**SUPERCRITICAL WATER OXIDATION
BENCHSCALE TESTING
METALLURGICAL ANALYSIS REPORT**

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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 Modell 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 can not 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.

Brad C Norby

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Attachment
/dj

<p>Table 1</p> <p>Supercritical Water Oxidation Thickness Measurements</p> <p>Inconel 625 Wire Exposed for 20 Hours</p>					
Wire Location in Reactor	Wire Sample Number	Sensitized Wire Thickness (inches)	Wire Thickness after Exposure (inches)	Estimated Metal Loss (0.001")	Estimated Corrosion Rate (mpy)
5'6"-6'0"	1	0.062	0.0612	0.02	7
	2	0.061	0.0616	0.00	0
	3	0.061	0.0616	0.00	0
	4	0.061	0.0616	0.00	0
6'6"-7'0"	1	0.061	0.062	0.00	0
	2	0.061	0.0612	0.02	7
	3	0.0612	0.0622	0.00	0
	4	0.0612	0.0616	0.00	0
7'6"-8'0"	1	0.0614	0.061	0.12	51
	2	0.0616	0.061	0.12	51
	3	0.0616	0.0608	0.22	95
	4	0.0614	0.061	0.12	51
9'0"-9'6"	1	0.0612	0.0612	0.02	7
	2	0.061	0.0612	0.02	7
	3	0.0612	0.0612	0.02	7
	4	0.0612	0.061	0.12	51
9'6"-10'0"	1	0.0612			
	2	0.0612			
	3	0.0612			
	4	0.0614			
12'0"-12'8"	1	0.0614	0.057	2.12	927
	2	0.0612	0.053	4.12	1803

Table 1

Supercritical Water Oxidation Thickness Measurements
Inconel 625 Wire Exposed for 20 Hours

Wire Location in Reactor	Wire Sample Number	Sensitized Wire Thickness (inches)	Wire Thickness after Exposure (inches)	Estimated Metal Loss (0.001")	Estimated Corrosion Rate (mpy)
	3	0.0614	0.052	4.62	2022
	4	0.061	0.058	1.62	708
16'0"-16'6"	1	0.0608	0.057	2.12	927
	2	0.0612	0.057	2.12	927
	3	0.061	0.056	2.62	1146
	4	0.061	0.056	2.62	1146
18'0"-18'6"	1	0.0612	0.0596	0.82	357
	2	0.0614	0.0592	1.02	445
	3	0.061	0.059	1.12	489
	4	0.0608	0.06	0.62	270
18'6"-19'0"	1	0.0608	0.058	1.62	708
	2	0.061	0.055	3.12	1365
	3	0.0612	0.056	2.62	1146
	4	0.061	0.053	4.12	1803
19'0"-19'6"	1	0.0612	0.054	3.62	1584
	2	0.0604	0.0546	3.32	1452
	3	0.0612	0.054	3.62	1584
	4	0.061	0.055	3.12	1365
19'6"-20'0"	1	0.0612	0.0554	2.92	1277
	2	0.0608	0.057	2.12	927
	3	0.0614	0.0512	5.02	2197
	4	0.0608	0.052	4.62	2022
25'0"-25'6"	1	0.0614	0.055	3.12	1365

Table 1

Supercritical Water Oxidation Thickness Measurements
Inconel 625 Wire Exposed for 20 Hours

Wire Location in Reactor	Wire Sample Number	Sensitized Wire Thickness (inches)	Wire Thickness after Exposure (inches)	Estimated Metal Loss (0.001")	Estimated Corrosion Rate (mpy)
	2	0.0616	0.053	4.12	1803
	3	0.0614	0.0534	3.92	1715
	4	0.0616	0.054	3.62	1584
25'6"-26'0"	1	0.0612	0.053	4.12	1803
	2	0.0614	0.059	1.12	489
	3	0.0618	0.06	0.62	270
	4	0.062	0.059	1.12	489
30'0"-30'6"	1	0.0608	0.06	0.62	270
	2	0.0608	0.06	0.62	270
	3	0.0618	0.0596	0.82	357
	4	0.061	0.0598	0.72	314
37'0"-37'6"	1	0.061	0.06	0.62	270
	2	0.0612	0.062	0.00	0
	3	0.0612	0.061	0.12	51
	4	0.061	0.0604	0.42	182
38'0"-38'6"	1		0.061	0.12	51
	2		0.061	0.12	51
	3		0.061	0.12	51
	4		0.061	0.12	51
47'6"-48'0"	1		0.061	0.12	51
	2		0.061	0.12	51
	3		0.061	0.12	51
	4		0.061	0.12	51

