Sta-4 (3)

FEB 24 1997 **ENGINEERING DATA TRANSMITTAL**

1. EDT 619139

2. To: (Receiving Organization) Distribution	3. From: (Originating Organization) Characterization Equipment Engineering	4. Related EDT No.: N/A
5. Proj./Prog./Dept./Div.: 8C420/N4H3A	6. Design Authority/ Design Agent/Cog. Engr.: A.B. Cockrell	7. Purchase Order No.: A05056-Y8
8. Originator Remarks: ETN-96-003 This test report will support inspection of quadralatch fingers for RMCS.		9. Equip./Component No.: N/A
		10. System/Bldg./Facility: 200 General
11. Receiver Remarks: 11A. Design Baseline Document? [X] Yes [] No		12. Major Assm. Dwg. No.: H-2-690139
		13. Permit/Permit Application No.: N/A
		14. Required Response Date: N/A

15. DATA TRANSMITTED

(A) Item No.	(B) Document/Drawing No.	(C) Sheet No.	(D) Rev. No.	(E) Title or Description of Data Transmitted	(F) Approval Designator	(G) Reason for Transmittal	(H) Originator Disposition	(I) Receiver Disposition
1	HNF-SD-WM-TRP-277	N/A	0	MATERIAL VERIFICATION OF QUADRALATCH FINGERS BY X-RAY FLUORESCENCE	N/A	1	1	1

16. KEY

Approval Designator (F)	Reason for Transmittal (G)	Disposition (H) & (I)
E, S, Q, D or N/A (see WHC-CM-3-5, Sec.12.7)	1. Approval 2. Release 3. Information 4. Review 5. Post-Review 6. Dist. (Receipt Acknow. Required)	1. Approved 2. Approved w/comment 3. Disapproved w/comment 4. Reviewed no/comment 5. Reviewed w/comment 6. Receipt acknowledged

17. SIGNATURE/DISTRIBUTION
(See Approval Designator for required signatures)

(G) Reason	(H) Disp.	(J) Name	(K) Signature	(L) Date	(M) MSIN	(G) Reason	(H) Disp.	(J) Name	(K) Signature	(L) Date	(M) MSIN
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MATERIAL VERIFICATION OF QUADRALATCH FINGERS BY X-RAY FLUORESCENCE

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U.S. Department of Energy Contract DE-AC06-96RL13200

EDT/ECN: 619139 UC: 2070
Org Code: 8C420 Charge Code: N4H3A
B&R Code: EW3120074 Total Pages: 21

Key Words: X-ray, fluorescence, quadralatch, fingers, stainless

Abstract: A sample of cast quadralatch fingers were measured with an X-ray fluorescence spectrometer. The results are compared with measurements of standard 300-series and 400-series stainless steel billets. Use of this measuring technique for material verification of fingers is described.

DATE: STA. 4 ID: 3
FEB 24 1997

Release Approval 2/24/97 Date
Release Stamp

Approved for Public Release

MATERIAL VERIFICATION OF QUADRALATCH FINGERS BY X-RAY FLUORESCENCE

PR Deichelbohrer
Numatec Hanford Corporation

February, 1997

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MATERIAL VERIFICATION OF QUADRALATCH
FINGERS BY X-RAY FLUORESCENCE

1.0 STAINLESS STEEL QUADRALATCH FINGERS

The quadralatch is a four-fingered device mounted on top of the universal sampler which is used in taking samples from waste tanks to characterize the contents.

Concerns about the potential for flammable gas being present in waste tanks has led to the requirement that the quadralatch fingers (as well as a number of other sampler components) be made of 300-series stainless steel. Testing has shown that this material is significantly less likely to produce mechanical sparks that ignite flammable gasses (Ref. 1).

The geometric design of the fingers was improved when the material was changed from the old carbon steel to 304 stainless (Ref. 2). Subsequently, fingers have been fabricated by investment casting and, also, by machining from a bar or tubing.

Safety Equipment List, WHC-SD-WM-SEL-032, Rev 1 requires that the finger material be classified Safety Class reflecting its importance in spark generation prevention. Consequently, a means is needed to determine finger material composition especially for the investment cast fingers which are sometimes procured from non-evaluated suppliers.

2.0 X-RAY FLUORESCENCE TESTING

X-ray fluorescence is a convenient means of measuring metal make-up of alloys. The method utilizes a probe which contains a radioactive source and an energy-sensitive X-ray detector. Gamma rays from the source excite atoms in the alloy under investigation (ALI), which in turn produce X-rays which are characteristic of their elemental identity. The intensity of each X-ray energy is displayed on a graph.

The sensitivity of the instrument is different for each element. Sensitivity is greatest for the heavy metals (Fe, Ni, Cr etc.). It is not very sensitive to carbon, for example. Consequently, X-ray fluorescence is commonly used with stainless steels, but not with carbon steels.

X-rays energies in the KEV range are produced, so the probe interrogates material to a depth of several tens of microns.

3.0 STAINLESS STEELS

The main constituent of stainless steel is iron and chromium. Selected percentages of other elements (nickel, carbon, manganese, silicon, etc.) determine the alloy's specific properties (Ref. 3). As illustrated in the Table below, the major difference between 300-series and 400-series stainless steel is the concentration of nickel. 400-series stainless has very little nickel.

Percentages of Elements in Selected Stainless Steels								
Alloy	Fe	C	Mn	Si	Cr	Ni	P	S
304	66-71	.08	2	1	18-20	8-10.5	.045	.03
416	83-85	.15	1.25	1	12-14	0	.04	.15
410	84-86	.15	1	1	11.5-13.5	.75	.04	.03

4.0 X-RAY FLUORESCENCE OF STAINLESS STEEL STANDARDS

4.1 STANDARD SPECTRA

Three billets of known stainless steel type were run on the X-ray fluorescence spectrometer.

Figure 1 is the X-ray fluorescence spectrum of **304 Stainless Steel**. The nickel peak is clearly present.

Figure 2 is an expansion of Figure 1 (**304 SS**) in the Cr-Ni region. Heights of the peaks are noted (in counts) to provide analytical measurement.

Figure 3 is the spectrum of **416 Stainless Steel**. The nickel peak is clearly absent.

Figure 4 is an expansion of Figure 3 (**416 SS**) in the Cr-Ni region. (A very small nickel peak is visible at this magnification.) Heights of the peaks are noted, in counts, to provide analytical measurement.

Figure 5 is the spectrum of **410 Stainless Steel**. The nickel peak is clearly absent.

Figure 6 is an expansion of Figure 5 (**410 SS**) in the Cr-Ni region. The nickel peak is absent in this spectrum. Heights of the peaks are noted, in counts, to provide analytical measurement.

4.2 PEAK HEIGHT AND PERCENTAGE

The height of a particular peak is proportional to:

- the concentration of the element in the AUI
- sensitivity of the spectrometer to that element
- counting time
- billet size, etc.

As a result, a 2% concentration of one element will produce a different peak height than for a different element. In the following Table the observed peak heights are compared with the concentrations expected in three types of stainless.

