken, spectra 5 is the only location where sulfur was found
Figure 18

SEM photograph showing large deposit on one side of wire and bright areas on the surface of the wire

Figure 19

SEM photograph of the scale on the wire in Figure 18 at higher magnification, spectrum 8 is high in Ce and Pb, the nodule of spectrum 10 is high in Cl
Figure 20

SEM photograph showing bright deposits on the wires

Figure 21

SEM photograph of the bright scale on the wire in Figure 20 at higher magnification, spectra 12 and 13 are high in Ce and Pb
Figure 22

SEM photograph showing dark scale deposit

Figure 23

SEM photograph of deposit on the wire in Figure 22 at higher magnification, deposit is mostly oxides of Inconel 625 with some Ce and Pb
Figure 24

SEM photograph showing continuous deposit around the wire

Figure 25

SEM photograph of deposit on the wire in Figure 24 at higher magnification, deposit is high in Cl and Zn with Ce and Pb present
Figure 26

SEM photograph showing little scale deposit

Figure 27

SEM photograph of deposit on the wire in Figure 26 at higher magnification, deposit is oxides of Inconel 625 with Zn present
Figure 28

SEM photograph of the edge of the sensitized wire, the oxide layer is high in Cr (spectrum 22)

Figure 29

SEM photograph showing large oxide area
SEM photograph of deposit on the wire in Figure 29 at higher magnification, deposit is oxides of Inconel 625, spectrum 26 is almost entirely nickel oxide.

Effect of sensitizing heat treatment on corrosion of Inconel 625 in boiling 65% HNO₃.
Accelerating voltage  
30.0 KeV

Beam - sample incidence angle  
90.0 degrees

Xray emergence angle  
35.0 degrees

Xray - window incidence angle  
0.0 degrees

STANDARDLESS EDS ANALYSIS  
(ZAF CORRECTIONS VIA MAGIC V)

<table>
<thead>
<tr>
<th>ELEMENT &amp; LINE</th>
<th>WEIGHT PERCENT</th>
<th>ATOMIC PERCENT</th>
<th>PRECISION 2 SIGMA</th>
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TOTAL 99.99

ITERATIONS 6

*NOTE: ATOMIC PERCENT is normalized to 100

**NOTE: K-RATIO = K-RATIO x R
where R = reference (standard) / reference (sample)

NORMALIZATION FACTOR: 1.000
Accelerating voltage 30.0 KeV
Beam - sample incidence angle 90.0 degrees
Xray emergence angle 35.0 degrees
Xray - window incidence angle 0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

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TOTAL 100.00

* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: 1.892
Accelerating voltage 30.0 KeV
Beam - sample incidence angle 90.0 degrees
Xray emergence angle 35.0 degrees
Xray - window incidence angle 0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

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* DETERMINED BY STOICHIOMETRY

**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: 1.889
19-Jan-1993 14:53:30     SCWD-4

Accelerating voltage  30.0 KeV
Beam - sample incidence angle  90.0 degrees
Xray emergence angle  35.0 degrees
Xray - window incidence angle  0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

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TOTAL 100.00

* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: 1.891
Accelerating voltage 30.0 KeV
Beam - sample incidence angle 90.0 degrees
X-ray emergence angle 35.0 degrees
X-ray - window incidence angle 0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

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TOTAL

* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: 2.441
Accelerating voltage 30.0 KeV
Beam - sample incidence angle 90.0 degrees
Xray emergence angle 35.0 degrees
Xray - window incidence angle 0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

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TOTAL 100.01

* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: 1.892
19-Jan-1993 15:08:36

Accelerating voltage 30.0 KeV
Beam - sample incidence angle 90.0 degrees
Xray emergence angle 35.0 degrees
Xray - window incidence angle 0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

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TOTAL 100.00

* DETERMINED BY STOICHIOMETRY ITERATIONS 20

**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: 1.890
Accelerating voltage
Beam - sample incidence angle
X-ray emergence angle
X-ray - window incidence angle

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

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<th>ELEMENT &amp; LINE</th>
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<th>FORMULA</th>
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TOTAL
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* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: 1.887
Accelerating voltage: 30.0 KeV
Beam - sample incidence angle: 90.0 degrees
X-ray emergence angle: 35.0 degrees
X-ray - window incidence angle: 0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