Table 1

Supercritical Water Oxidation Thickness Measurements
Inconel 625 Wire Exposed for 20 Hours

Wire Location in Reactor	Wire Sample Number	Sensitized Wire Thickness (inches)	Wire Thickness after Exposure (inches)	Estimated Metal Loss (0.001")	Estimated Corrosion Rate (mpy)
52'0"-52'6"	1		0.063	0	0
	2		0.061	0.12	51
	3		0.061	0.12	51
	4		0.0605	0.73	160
53'6"-54'0"	1	0.0614	0.0604	0.42	182
	2	0.0616	0.0602	0.52	226
	3	0.0614	0.061	0.12	51
	4	0.0614	0.0602	0.52	226
54'0"-54'6"	1	0.0616	0.049	6.12	2679
	2	0.0616	0.049	6.12	2679
	3	0.0616	0.051	5.12	2241
	4	0.0616	0.057	2.12	927
Average		0.06123			
Standard Deviation		0.00030			
Variance		9.25×10^{-8}			

Table 2

Supercritical Water Oxidation Thickness Measurements
Inconel 625 Wire Exposed for 40 Hours

Wire Location in Reactor	Wire Sample Number	Sensitized Wire Thickness (inches)	Wire Thickness after Exposure (inches)	Estimated Metal Loss (0.001")	Estimated Corrosion Rate (mpy)
5'6"-6'0"	1	0.062	0.061	0.12	25
	2	0.061	0.0612	0.02	4
	3	0.061	0.0614	0.00	0
	4	0.061	0.0612	0.02	4
6'6"-7'0"	1	0.061	0.061	0.12	25
	2	0.061	0.061	0.12	25
	3	0.0612	0.061	0.12	25
	4	0.0612	0.0608	0.22	47
7'6"-8'0"	1	0.0614	0.0614	0.00	0
	2	0.0616	0.0616	0.00	0
	3	0.0616	0.0612	0.02	4
	4	0.0614	0.061	0.12	25
9'0"-9'6"	1	0.0612	0.0612	0.02	4
	2	0.061	0.0612	0.02	4
	3	0.0612	0.0612	0.02	4
	4	0.0612	0.061	0.12	25
9'6"-10'0"	1	0.0612	0.06	0.62	135
	2	0.0612	0.0602	0.52	113
	3	0.0612	0.0604	0.42	91
	4	0.0614	0.0602	0.52	113
12'0"-12'8"	1	0.0614	0.05	5.62	1230
	2	0.0612	0.048	6.62	1449

Table 2

Supercritical Water Oxidation Thickness Measurements
Inconel 625 Wire Exposed for 40 Hours

Wire Location in Reactor	Wire Sample Number	Sensitized Wire Thickness (inches)	Wire Thickness after Exposure (inches)	Estimated Metal Loss (0.001")	Estimated Corrosion Rate (mpy)
	3	0.0614	0.054	3.62	792
	4	0.061	0.054	3.62	792
16'0"-16'6"	1	0.0608	0.0544	3.42	748
	2	0.0612	0.055	3.12	682
	3	0.061	0.053	4.12	901
	4	0.061	0.053	4.12	901
18'0"-18'6"	1	0.0612	0.054	3.62	792
	2	0.0614	0.052	4.62	1011
	3	0.061	0.046	7.62	1668
	4	0.0608	0.05	5.62	1230
18'6"-19'0"	1	0.0608	0.052	4.62	1011
	2	0.061	0.053	4.12	901
	3	0.0612	0.05	5.62	1230
	4	0.061	0.048	6.62	1449
19'0"-19'6"	1	0.0612	0.052	4.62	1011
	2	0.0604	0.045	8.12	1777
	3	0.0612	0.048	6.62	1449
	4	0.061	0.047	7.12	1558
19'6"-20'0"	1	0.0612	0.04	10.62	2325
	2	0.0608	0.05	5.62	1230
	3	0.0614	0.051	5.12	1120
	4	0.0608	0.051	5.12	1120
25'0"-25'6"	1	0.0614	0.054	3.62	792

Table 2

Supercritical Water Oxidation Thickness Measurements
Inconel 625 Wire Exposed for 40 Hours