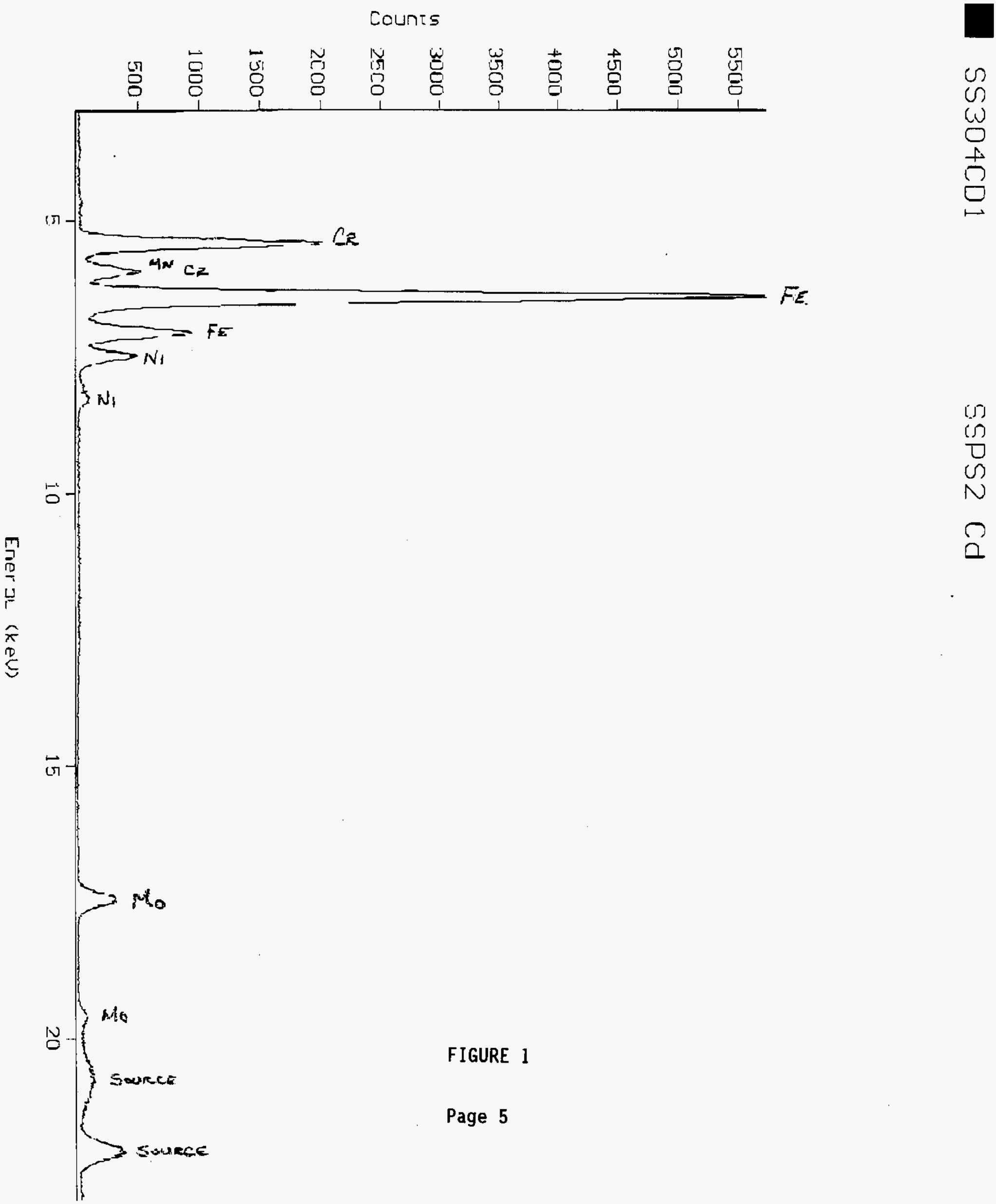

Counts
500
1000
1500
2000
2500
3000
3500
4000
4500
5000
5500
5
10
15
20
Energy (keV)
Cr
Mn Cr
Fe
Fe
Ni
Ni
Mo
Mo
Source
Source
SS304CD1
SSPS2 Cd

FIGURE 1

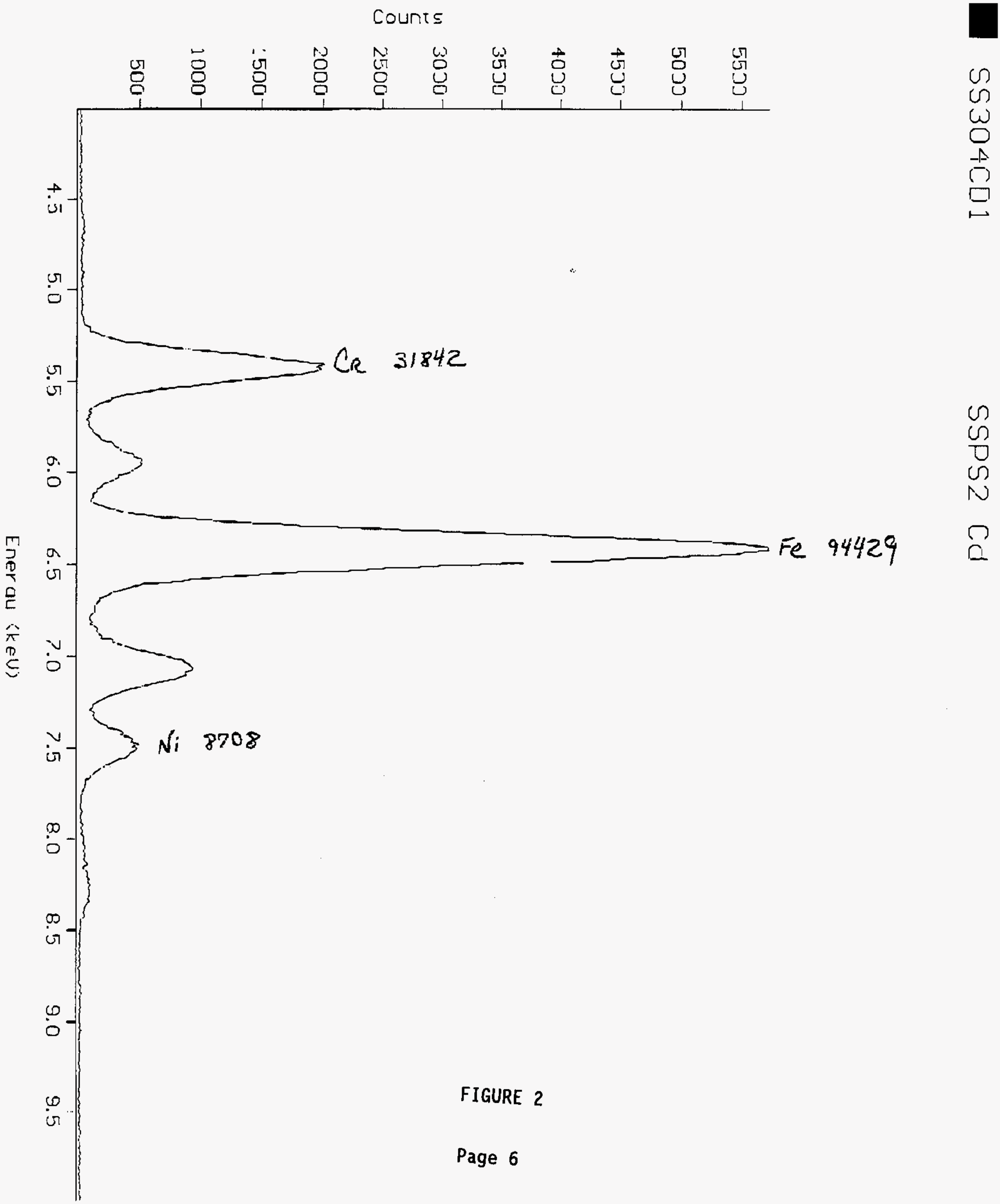

Counts
500
1000
1500
2000
2500
3000
3500
4000
4500
5000
5500
Energy (keV)
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
9.5
Cr 31842
Fe 94429
Ni 8708
SS304CD1
SSPS2 Cd