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<td>99.99</td>
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</table>

* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: 1.892
Accelerating voltage: 30.0 KeV
Beam-sample incidence angle: 90.0 degrees
X-ray emergence angle: 35.0 degrees
X-ray-window incidence angle: 0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

<table>
<thead>
<tr>
<th>ELEMENT &amp; LINE</th>
<th>K-RATIO**</th>
<th>WEIGHT PERCENT</th>
<th>PRECISION 2 SIGMA</th>
<th>FORMULA</th>
<th>OXIDE PERCENT</th>
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<tbody>
<tr>
<td>Al KA</td>
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</tr>
</tbody>
</table>

TOTAL

* DETERMINED BY STOICHIOMETRY
ITERATIONS 7

**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: 1.890
Accelerating voltage $\quad$ 30.0 KeV
Beam - sample incidence angle $\quad$ 90.0 degrees
Xray emergence angle $\quad$ 35.0 degrees
Xray - window incidence angle $\quad$ 0.0 degrees

**STANDARDLESS EDS ANALYSIS**
(ZAF CORRECTIONS VIA MAGIC V)

<table>
<thead>
<tr>
<th>ELEMENT</th>
<th>WEIGHT &amp; LINE</th>
<th>ATOMIC &amp; LINE</th>
<th>ATOMIC PRECISION</th>
<th>2 SIGMA</th>
<th>K-RATIO**</th>
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TOTAL $\quad$ 100.01

ITERATIONS $\quad$ 7

*NOTE: ATOMIC PERCENT is normalized to 100

**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: $\quad$ 1.000
13-Jan-1993 15:29:17  SCWD-12

Accelerating voltage  30.0 KeV
Beam - sample incidence angle  90.0 degrees
Xray emergence angle  35.0 degrees
Xray - window incidence angle  0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

<table>
<thead>
<tr>
<th>ELEMENT &amp; LINE</th>
<th>K-RATIO**</th>
<th>WEIGHT</th>
<th>PRECISION</th>
<th>FORMULA</th>
<th>OXIDE</th>
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</tbody>
</table>

TOTAL 100.00

* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: 1.459
Accelerating voltage 30.0 KeV
Beam - sample incidence angle 90.0 degrees
Xray emergence angle 35.0 degrees
Xray - window incidence angle 0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

<table>
<thead>
<tr>
<th>ELEMENT &amp; LINE</th>
<th>K-RATIO**</th>
<th>WEIGHT PERCENT</th>
<th>PRECISION 2 SIGMA</th>
<th>FORMULA</th>
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<tbody>
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<td>100.00</td>
</tr>
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* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: 1.458
Accelerating voltage: 30.0 KeV
Beam - sample incidence angle: 90.0 degrees
X-ray emergence angle: 35.0 degrees
X-ray - window incidence angle: 0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

<table>
<thead>
<tr>
<th>ELEMENT &amp; LINE</th>
<th>K-RATIO**</th>
<th>PERCENT</th>
<th>PRECISION 2 SIGMA</th>
<th>FORMULA</th>
<th>OXIDE</th>
<th>PERCENT</th>
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</tbody>
</table>

TOTAL: 100.01

* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: 1.890
19-Jan-1993 15:39:08

Accelerating voltage 30.0 KeV
Beam - sample incidence angle 90.0 degrees
Xray emergence angle 35.0 degrees
Xray - window incidence angle 0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

<table>
<thead>
<tr>
<th>ELEMENT &amp; LINE</th>
<th>K-RATIO**</th>
<th>WEIGHT</th>
<th>PRECISION 2 SIGMA</th>
<th>FORMULA</th>
<th>OXIDE PERCENT</th>
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</tbody>
</table>

TOTAL 100.00

* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: 1.891
Accelerating voltage 30.0 KeV
Beam-sample incidence angle 90.0 degrees
X-ray emergence angle 35.0 degrees
X-ray-window incidence angle 0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