Wire Location in Reactor	Wire Sample Number	Sensitized Wire Thickness (inches)	Wire Thickness after Exposure (inches)	Estimated Metal Loss (0.001")	Estimated Corrosion Rate (mpy)
	2	0.0616	0.054	3.62	792
	3	0.0614	0.058	1.62	354
	4	0.0616	0.051	5.12	1120
25'6"-26'0"	1	0.0612	0.06	0.62	135
	2	0.0614	0.044	8.62	1887
	3	0.0618	0.048	6.62	1449
	4	0.062	0.054	3.62	792
30'0"-30'6"	1	0.0608	0.0596	0.82	179
	2	0.0608	0.0594	0.92	201
	3	0.0618	0.06	0.62	135
	4	0.061	0.0594	0.92	201
37'0"-37'6"	1	0.061	0.6	0.00	0
	2	0.0612	0.6	0.00	0
	3	0.0612	0.0604	0.42	91
	4	0.061	0.06	0.62	135
38'0"-38'6"	1		0.0605	0.37	80
	2		0.06	0.62	135
	3		0.0602	0.52	113
	4		0.0602	0.52	113
47'6"-48'0"	1		0.0605	0.37	80
	2		0.0605	0.37	80
	3		0.0606	0.32	69
	4		0.0605	0.37	80

Table 2					
Supercritical Water Oxidation Thickness Measurements Inconel 625 Wire Exposed for 40 Hours					
Wire Location in Reactor	Wire Sample Number	Sensitized Wire Thickness (inches)	Wire Thickness after Exposure (inches)	Estimated Metal Loss (0.001")	Estimated Corrosion Rate (mpy)
52'0"-52'6"	1		0.061	0.12	25
	2		0.061	0.12	25
	3		0.060	0.62	135
	4		0.0605	0.37	80
53'6"-54'0"	1	0.0614	0.0602	0.52	113
	2	0.0616	0.06	0.62	135
	3	0.0614	0.057	2.12	463
	4	0.0614	0.058	1.62	354
	5		0.039	11.12	2427
54'0"-54'6"	1	0.0616	0.061	0.12	25
	2	0.0616	0.061	0.12	25
	3	0.0616	0.061	0.12	25
	4	0.0616	0.061	0.12	25
	5		0.055	3.12	681
Average		0.06123			
Standard Deviation		0.00030			
Variance		9.25×10^{-8}			

SCWO Reactor Corrosion

Inconel 625 40 hour test

Corrosion rate (mpy)

Reactor Location (feet)
Figure 1

SCWO Reactor Corrosion

Inconel 625 20 hour test

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)

Edges of sensitized wires located between 19.5 to 20 ft. showing the thin oxide layer and possible precipitates (400X as polished)

Figure 14

Microstructure of sensitized wire between 9 and 9.5 ft. (100X etched with 10% HCl, 10% $C_2H_3O_2H$, 10% HNO_3)

Figure 13

Figure 15

Sensitized wires taken from between 9 and 9.5 ft. showing the difference in grain sizes (100X etched with 10% HCl, 10% $C_2H_3O_2H$, 10% HNO_3)

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

Figure 30

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

Figure 31

Effect of sensitizing heat treatment on corrosion of Inconel 625 in boiling 65% HNO_3

19-Jan-1993 14:41:27

SCWD-1

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	WEIGHT PERCENT	ATOMIC PERCENT*	PRECISION 2 SIGMA	K-RATIO**
Al KA	0.81	1.80	0.15	0.0019
Ti KA	0.09	0.11	0.04	0.0008
Cr KA	22.22	25.59	0.27	0.2160
Fe KA	0.82	0.88	0.08	0.0082
Ni KA	60.74	61.96	0.52	0.5745
Nb KA	4.37	2.82	0.78	0.0396
Mo KA	10.94	6.83	1.31	0.0976
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

19-Jan-1993 14:47:11

SCWO-2

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Al KA	0.0032	0.93	0.12	Al2O3	1.75
Ti KA	0.0024	0.27	0.05	TiO2	0.45
Cr KA	0.3337	37.17	0.30	Cr2O3	54.33
Fe KA	0.0022	0.27	0.06	FeO	0.35
Ni KA	0.0735	8.30	0.18	NiO	10.56
Nb KA	0.0770	8.76	0.82	Nb2O5	12.52
Mo KA	0.1141	13.35	1.15	MoO3	20.03
O *		30.95			
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