FIGURE 2

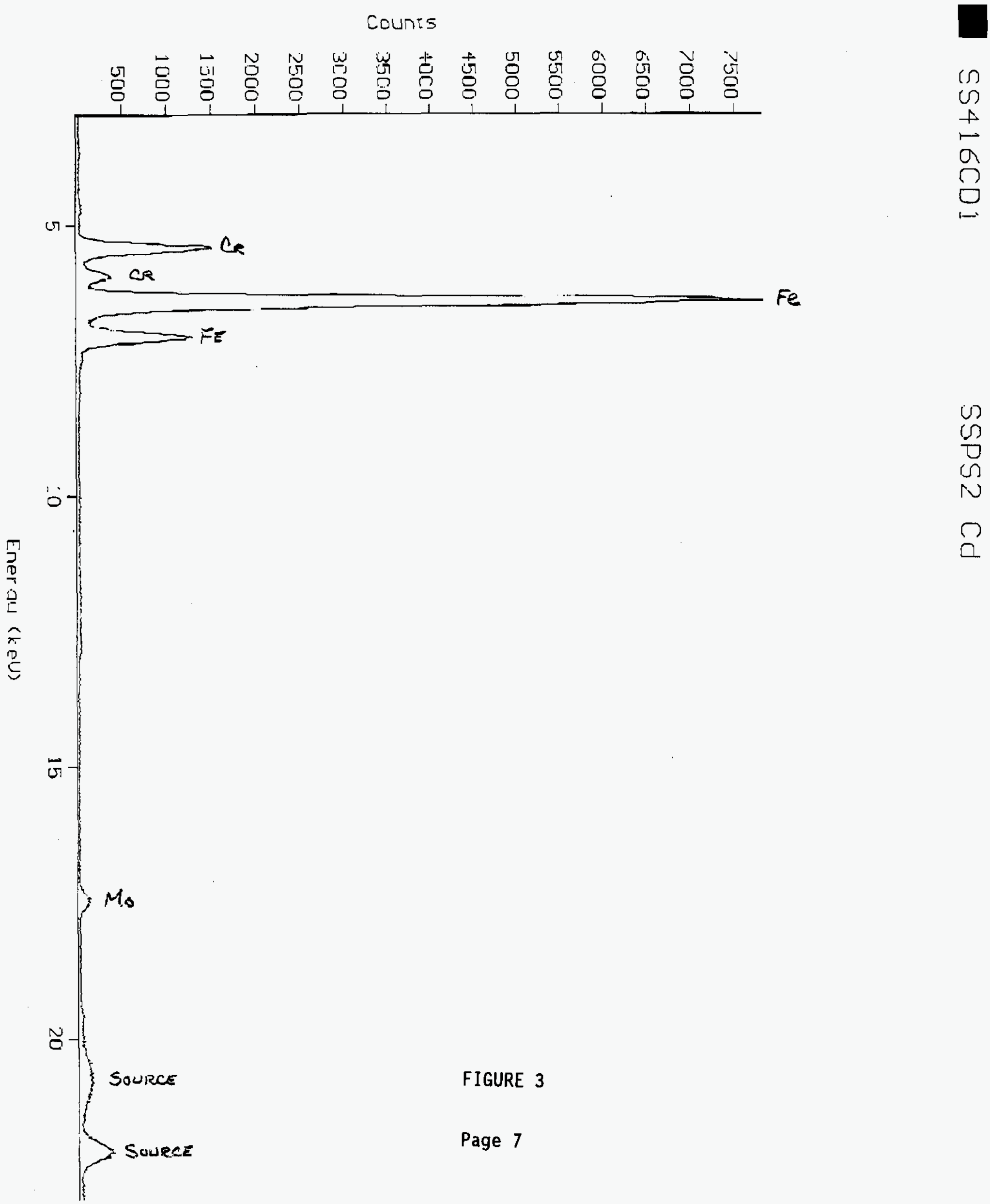

Counts
500
1000
1500
2000
2500
3000
3500
4000
4500
5000
5500
6000
6500
7000
7500
Energy (keV)
5
10
15
20
Cr
CR
Fe
FE
Mo
Source
Source
SS416CD1
SSPS2 Cd

