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LONG TERM LABORATORY CORROSION MONITORING OF CALCINE BIN SET MATERIALS EXPOSED TO ZIRCONIA CALCINE

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ABSTRACT

Corrosion testing of Type 1025 carbon steel, 405, 304, 304L, 316L, and 347 stainless steels, and 6061-T6 aluminum were conducted in synthetic zirconia calcine to model long term corrosion performance of bin set material. Testing was conducted over a period of 17 years. The existing calcine bin set #1 is constructed of Type 405 stainless steel, 2 through 4 are constructed of Type 304 stainless steel and 5 through 7 are constructed of Type 304L stainless steel. The highest rate observed for Type 304L stainless steel was 8.1 x 10^{-7} inches per month. This would equal a wall thickness loss of about 5 mils after 500 years of storage.

Currently, the established schedule for removal of corrosion test coupons from the calcine storage bins is at the end of the 10^{th}, 100^{th}, 250^{th}, and 450^{th} year of solid storage service. Very low corrosion rates and metal oxide data determined from the long term laboratory test, in conjuntion with corrosion rates from the coupon assessment of the second bin set, indicate this schedule should be revised from 10 years to 50 years for the first assessment.

SUMMARY

High-level liquid wastes were generated at the Idaho Chemical Processing Plant as the result of uranium recovery operations from spent nuclear reactor fuel. These highly acidic wastes originated from the first cycle solvent extraction of the dissolved fuel elements and consisted of the dissolved fuel cladding elements, fission products, process adjustment chemicals, and decontamination chemicals.

These liquid wastes are converted to a solid form by calcination. This results in a volume reduction of the liquid to solid of approximately 8:1. Conversion to a solid form is benifical for long term interim dry storage due to storage costs and reduced long term corrosion rates.

Long term laboratory corrosion testing using synthetic zirconium calcine was undertaken in 1966 to evaluate candidate materials for future construction of calcine storage bins. These corrosion tests ran for 17 years at 350^0 C. The candidate materials tested were Type 1025 carbon steel, Type 405, 304, 304L, and 316L stainless steels, and 6061-T6 aluminum. The first calcine storage bin set was constructed with Type 405 stainless steel. A decision was made during the late 1960's to construct calcine storage bin sets 2 through 4 out of Type 304 stainless steel. Storage bin sets 5 through 7 were constructed at a later date out of Type 304L stainless steel. A maximum metal loss for Type 304L stainless steel of about 5 mils has been projected for the 500 year service life based on these tests.

Currently, the established schedule for removal of corrosion test coupons from the calcine storage bins is at the end of the 10th, 100th, 250th, and 450th year of solid storage service. Very low corrosion rates and metal oxide data determined from long term laboratory data, in conjuntion with corrosion rates from the coupon assessment of the second bin set, indicate this schedule should be revised from 10 years to 50 years for the first assessment.

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I. Introduction:

The worlds first plant scale facility for reduction of high and intermediate level liquid nuclear waste to a dry granualar solid waste was completed in 1963. This facility (The Demonstration Waste Calcining Facility (DWCF), later called the Waste Calcining Facility (WCF)) was built as a part of the Idaho Chemical Processing Plant (ICPP) at the Idaho National Engineering Laboratory (INEL). The WCF was originally heated by a recirculating liquid NaK (the eutectic alloy of 78 % potassium and 22 % sodium) heat transfer unit. The WCF was modified in 1970 to make use of the in-bed combustion of a hydrocarbon-oxygen system to provide heat for the operation. The system provided greater heat input and permitted higher throughput of the waste. After reducing 4,000,000 gallons of liquid nuclear waste to dry granular solids, the WCF was replaced by a New Waste Calcining Facility (NWCF) which is currently being operated at the ICPP. The NWCF uses the in-bed combustion system for calcination.