19-Jan-1993 14:50:14

SCWD-3

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Al KA	0.0101	3.96	0.32	Al2O3	7.49
Ti KA	0.0007	0.07	0.03	TiO2	0.12
Cr KA	0.1147	11.80	0.22	Cr2O3	17.25
Fe KA	0.0028	0.27	0.07	FeO	0.35
Ni KA	0.4577	48.48	0.52	NiO	61.69
Nb KA	0.0320	3.74	0.75	Nb2O5	5.35
Mo KA	0.0434	5.17	1.04	MoO3	7.75
O *		26.50			
TOTAL					99.99

* DETERMINED BY STOICHIOMETRY
ITERATIONS 7

**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)

ELEMENT & LINE	K-RATIO**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Al KA	0.0022	0.66	0.11	Al2O3	1.25
Ti KA	0.0025	0.27	0.05	TiO2	0.45
Cr KA	0.4167	45.63	0.34	Cr2O3	66.69
Fe KA	0.0141	1.77	0.10	FeO	2.28
Ni KA	0.0302	3.48	0.14	NiO	4.43
Nb KA	0.0688	7.89	0.87	Nb2O5	11.29
Mo KA	0.0773	9.08	1.10	MoO3	13.62
O *		31.22			
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

19-Jan-1993 14:56:20

SCW0-5

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
S KA	0.1383	20.90	0.24	SO3	52.19
Cr KA	0.0398	4.18	0.12	Cr2O3	6.12
Fe KA	0.0004	0.04	0.02	FeO	0.05
Ni KA	0.3118	32.72	0.36	NiO	41.64
O *		42.15			
TOTAL					99.99

* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

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

NORMALIZATION FACTOR: 2.441

19-Jan-1993 15:00:03

SCWO-6

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Al KA	0.0018	0.55	0.09	Al2O3	1.03
Ti KA	0.0020	0.21	0.05	TiO2	0.35
Cr KA	0.4341	47.16	0.35	Cr2O3	68.92
Fe KA	0.0154	1.94	0.10	FeO	2.49
Ni KA	0.0371	4.29	0.15	NiO	5.46
Nb KA	0.0407	4.67	0.68	Nb2O5	6.68
Mo KA	0.0834	9.82	1.08	MoO3	14.74
Ce LA	0.0027	0.28	0.08	Ce2O3	0.33
O *		31.09			

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

SCWO-7

STANDARDLESS EDS ANALYSIS
(ZAF CORRECTIONS VIA MAGIC V)

TOTAL	100.00
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****NOTE:** K-RATIO = K-RATIO x R
where R = reference(standard)/reference(sample)

A-7

19-Jan-1993 15:15:07

SCWD-8

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Al KA	0.0018	0.65	0.13	Al2O3	1.22
Cl KA	0.0051	1.03	0.13	Cl	1.03
Cr KA	0.0761	8.63	0.24	Cr2O3	12.61
Ni KA	0.0182	2.06	0.15	NiO	2.62
Nb KA	0.0289	3.00	0.69	Nb2O5	4.29
Mo KA	0.0698	7.31	1.33	MoO3	10.97
Ce LA	0.3674	42.13	0.50	Ce2O3	49.34
Pb LA	0.1408	16.64	0.68	PbO	17.92
O *		18.57			

TOTAL

100.02

* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

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

NORMALIZATION FACTOR: 1.887

19-Jan-1993 15:18:50

SCWD-9

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Al KA	0.0056	1.66	0.13	Al2O3	3.14
Cl KA	0.0032	0.56	0.06	Cl	0.56
Ti KA	0.0038	0.43	0.05	TiO2	0.72
Cr KA	0.2976	33.11	0.27	Cr2O3	48.39
Fe KA	0.0065	0.78	0.07	FeO	1.00
Ni KA	0.1015	11.38	0.20	NiO	14.48
Nb KA	0.1123	12.86	0.91	Nb2O5	18.40
Mo KA	0.0756	8.86	0.98	MoO3	13.30
O *		30.35			
TOTAL					99.99

* 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 15:22:31

SCWO-10

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Al KA	0.0042	1.69	0.17	Al2O3	3.20
Cl KA	0.1248	19.73	0.26	Cl	19.73
Cr KA	0.0161	1.67	0.09	Cr2O3	2.44
Ni KA	0.5601	58.38	0.50	NiO	74.29
Pb LA	0.0024	0.33	0.12	PbO	0.35
O *		18.21			
TOTAL					100.01