<table>
<thead>
<tr>
<th>ELEMENT &amp; LINE</th>
<th>K-RATIO**</th>
<th>WEIGHT PERCENT</th>
<th>PRECISION 2 SIGMA</th>
<th>OXIDE FORMULA</th>
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<td>MoO3</td>
<td>26.66</td>
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</table>

TOTAL 100.01

* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: 1.890
Accelerating voltage: 30.0 KeV
Beam-sample incidence angle: 90.0 degrees
X-ray emergence angle: 35.0 degrees
X-ray-window incidence angle: 0.0 degrees

**STANDARDLESS EDS ANALYSIS**
(ZAF CORRECTIONS VIA MAGIC V)

<table>
<thead>
<tr>
<th>ELEMENT &amp; LINE</th>
<th>K-RATIO** PERCENT</th>
<th>WEIGHT PERCENT</th>
<th>PRECISION 2 SIGMA</th>
<th>FORMULA</th>
<th>OXIDE PERCENT</th>
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<td>4.10</td>
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<td>100.01</td>
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</tbody>
</table>

* DETERMINED BY STOICHIOMETRY

**ITERATIONS 20**

**NOTE: K-RATIO = K-RATIO \times R**
where \( R = \text{reference (standard)}/\text{reference (sample)} \)

**NORMALIZATION FACTOR:** 1.890
Accelerating voltage 30.0 KeV
Beam - sample incidence angle 90.0 degrees
Xray emergence angle 35.0 degrees
Xray - window incidence angle 0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

<table>
<thead>
<tr>
<th>ELEMENT &amp; LINE</th>
<th>K-RATIO**</th>
<th>PERCENT</th>
<th>PRECISION (2 SIGMA)</th>
<th>FORMULA</th>
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<tr>
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</tbody>
</table>

TOTAL 100.01

* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: 1.891
Accelerating voltage \hspace{1cm} 30.0 KeV \\
Beam - sample incidence angle \hspace{1cm} 90.0 degrees \\
Xray emergence angle \hspace{1cm} 35.0 degrees \\
Xray - window incidence angle \hspace{1cm} 0.0 degrees \\

STANDARDLESS EDS ANALYSIS \\
(ZAF CORRECTIONS VIA MAGIC V)

<table>
<thead>
<tr>
<th>ELEMENT &amp; LINE</th>
<th>K-RATIO**</th>
<th>PERCENT</th>
<th>PRECISION 2 SIGMA</th>
<th>FORMULA</th>
<th>OXIDE PERCENT</th>
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<tbody>
<tr>
<td>Cr KA</td>
<td>0.0107</td>
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<td>0.09</td>
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<td>Ni KA</td>
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<td>0.30</td>
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<td>0.3862</td>
<td>29.51</td>
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</table>

TOTAL \hspace{1cm} 100.00

* DETERMINED BY STOICHIOMETRY

ITERATIONS \hspace{1cm} 20

**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: \hspace{1cm} 1.464

A-19
Accelerating voltage: 30.0 KeV
Beam-sample incidence angle: 90.0 degrees
X-ray emergence angle: 35.0 degrees
X-ray-window incidence angle: 0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

<table>
<thead>
<tr>
<th>ELEMENT &amp; LINE</th>
<th>K-RATIO**</th>
<th>WEIGHT PERCENT</th>
<th>PRECISION 2 SIGMA</th>
<th>FORMULA</th>
<th>OXIDE PERCENT</th>
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<tbody>
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<td>0.17</td>
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<tr>
<td>Mo KA</td>
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<tr>
<td>O *</td>
<td></td>
<td>27.38</td>
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TOTAL: 100.01

*DETERMINED BY STOICHIOMETRY

ITERATIONS: 20

**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: 1.894
Accelerating voltage 30.0 KeV
Beam - sample incidence angle 90.0 degrees
Xray emergence angle 35.0 degrees
Xray - window incidence angle 0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

<table>
<thead>
<tr>
<th>ELEMENT &amp; LINE</th>
<th>K-RATIO**</th>
<th>WEIGHT PERCENT</th>
<th>PRECISION 2 SIGMA</th>
<th>FORMULA</th>
<th>OXIDE PERCENT</th>
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<tbody>
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<td>Cr KA</td>
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<td>Ni KA</td>
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<td>Mo KA</td>
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<td>O *</td>
<td>0.1395</td>
<td>16.41</td>
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<td>TOTAL</td>
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</tbody>
</table>