Conversion of liquid nuclear wastes to dry solids reduces the volume of waste to be stored at an approximate ratio of 8 gallons of liquid to one gallon of solids. In addition to the volume reduction of nuclear waste there is the safety advantage of reducing the potential for corrosion of the containment vessels during prolonged storage.

The earliest liquid wastes calcined were aluminum nitrate and the resulting calcine product was in amorphous form with some alpha alumina. This alumina calcine was produced at operating temperatures of 400° C and typically contained two weight percent water. The desired product was a granular solid with a typical mass mean particle diameter of 0.56-0.7 millimeter and a typical bulk density of 1.1 g/cm^3.

Zirconia waste calcination was started in 1966. Calcium nitrate is added to this waste stream to help stabilize the fluoride in the zirconium waste. The zirconia product has a typical water content of 0.6 weight percent, a typical mass median particle diameter of 0.2-0.5 millimeters and typical bulk density of 1.6 g/cm^3.

Representative chemical compositions of the calcines are listed in Table I.[1]

The first radioactive calcine produced at the ICPP was alumina calcine and was stored in the first set of calcine storage bins. These bins were constructed of Type 405 stainless steel with open space between the tanks to provide cooling by air circulation as shown in Figure 1. This concentric calcine bin set was built to section VIII of the ASME Boiler and Pressure Vessel code, 1956 edition.

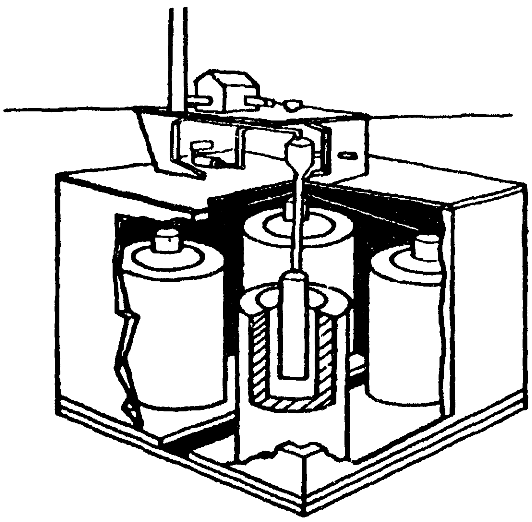


Figure 1 -Bin Set 1

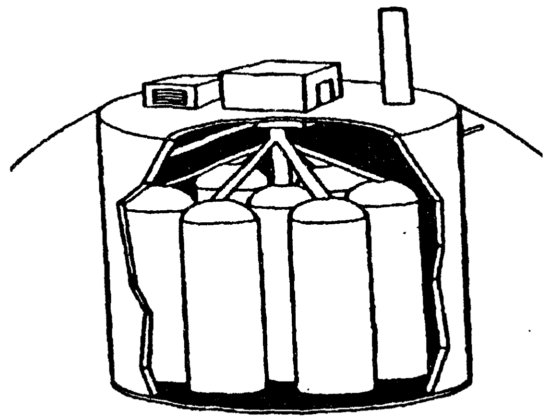


Figure 2 - Bin Set 2

The second (Figure 2), third (Figure 3), and fourth (Figure 4) sets of calcine bins were built as cylindrical tanks using Type 304 stainless steel with a maximum carbon content of 0.6 weight percent. These vessels were built to section VIII of the ASME Boiler and Pressure Vessel code. All subsequent calcine storage bin sets have been designed as annular type tanks to allow cooling air to flow up the center annulus as well as around the outside wall of the tank (Figures 5 and 6). These tanks were built using Type 304L stainless steel and are designed to meet the ASME unfired pressure vessel codes, ASME, Section VIII, Div. 2 and Section IX. Table II lists calcine bin sets, vessel configuration, material of construction, type of material stored, and date of filling.