* DETERMINED BY STOICHIOMETRY
ITERATIONS 7

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

NORMALIZATION FACTOR: 1.890

19-Jan-1993 15:27:02

SCWD-11

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	WEIGHT PERCENT	ATOMIC PERCENT*	PRECISION 2 SIGMA	K-RATIO**
Al KA	7.32	14.77	0.27	0.0153
Cl KA	0.74	1.13	0.11	0.0039
Cr KA	3.73	3.91	0.10	0.0386
Fe KA	0.32	0.31	0.05	0.0039
Ni KA	83.48	77.38	0.49	0.8217
Mo KA	4.42	2.50	1.00	0.0385
TOTAL	100.01			

ITERATIONS 7

*NOTE: ATOMIC PERCENT is normalized to 100

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

NORMALIZATION FACTOR: 1.000

19-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)

ELEMENT & LINE	K-RATIO**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Cr KA	0.0110	1.17	0.21	Cr2O3	1.71
Fe KA	0.0100	1.29	0.18	FeO	1.66
Ni KA	0.0057	0.67	0.14	NiO	0.85
Ce LA	0.6654	71.42	0.76	Ce2O3	83.65
Pb LA	0.0965	11.26	0.77	PbO	12.13
O *		14.19			
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

19-Jan-1993 15:32:46

SCWD-13

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Cr KA	0.0254	2.73	0.22	Cr2O3	3.99
Fe KA	0.0021	0.27	0.10	FeO	0.35
Ni KA	0.0090	1.05	0.15	NiO	1.33
Ce LA	0.6440	69.38	0.68	Ce2O3	81.26
Pb LA	0.1038	12.13	0.69	PbO	13.07
O *		14.44			
TOTAL					100.00

* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

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

NORMALIZATION FACTOR: 1.458

19-Jan-1993 15:36:34

SCWO-14

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Al KA	0.0154	5.15	0.21	Al2O3	9.74
Ti KA	0.0063	0.65	0.06	TiO2	1.08
Cr KA	0.3640	38.50	0.30	Cr2O3	56.27
Fe KA	0.0030	0.36	0.03	FeO	0.46
Ni KA	0.1606	18.18	0.26	NiO	23.14
Nb KA	0.0135	1.58	0.43	Nb2O5	2.26
Mo KA	0.0259	3.09	0.79	MoO3	4.64
Ce LA	0.0152	1.56	0.12	Ce2O3	1.83
Pb LA	0.0043	0.56	0.20	PbO	0.60
O *		30.38			
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

SCW0-15

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Al KA	0.0029	0.78	0.13	Al2O3	1.48
Ti KA	0.0020	0.24	0.05	TiO2	0.40
Cr KA	0.3162	35.94	0.35	Cr2O3	52.53
Fe KA	0.0062	0.77	0.09	FeO	0.99
Ni KA	0.0217	2.45	0.14	NiO	3.11
Nb KA	0.0709	7.94	0.94	Nb2O5	11.36
Mo KA	0.1726	20.08	1.58	MoO3	30.13
O *		31.80			
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

19-Jan-1993 15:41:42

SCWO-16

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Al KA	0.0053	1.68	0.16	Al2O3	3.17
Cl KA	0.0173	3.30	0.13	Cl	3.30
Ti KA	0.0027	0.34	0.06	TiO2	0.56
Cr KA	0.0870	9.84	0.18	Cr2O3	14.38
Fe KA	0.0088	0.94	0.09	FeO	1.20
Ni KA	0.2583	27.61	0.34	NiO	35.14
Nb KA	0.0964	10.90	1.01	Nb2O5	15.59
Mo KA	0.1526	17.77	1.46	MoO3	26.66
O *		27.63			

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 15:47:08

SCWD-17

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Al KA	0.0029	0.90	0.12	Al2O3	1.70
Cl KA	0.0210	4.10	0.15	Cl	4.10
Ti KA	0.0024	0.31	0.06	TiO2	0.51
Cr KA	0.0569	6.53	0.16	Cr2O3	9.54
Fe KA	0.0012	0.12	0.04	FeO	0.16
Ni KA	0.2626	27.84	0.35	NiO	35.43
Zn KA	0.0022	0.26	0.08	ZnO	0.32
Nb KA	0.0866	9.70	0.96	Nb2O5	13.87
Mo KA	0.1872	21.66	1.53	MoO3	32.50
Ce LA	0.0046	0.54	0.11	Ce2O3	0.63
Pb LA	0.0092	1.15	0.25	PbO	1.24
O *		26.90			
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 16:07:21