FIGURE 3

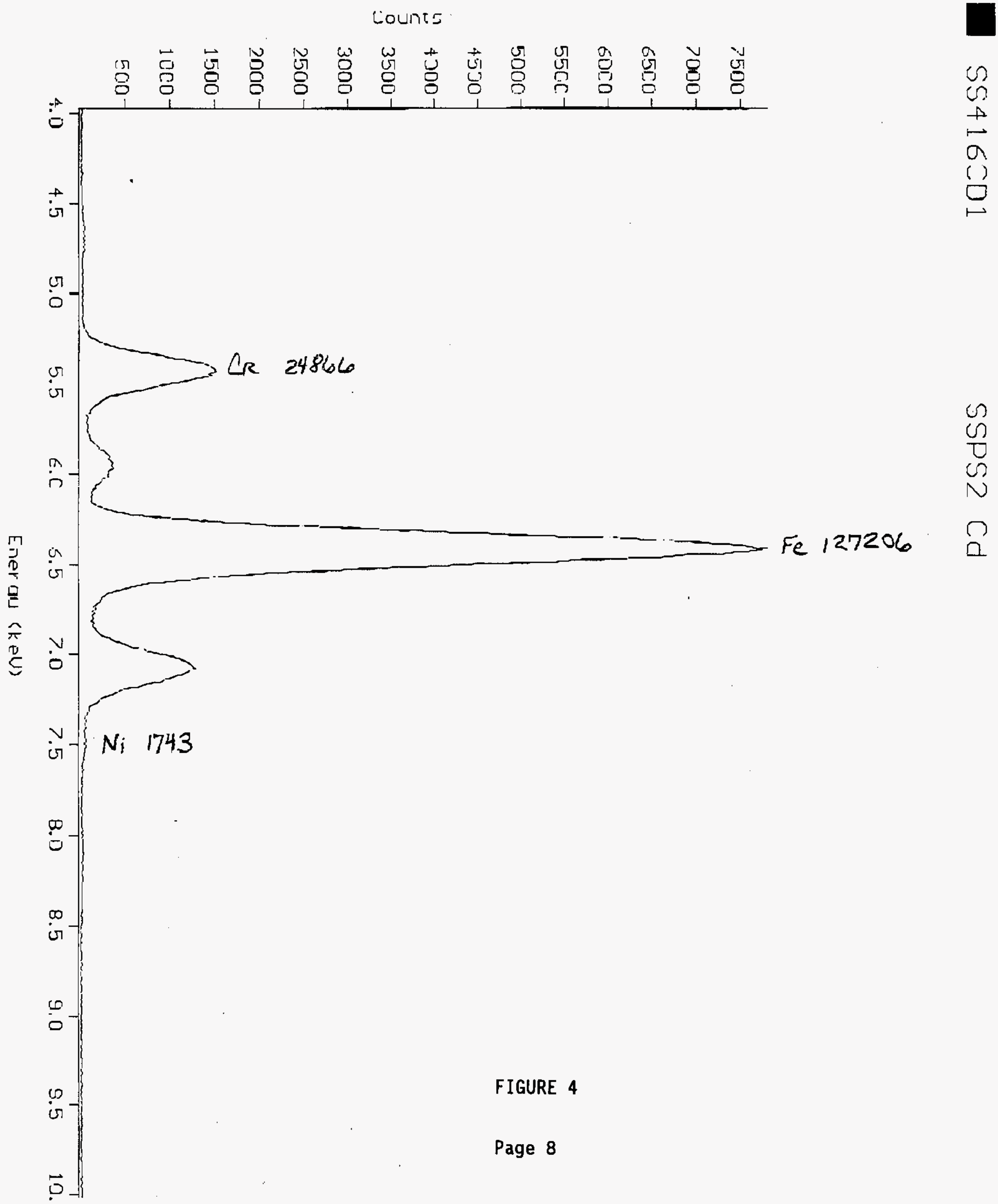
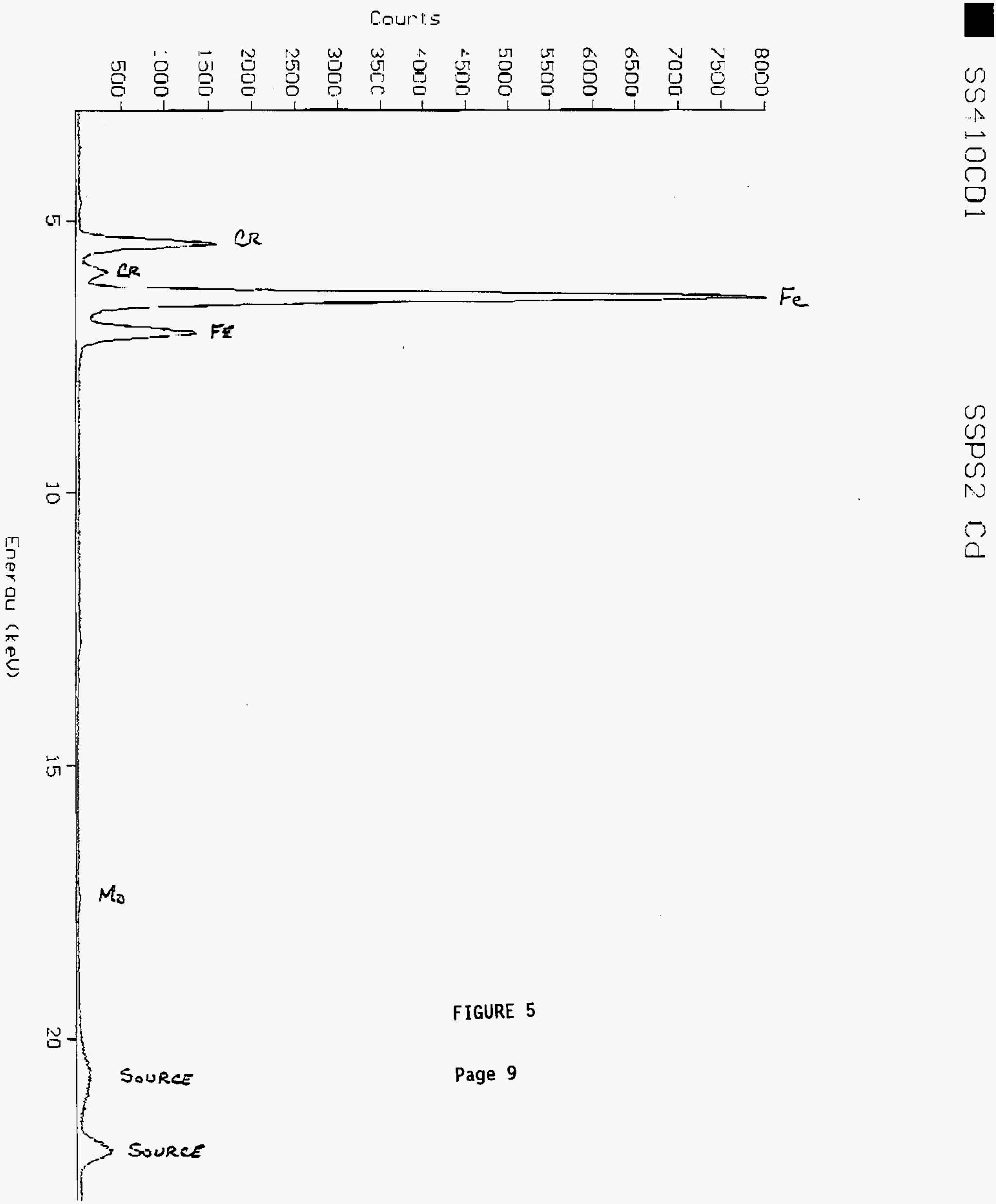

Counts
500
1000
1500
2000
2500
3000
3500
4000
4500
5000
5500
6000
6500
7000
7500
Energy (keV)
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
9.5
10.0
Cr 24866
Fe 127206
Ni 1743
SS416CD1
SSPS2 Cd

FIGURE 4


Counts
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1000
1500
2000
2500
3000
3500
4000
4500
5000
5500
6000
6500
7000
7500
8000
5
10
15
20
Energy (keV)
CR
CR
Fe
FE
Mo
SOURCE
SOURCE
SS410CD1
SSPS2 Cd