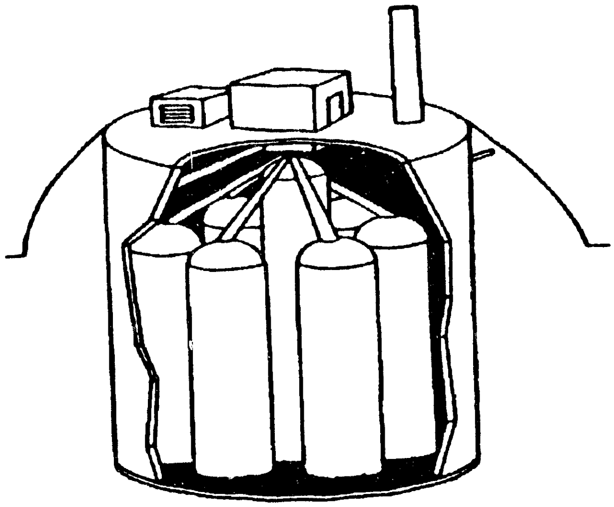


Figure 3 - Bin Set 3

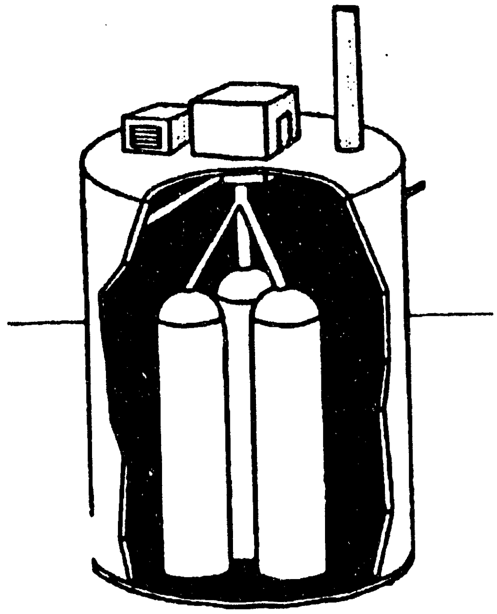


Figure 4 - Bin Set 4

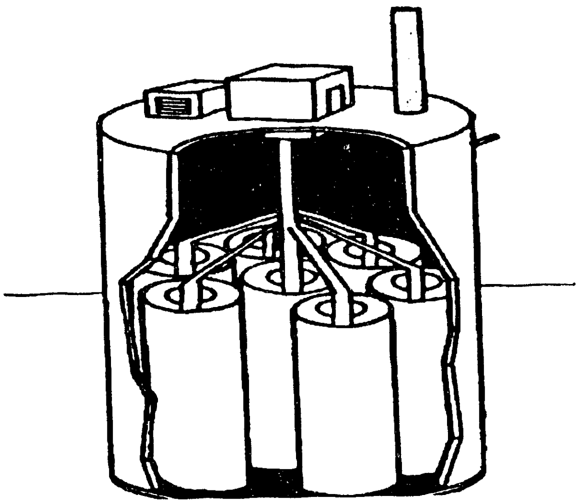


Figure 5 - Bin Set 5

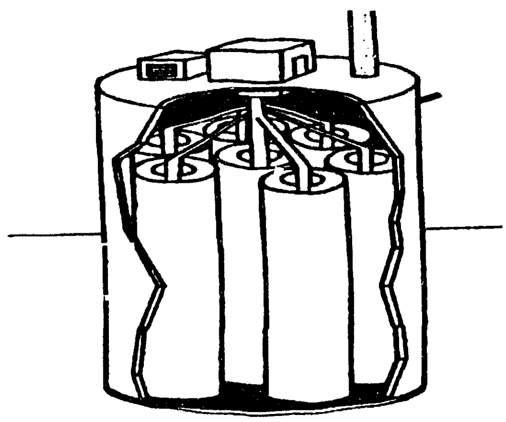


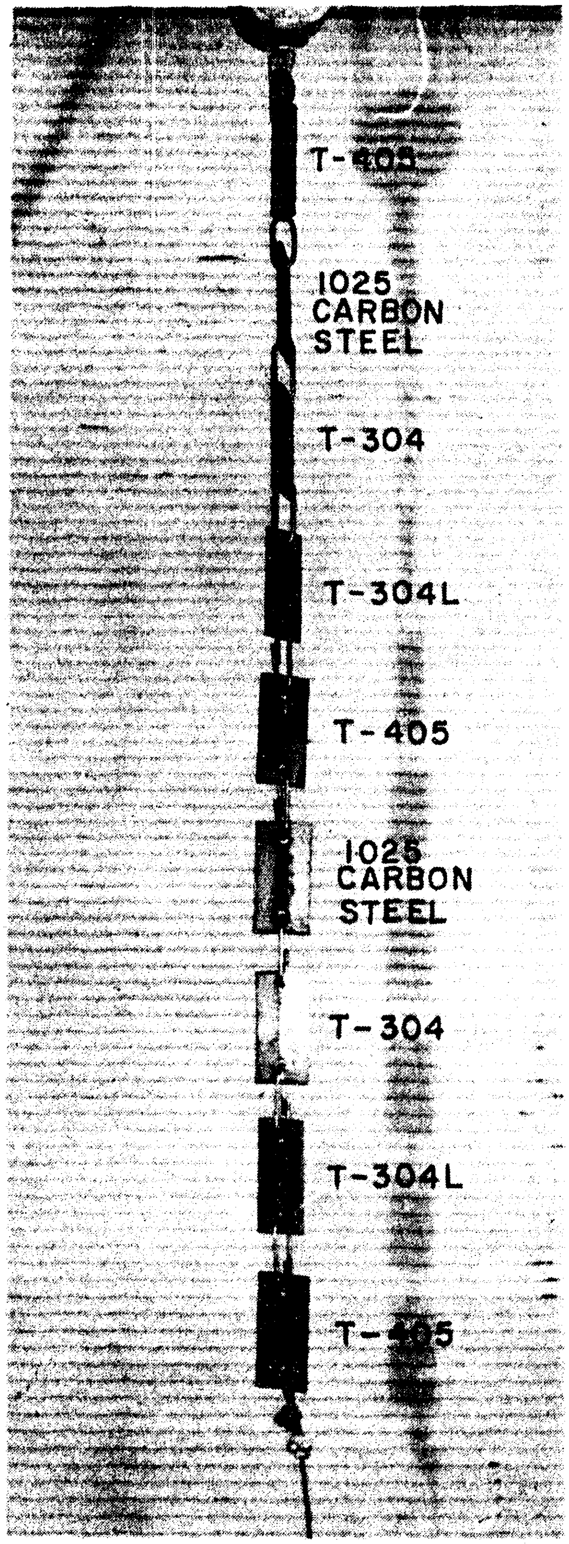
Figure 6 - Bin Sets 6 & 7

Table II

Summary of Calcine Bin Set Information

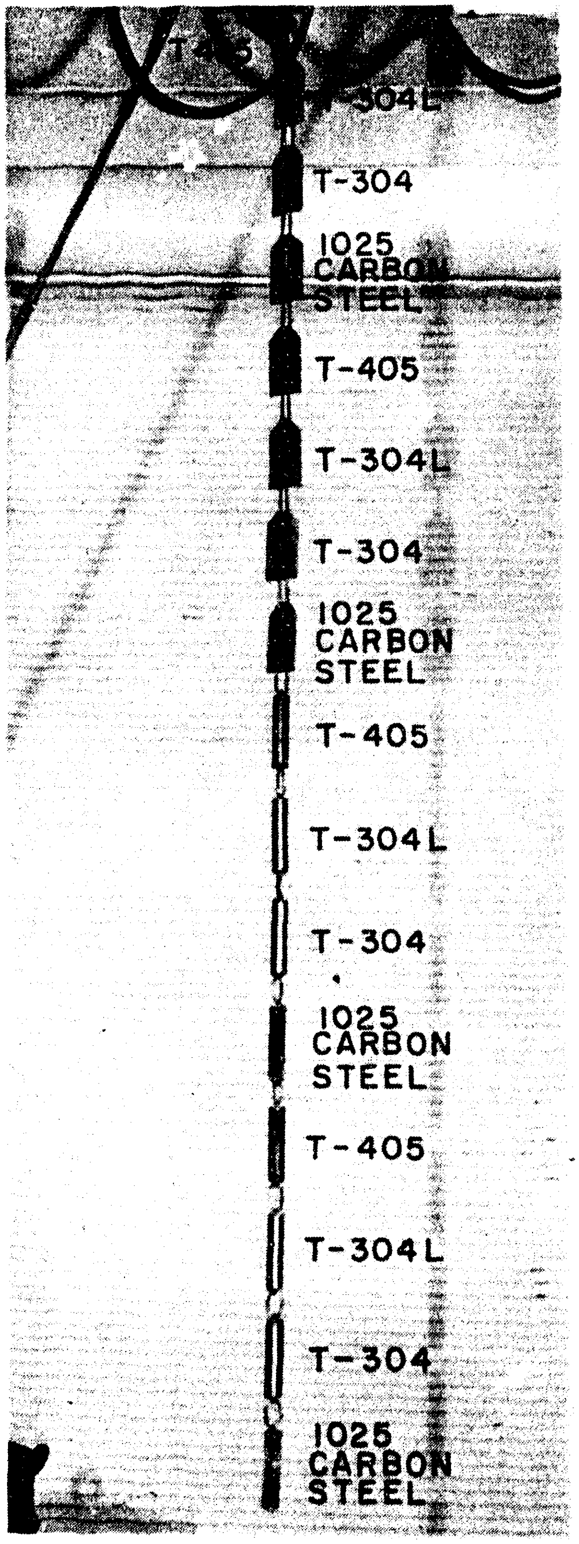
Bin Set #	Vessel Configuration	Material of Construction	Type of Calcine	Bin Set Filled
1	Concentric Tank	Type 405 SS	Alumina	Start-December 63 Filled-October 64
2	Cylindrical	Type 304 SS (0.06% C max)	Alumina Zirconia SS Sulfate	Start-April 66 Filled-October 72