SCWD-18

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Al KA	0.0068	1.98	0.21	Al2O3	3.74
Cl KA	0.0020	0.41	0.09	Cl	0.41
Ti KA	0.0088	1.11	0.11	TiO2	1.85
Cr KA	0.1367	15.76	0.30	Cr2O3	23.03
Fe KA	0.0012	0.14	0.05	FeO	0.18
Ni KA	0.1585	17.13	0.37	NiO	21.80
Zn KA	0.0033	0.37	0.10	ZnO	0.46
Nb KA	0.2269	25.78	1.72	Nb2O5	36.88
Mo KA	0.0489	5.66	1.06	MoO3	8.49
Ce LA	0.0075	0.87	0.16	Ce2O3	1.02
Pb LA	0.0161	2.00	0.36	PbO	2.15
O *		28.80			
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

19-Jan-1993 16:09:56

SCWO-19

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Cr KA	0.0107	1.27	0.09	Cr2O3	1.85
Ni KA	0.2380	25.03	0.30	NiO	31.85
Mo KA	0.3862	44.19	1.90	MoO3	66.30
O *		29.51			
TOTAL					100.00

* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

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

NORMALIZATION FACTOR: 1.464

19-Jan-1993 16:13:03

SCWD-20

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Al KA	0.0049	1.82	0.17	Al2O3	3.44
Ti KA	0.0025	0.28	0.05	TiO2	0.47
Cr KA	0.2133	22.67	0.24	Cr2O3	33.13
Fe KA	0.0109	1.18	0.09	FeO	1.52
Ni KA	0.2597	27.85	0.32	NiO	35.44
Zn KA	0.0596	7.07	0.22	ZnO	8.79
Nb KA	0.0536	6.23	0.78	Nb2O5	8.91
Mo KA	0.0468	5.53	0.92	MoO3	8.30
O *		27.38			

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

19-Jan-1993 16:16:09

SCWD-21

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Al KA	0.0118	4.19	0.20	Al2O3	7.91
Ti KA	0.0011	0.14	0.04	TiO2	0.23
Cr KA	0.0521	5.65	0.13	Cr2O3	8.26
Ni KA	0.3844	40.30	0.38	NiO	51.28
Zn KA	0.0055	0.65	0.09	ZnO	0.81
Nb KA	0.0422	4.82	0.69	Nb2O5	6.90
Mo KA	0.1395	16.41	1.30	MoO3	24.61
O *		27.85			
TOTAL					100.01

* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

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

NORMALIZATION FACTOR: 1.888

19-Jan-1993 16:20:05

SCWD-22

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Ti KA	0.0062	0.56	0.05	TiO2	0.93
Cr KA	0.6148	64.43	0.38	Cr2O3	94.17
Ni KA	0.0062	0.73	0.08	NiO	0.93
Nb KA	0.0151	1.77	0.44	Nb2O5	2.53
Mo KA	0.0081	0.96	0.32	MoO3	1.44
O *		31.55			
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

19-Jan-1993 16:22:51

SCWD-23

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Al KA	0.0078	2.56	0.17	Al2O3	4.84
Cl KA	0.0033	0.49	0.06	Cl	0.49
Ti KA	0.0066	0.66	0.06	TiO2	1.10
Cr KA	0.4378	46.11	0.34	Cr2O3	67.40
Fe KA	0.0200	2.45	0.11	FeO	3.15
Ni KA	0.1094	12.61	0.24	NiO	16.05
Nb KA	0.0221	2.59	0.58	Nb2O5	3.70
Mo KA	0.0184	2.19	0.62	MoO3	3.29
O *		30.35			

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

19-Jan-1993 16:25:18

SCWD-24

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	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			
TOTAL					100.00

* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

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

NORMALIZATION FACTOR: 1.892

19-Jan-1993 16:27:37

SCWD-25

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**	WEIGHT PERCENT	PRECISION 2 SIGMA	FORMULA	OXIDE PERCENT
Al KA	0.0027	0.89	0.11	Al2O3	1.69
Ti KA	0.0037	0.35	0.05	TiO2	0.59
Cr KA	0.5105	53.30	0.37	Cr2O3	77.89
Fe KA	0.0021	0.26	0.06	FeO	0.34
Ni KA	0.1043	12.10	0.24	NiO	15.40
Nb KA	0.0150	1.76	0.43	Nb2O5	2.51
Mo KA	0.0088	1.05	0.34	MoO3	1.58
O *		30.28			
TOTAL					99.99

* DETERMINED BY STOICHIOMETRY
ITERATIONS 20

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

NORMALIZATION FACTOR: 1.893