* DETERMINED BY STOICHIOMETRY
**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: 1.888
Accelerating voltage 30.0 KeV
Beam - sample incidence angle 90.0 degrees
Xray emergence angle 35.0 degrees
Xray - window incidence angle 0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

<table>
<thead>
<tr>
<th>ELEMENT &amp; LINE</th>
<th>K-RATIO**</th>
<th>WEIGHT</th>
<th>PRECISION</th>
<th>OXIDE</th>
<th>PERCENT</th>
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<tbody>
<tr>
<td>Ti KA</td>
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<td>TiO₂</td>
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<tr>
<td>Cr KA</td>
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<td>Nb₂O₅</td>
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<tr>
<td>Mo KA</td>
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<td>0.96</td>
<td>0.32</td>
<td>MoO₃</td>
<td>1.44</td>
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<tr>
<td>O *</td>
<td>31.55</td>
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</tbody>
</table>

TOTAL 100.00

* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

**NOTE: K-RATIO = K-RATIO x R
where R = reference (standard)/reference (sample)

NORMALIZATION FACTOR: 1.669
Accelerating voltage 30.0 KeV
Beam - sample incidence angle 90.0 degrees
Xray emergence angle 35.0 degrees
Xray - window incidence angle 0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

<table>
<thead>
<tr>
<th>ELEMENT &amp; LINE</th>
<th>K-RATIO**</th>
<th>WEIGHT PERCENT</th>
<th>PRECISION 2 SIGMA</th>
<th>FORMULA</th>
<th>OXIDE PERCENT</th>
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<tbody>
<tr>
<td>Al KA</td>
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<td>Al2O3</td>
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<td>Cr KA</td>
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<tr>
<td>O *</td>
<td>0.0184</td>
<td>30.35</td>
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</tr>
</tbody>
</table>

TOTAL 100.01

* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

**NOTE: K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

NORMALIZATION FACTOR: 1.893
Accelerating voltage  30.0 KeV
Beam - sample incidence angle  90.0 degrees
Xray emergence angle  35.0 degrees
Xray - window incidence angle  0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

| ELEMENT & LINE & K-RATIO** PERCENT & PRECISION & OXIDE FORMULA & OXIDE PERCENT |
|-----------------------|-----------------|-----------------|------------------|-----------------|
| Al KA                 | 0.0097          | 3.00            | 0.16             | Al2O3           | 5.67            |
| Ti KA                 | 0.0097          | 1.01            | 0.06             | TiO2            | 1.68            |
| Cr KA                 | 0.4124          | 44.51           | 0.33             | Cr2O3           | 65.06           |
| Fe KA                 | 0.0026          | 0.32            | 0.05             | FeO             | 0.41            |
| Ni KA                 | 0.0834          | 9.56            | 0.20             | NiO             | 12.16           |
| Nb KA                 | 0.0257          | 2.98            | 0.51             | Nb2O5           | 4.26            |
| Mo KA                 | 0.0604          | 7.17            | 0.91             | MoO3            | 10.76           |
| O *                   | 31.45           |                 |                  |                 | 100.00          |

* DETERMINED BY STOICHIOMETRY
ITERATIONS  20

**NOTE: K-RATIO = K-RATIO x R
where R = reference (standard) / reference (sample)

NORMALIZATION FACTOR:  1.892
Accelerating voltage 30.0 KeV
Beam - sample incidence angle 90.0 degrees
Xray emergence angle 35.0 degrees
Xray - window incidence angle 0.0 degrees

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

<table>
<thead>
<tr>
<th>ELEMENT &amp; LINE</th>
<th>WEIGHT PERCENT</th>
<th>PRECISION 2 SIGMA</th>
<th>FORMULA</th>
<th>OXIDE FORMULA PERCENT</th>
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<tbody>
<tr>
<td>Al KA</td>
<td>0.0027</td>
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<tr>
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ITERATIONS 20

**NOTE: K-RATIO = K-RATIO x R
where R = reference (standard)/reference (sample)

NORMALIZATION FACTOR: 1.893