3	Cylindrical	Type 304 SS (0.06 C% max)	SS Sulfate Zirconia - 3.5:1 Na Blend 5:1 Na Blend	Start-Feb 72 Filled-April 81
4	Cylindrical	Type 304SS (0.06 C% max)	Zirconia Zirconia-Na Blend	Start-Sept. 72 Filled-July 83
5	Annular Tanks	Type 304L SS	Zirconia Zirconia-Na Blend	Start-July 83 Filled-Jan. 92
6	Annular Tanks	Type 304L SS	Zirconia-Na Blend	Start-Jan. 93
7	Annular Tanks	Type 304L SS		

Corrosion test coupons were not included in the first set of calcine bins. Corrosion coupons have been placed in every set of calcine bins following the first bin set. The corrosion coupons are suspended in groups which include plate specimens of vessel welds (from weld tabs prepared when the vessels were fabricated) and plate specimens of Type 304 or Type 304L stainless steel prepared in the ICPP shop. There are also cylinderical specimens of Type 304 or 304L stainless steel which have been prepared in the ICPP shop.[2] Corrosion test coupons in the seventh bin set will include only coupons from fabricators weld tabs. Figures 7 and 8 are photographs of a corrosion coupon group which were removed from the second set of calcine bins. This group of coupons is not typical of later coupons in that later groups contain only Type 304 and 304L stainless steel.