FIGURE 5

HNF-SD-WM-TRP-277, Rev. 0

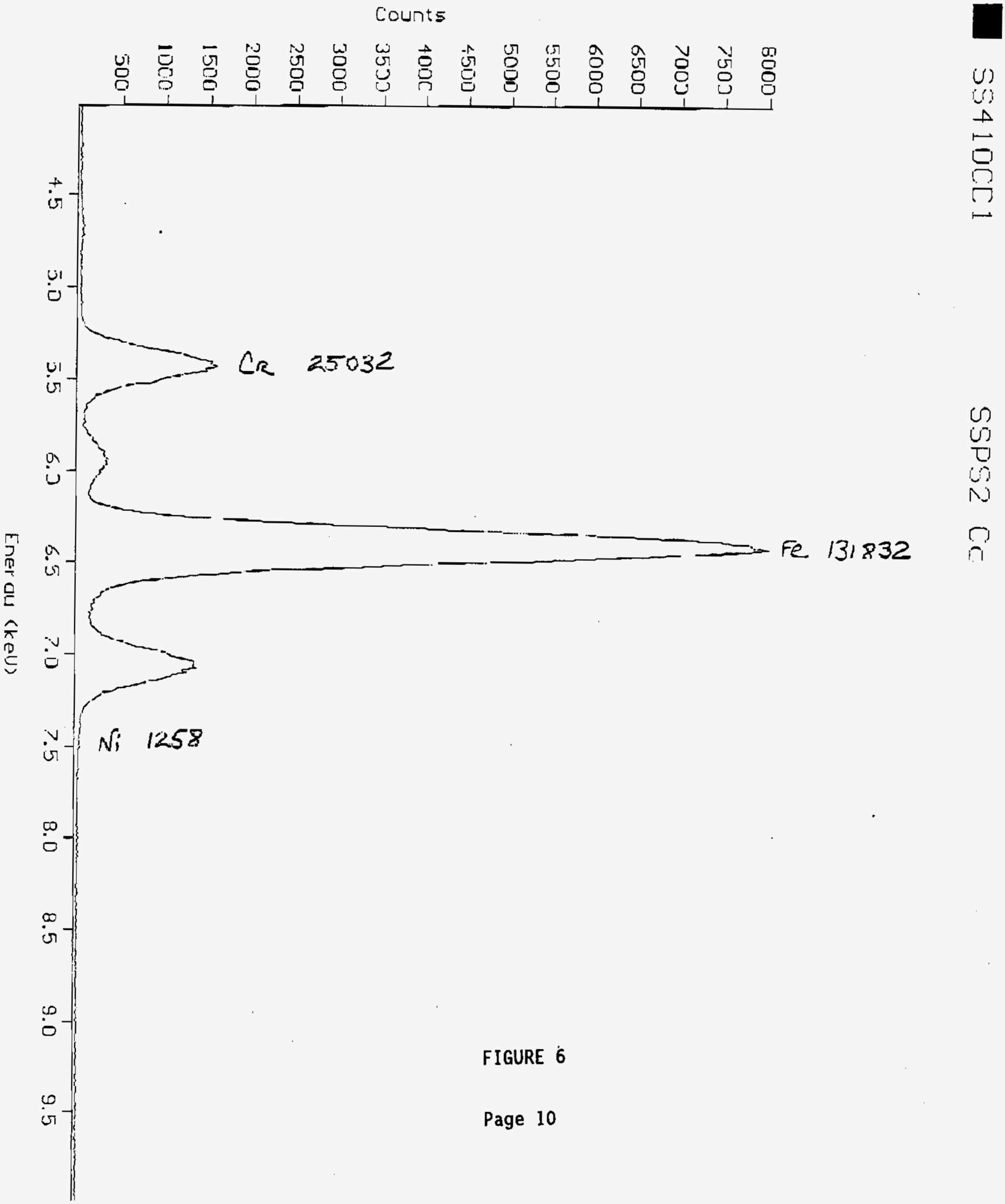


FIGURE 6

Peak Height and Percentages						
Alloy	Fe-%	Peak	Cr-%	Peak	Ni-%	Peak
304	66-71	94429	18-20	31842	8-12	8708
416	83-85	127206	12-14	24866	0	1743
410	84-86	131832	11.5-13.5	25032	.75	1258

As can be extracted from the table above, the sensitivities are approximately:
Fe: 1500 counts/%
Cr: 1900 counts/%
Ni: 1100 counts/%

5.0 X-RAY FLUORESCENCE TESTING OF FINGERS

Five cast stainless steel quadralatch fingers manufactured under contract PO A05056-Y8 were tested on the X-ray fluorescence spectrometer at Special Analytical Support, SGN Eurisys Services Corporation.

Figure 7 shows the spectra of all five of the fingers overlaid on each other. It should be noted that very little difference between the samples can be seen. Readings were taken at the center-inside of each finger.

Figure 8 is an expansion of the Cr-Ni region of Figure 7. Variations in peak height among the samples is shown in the Table below:

Variation in Finger Material Make Up (from Figure 8)			
	Avg. Count	1-sigma Variation	Percent Variation
Chromium (Cr)	17694	408	2.3%
Iron (Fe)	55811	1620	2.9%
Nickel (Ni)	6340	862	13.6%

Figure 9 was run to demonstrate repeatability of the X-ray fluorescence spectrometer. The same sample (sample 4) was run three times and the traces are overlaid on each other. No difference between the runs is discernable.

Figure 10 is six runs taken at different location of sample 3. A diagram in the corner of this figure shows the locations. It should be observed that the six runs are essentially equal indicating that the alloy constituents did not separate from each other at different locations of the casting. Some preliminary work with an eddy current probe had suggested that the casting might be inhomogeneous, but Figures 10 and 11 demonstrate good homogeneity.

Figure 11 is an expansion of Figure 10 in the Cr-Ni region.

Figures 7 through 11 illustrate that consistent results can be obtained from quadralatch fingers with X-ray fluorescence. Also, variations in makeup between different locations of the fingers appears to be negligible.