WELDED PLATE TEST COUPONS
(EXPOSURE: ALUMINUM CALCINE, 6 YEARS)

Figure 7-Corrosion Coupons



WELDED PLATE & CYLINDER
TEST COUPONS
(EXPOSURE: ZIRCONIUM CALCINE,
2 YEARS)

Figure 8-Corrosion coupons

II. Laboratory Corrosion Tests:

Equipment and Test Procedure:

Laboratory corrosion tests were initiated in October 1966 that exposed "as-welded" test coupons of Type 405, 304, 304L, 316L, and 347 stainless steel as well as carbon steel and aluminum 6061-T6 (Table III) in zirconia calcine. The coupons were suspended in a stainless steel rack and were electrically isolated from each other and the rack by using quartz tubes and quartz spacers on the rods supporting the coupons (Figure 9). The test rack and coupons were contained in a stainless steel cylindrically shaped vessel which was enclosed in a clam-shell type electric furnace (Figure 10) to maintain the elevated test temperature.

Table III

Weight Change (g) of Corrosion Coupons after
2 Years Exposure[a]

Alloy	Bar 1	Bar 2	Bar 3	Bar 4	Bar 5	Bar 6
405 SS	+0.007	+0.0016	+0.0018[b]	+0.0008	+0.0005[b]	+0.0006
304 SS	+0.0004	+0.0006[b]	+0.0013	+0.0006[b]	+0.0002	+0.0001[b]
304L SS	+0.0010[b]	+0.0013	+0.0015	+0.0009[b]	+0.0004	+0.0002[b]
316L SS	+0.0002[b]	+0.0002	+0.0008b	+0.0001	+0.0001	0.0000[b]
347 SS	+0.0007[b]	+0.0013	+0.0017[b]	+0.0008	+0.0004[b]	0.0000
Carbon Steel	+0.0011	+0.0012[b]	+0.0025	+0.0012	+0.0002	+0.0004
Aluminum	+0.0004	+0.0004[b]	+0.0016	+0.0002[2]	+0.0006[b]	+0.0002

(a) Average temperatures from thermocouple readings between Bar 2 - Bar 3 was 206° C, Bar 3 - Bar 4 was 201° C, and Bar 4 - Bar 5 was 154° C.

(b) Average of 2 coupons

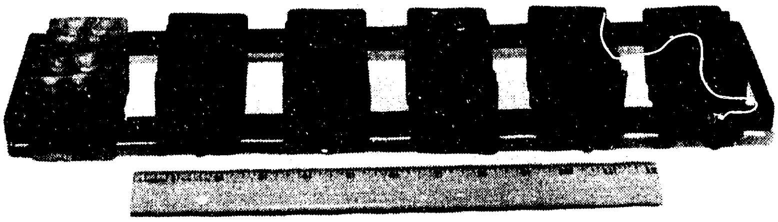


Figure 9 - Unirradiated Zr Calcine Coupons

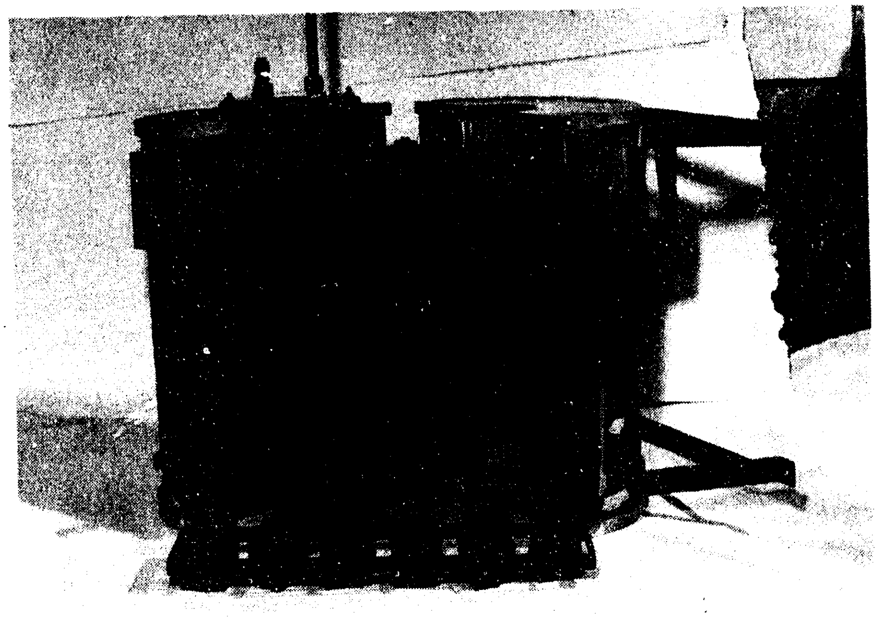


Figure 10 - Test Calcine Vessel and Heating Unit

The test vessel was equiped with a thermowell at the vessel mid-point which contained a thermocouple (TC) connected to a temperature controller system. The test vessel contained three other thermowells located at quarter length intervals of the vessel length. These thermowells contained thermocouples connected to a temperature readout to allow tracking of temperature at various levels in the test vessel as shown in Figure 11.

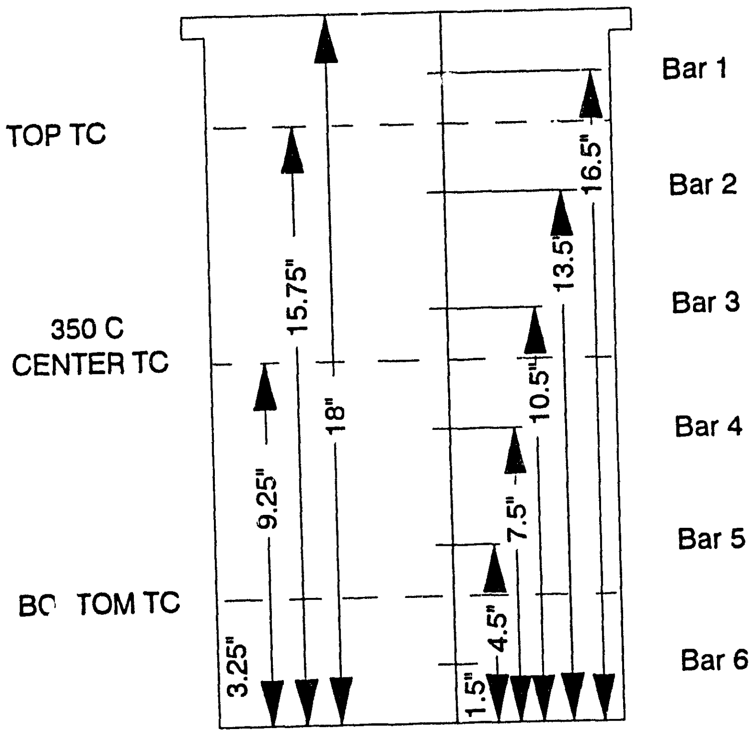


Figure 11 - Test Calciner Coupon and Thermocouple (TC) Locations

The test vessel was designed to be closed by a flanged head using a Teflon gasket with an open tube vented to the atmosphere to prevent any pressure or moisture build up.

The metal specimens were ultrasonically cleaned in distilled water, rinsed in acetone, dried for 30 minutes in an oven heated to 120 °F, air cooled and weighed between annual test cycles. Cleaned and weighed metal specimens were placed on the test rack which was then placed in the test vessel and enclosed with the electric heater. Table IV lists the chemical composition of the sample coupons.

Table IV
Analysis of Test Coupons

Material	C	Mn	P	S	Si	Ni	Cr	Cu	Mo	Co	Cb/Ta	Al	Weld Rod
Carbon Steel	No	Analysis	Available									Oxyweld	#65
304 SS	0.056	1.61	0.025	0.011	0.59	9.40	18.30	0.27	0.27	0.04			308
304L SS	0.03	0.46	0.023	0.017		10.20	19.07		0.23				308L
316L SS	0.022	1.83	0.026	0.012	0.42	13.52	17.68		?				316L
347 SS	0.07	1.69	0.034	0.022	0.63	10.44	17.05	0.20	0.29	0.17	0.87		347
405 SS	0.065	0.85	0.032	0.028	0.25		12.65					0.16	310
Aluminum 6061-T6	No	Analysis	Available										