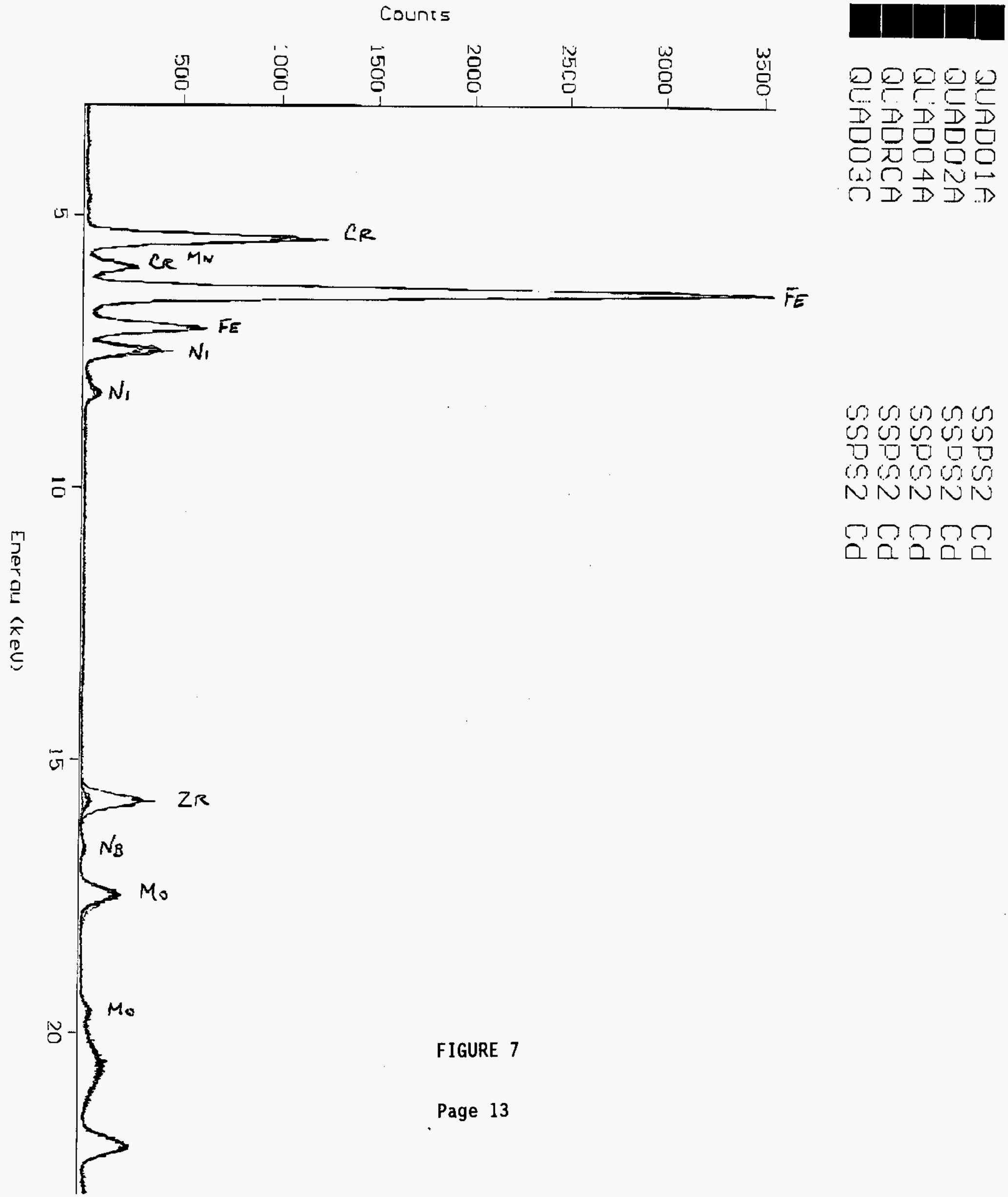

Counts
500
1000
1500
2000
2500
3000
3500
Energy (keV)
5
10
15
20
CR
CR MN
FE
FE
NI
NI
ZR
NB
MO
MO
QUAD01A SSPS2 Cd
QUAD02A SSPS2 Cd
QUAD04A SSPS2 Cd
QLADRCA SSPS2 Cd
QUAD03C SSPS2 Cd

FIGURE 7

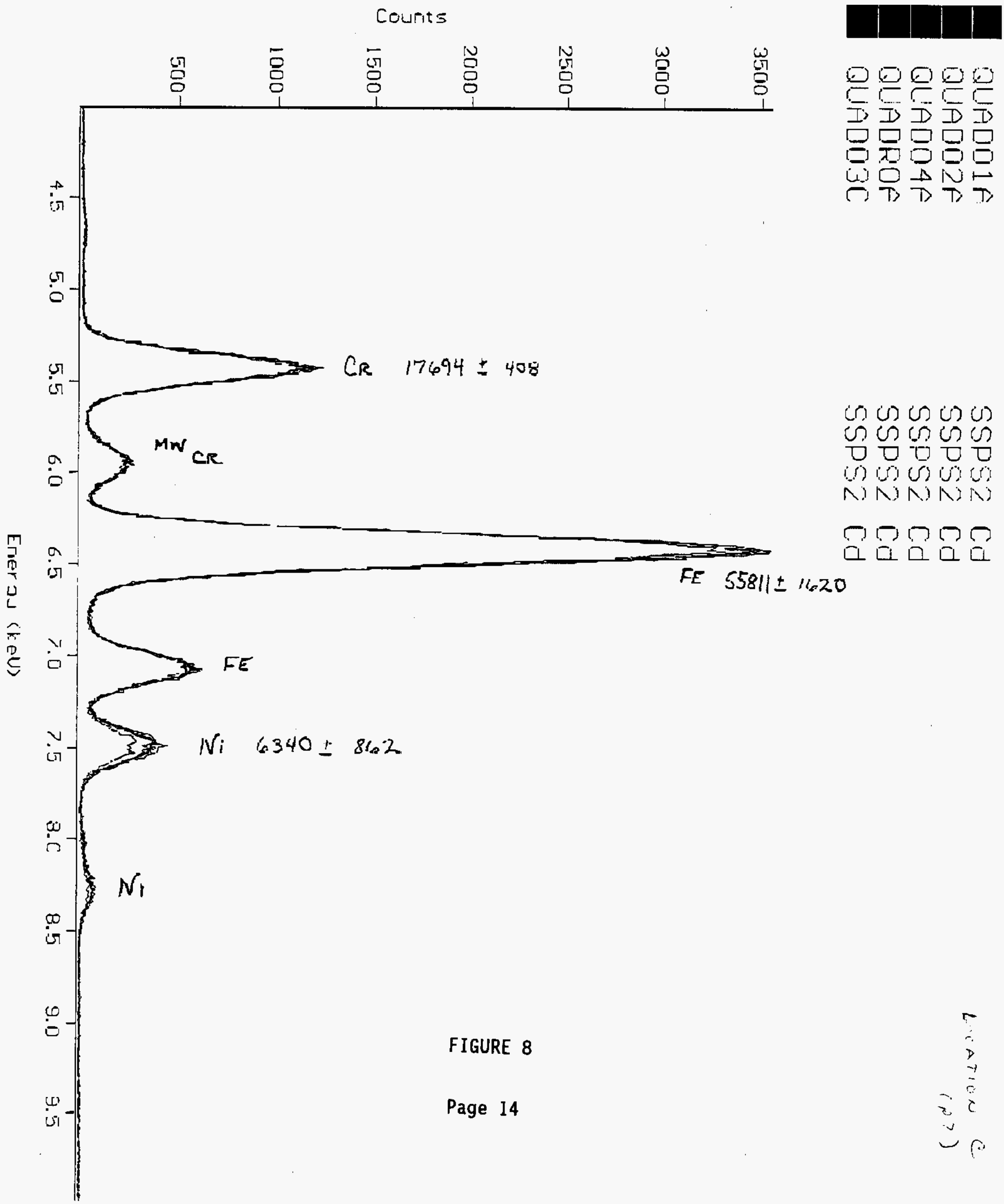

Counts
500
1000
1500
2000
2500
3000
3500
Energy (keV)
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
9.5
QUAD001F SSPS2 Cd
QUAD002F SSPS2 Cd
QUAD004F SSPS2 Cd
QUADR0F SSPS2 Cd
QUAD003C SSPS2 Cd
CR 17694 ± 408
MW CR
FE 55811 ± 1620
FE
Ni 6340 ± 862
Ni

FIGURE 8

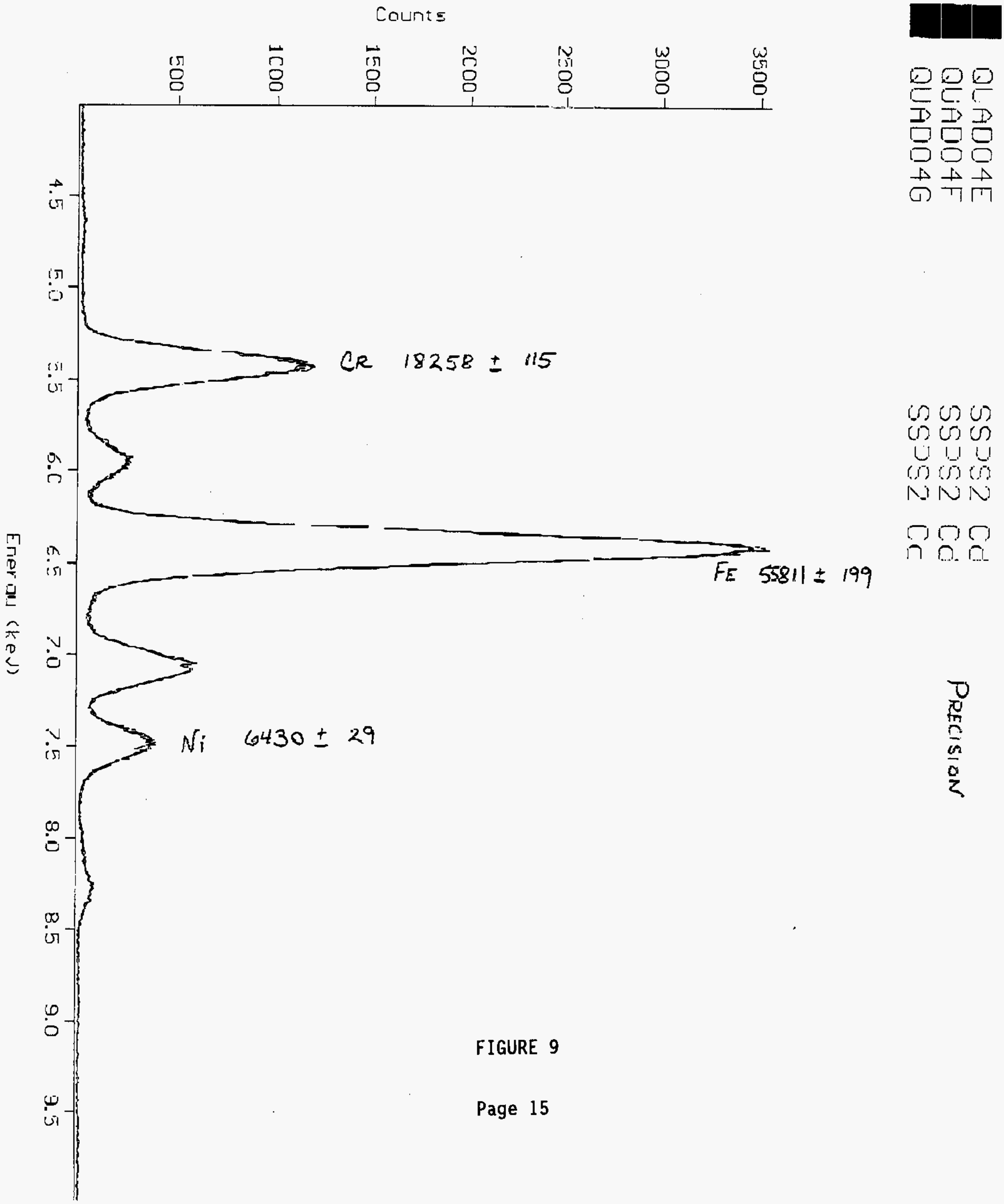

[redacted]
QUAD04E
QUAD04F
QUAD04G
PRECISION
Counts
500
1000
1500
2000
2500
3000
3500
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
9.5
Energy (keV)
CR 18258 ± 115
FE 55811 ± 199
Ni 6430 ± 29

FIGURE 9

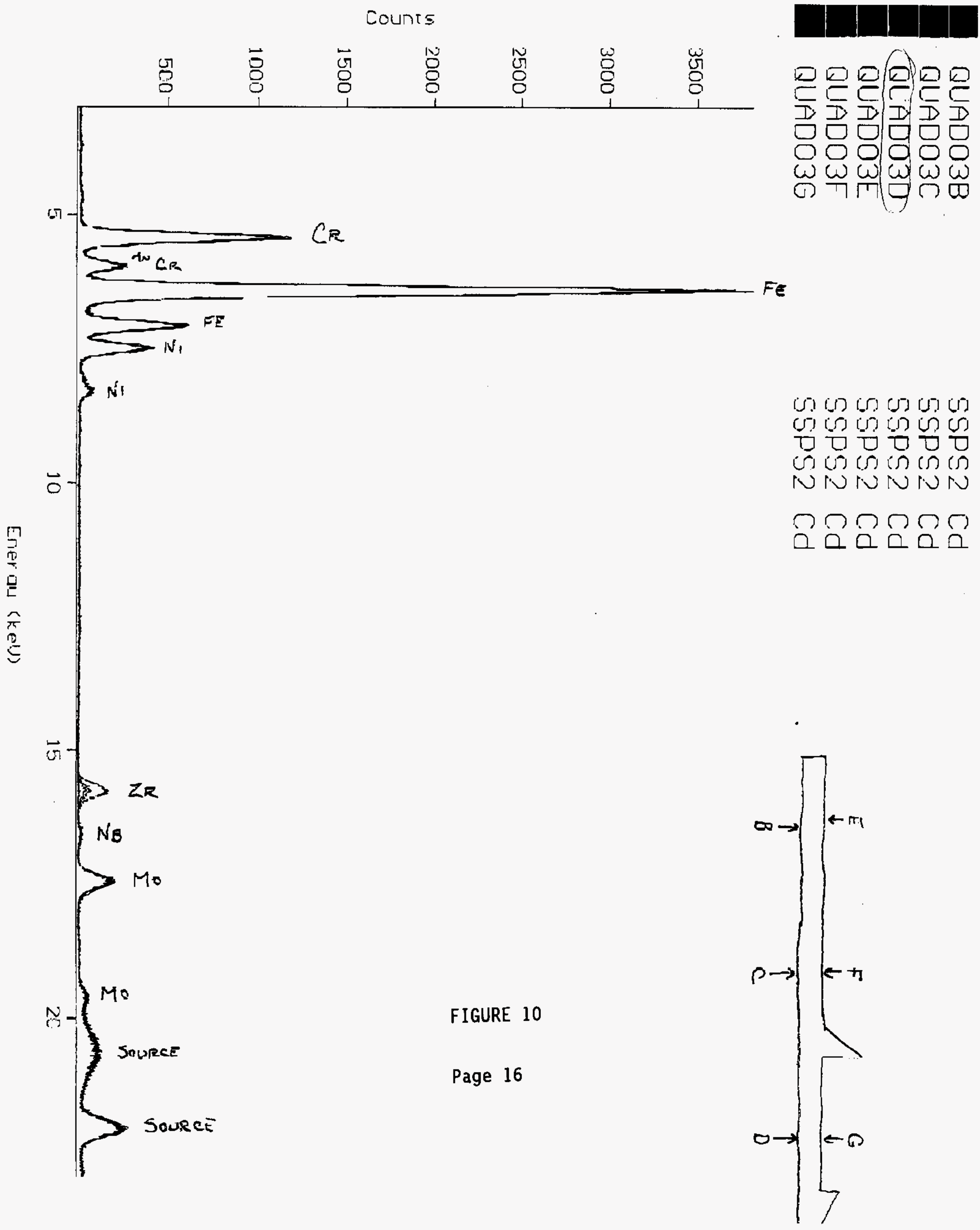
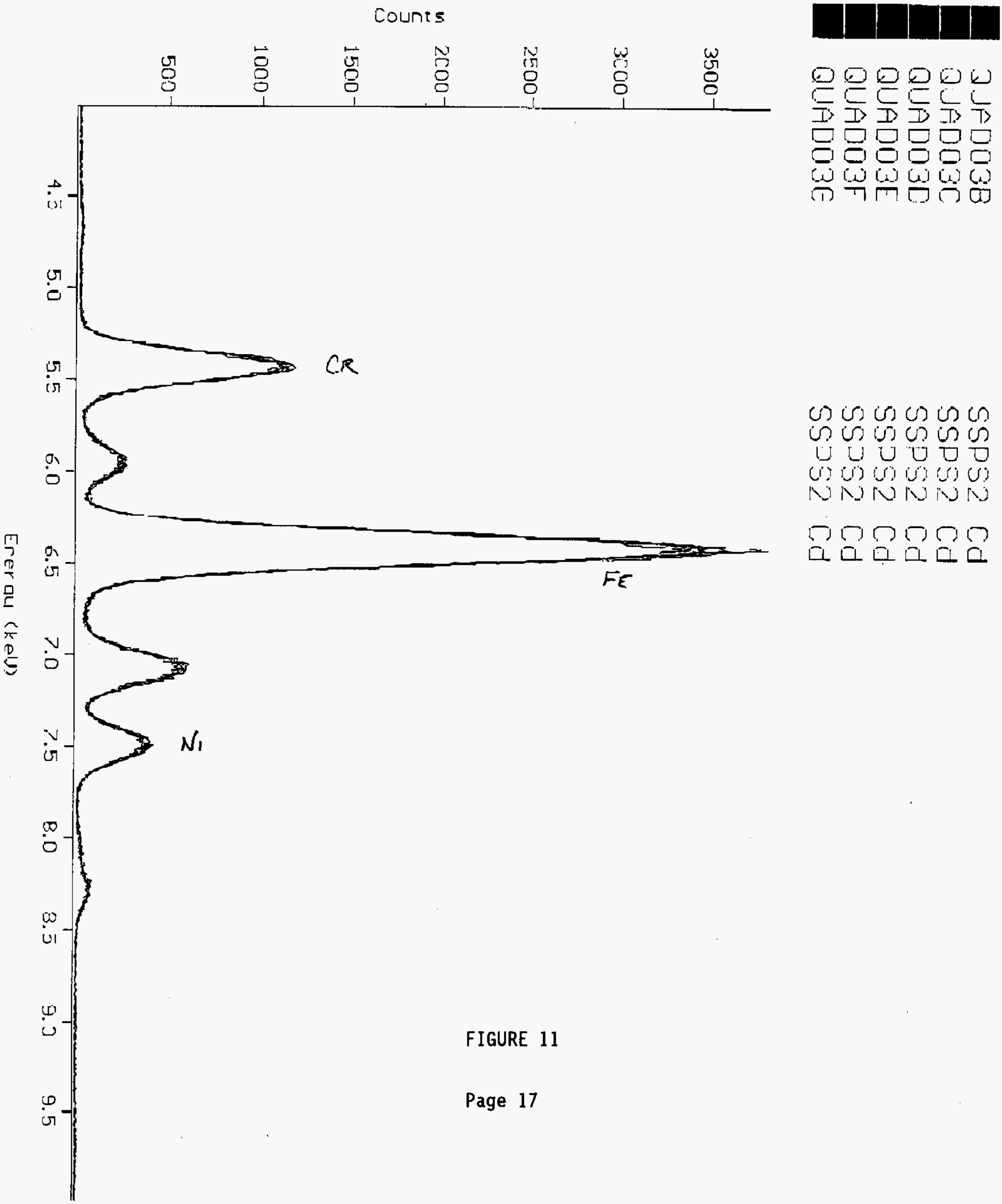

Counts
500
1000
1500
2000
2500
3000
3500
Energy (keV)
5
10
15
20
QUAD03B SSPS2 Cd
QUAD03C SSPS2 Cd
QUAD03D SSPS2 Cd
QUAD03E SSPS2 Cd
QUAD03F SSPS2 Cd
QUAD03G SSPS2 Cd
CR
Mn CR
Fe
FE
Ni
Ni
ZR
NB
Mo
Mo
SOURCE
SOURCE
B
C
D
E
F
G

FIGURE 10


Counts
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1000
1500
2000
2500
3000
3500
[redacted]
QUAD03C SSPS2 Cd
QUAD03D SSPS2 Cd
QUAD03E SSPS2 Cd
QUAD03F SSPS2 Cd
QUAD03G SSPS2 Cd
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
9.5
Energy (keV)
CR
FE
Ni

FIGURE 11

6.0 QUALIFICATION OF 300-SERIES STAINLESS STEEL

Type 304 stainless steel has at least 8% chromium. As can be seen in Reference 1, none of the 400 series steels have nearly that much chromium.

Comparison of the peaks in the X-ray fluorescence spectra can estimate the quantitative chromium content of an alloy. In the Table below the ratios of the nickel peak to that of iron and chromium are compared with the nickel concentrations.

	Ratio of Peak Height		Nickel Conc. in Alloy [%]
Alloy	Ni/Fe [%]	Ni/Cr [%]	
304	9.2	27.3	8-12
416	1.4	7.0	0
410	.95	5.0	.75
Fingers	10-13	30-42	

It can be seen that the fingers have more nickel than the 304 standard (as well as any of the 400-series standards) which indicates that they are not 400-series stainless.

If the ratio of Ni peak to the Fe peak height were greater than 9.2, the Table above indicates that the material is not 400-series stainless steel. However, if a number of 304 standards were run, a distribution of values around 9.2 would be expected. Considering the range of nickel concentrations which can be expected to be found in 304, the distribution of ratios would probably range from about 7 to 11.

Consequently if the Ni-Fe peak ratios is greater than 7.0 for a quadralatch finger, most likely, it is not 400-series stainless steel.

7.0 INSPECTION CRITERIA

Results from testing quadralatch fingers (Table above) indicate that the fingers exhibit a greater peak height for nickel than the 304 stainless standard. Used as a selection criteria, the nickel and iron peaks could be measured for each AUI and their ratio compared to a standard. If the ratio exceeds that of the standard, the part would be acceptable. Furthermore, a somewhat-lower acceptable value for the ratio could be inferred considering the range of nickel concentration naturally expected to be found in 304 billets.

A sample inspection plan for the purchased quadralatch fingers is needed which will not compromise the safety of Rotary Mode Core Sampling. In the Safety Analysis Report (Ref. 4) reference is made to explosive testing performed at the Bureau of Mines. Ten quadralatches were drop-tested in an explosive atmosphere. None caused ignition. These statistics imply, with 90% confidence, that the probably of ignition is less than 0.2.

If a sampling plan were used with an Acceptable Quality Level (AQL) equal to 1%, acceptance of a lot of quadralatch fingers would imply that 99% or more of them would be of the correct material. These defects would add to the 0.2 probability that was demonstrated by the Bureau of Mines testing resulting in a probability of 0.21 for ignition by a dropped quadralatch.

This increased risk is within the limits for Rotary Mode Core Sampling operability as calculated in the Safety Analysis Report.

8.0 REFERENCES

1) WHC-SD-WM-TRP-244, *Spark Resistant Metals*, authored by R. N. Johnson, PNNL.

2) H-2-690139 Casting, Finger

3) H. E. Bower and T. L. Gall "Metals Handbook, Desk Edition", American Society for Metals", Metals Park, Ohio

4) WHC-SD-WM-SAD-035, "*A Safety Assessment of Rotary Mode Core Sampling in Flammable Gas Single Shell Tanks: Hanford Site, Richland Washington*", Revision 0a.