The test vessel was then filled with a non-radioactive, synthetic zirconia calcine (Table V) that was produced during Run 14 in the twelve-inch diameter pilot plant calciner.[3]

Table V

Zirconia Calcine Composition from Run # 14

Zr(wt %)	Al(wt %)	Ca(wt %)	F(wt %)	Ru(wt %)
15.1	6.6	22.2	15.4	0.0007

A total of sixty coupons (seven to nine coupons of each alloy under test) were exposed for a total of seventeen years in nonradioactive zirconia calcine. These coupons are represented by Figures 12-25. The numbers under each coupon indicate the bar in which the coupon was located in the test vessel. Number 1 was at the top and number 6 was at the bottom of the test vessel.

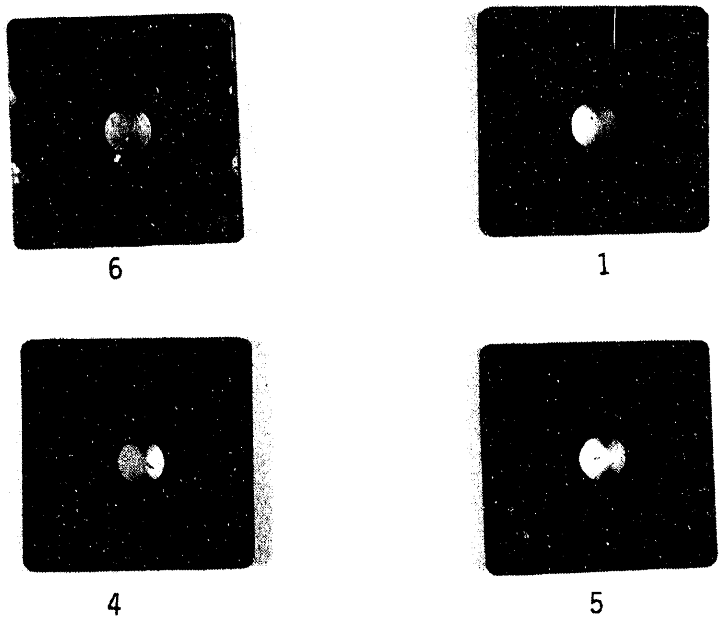


Figure 12-Type 304 SS

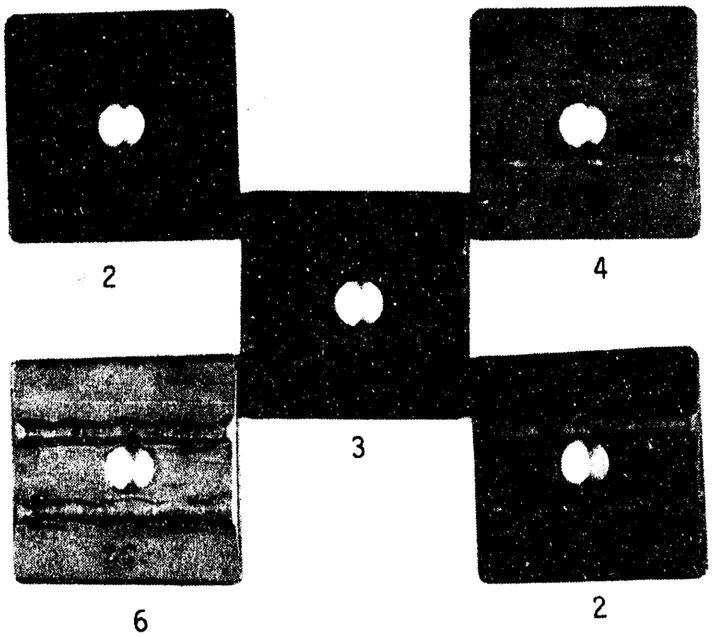


Figure 13- Type 304 SS

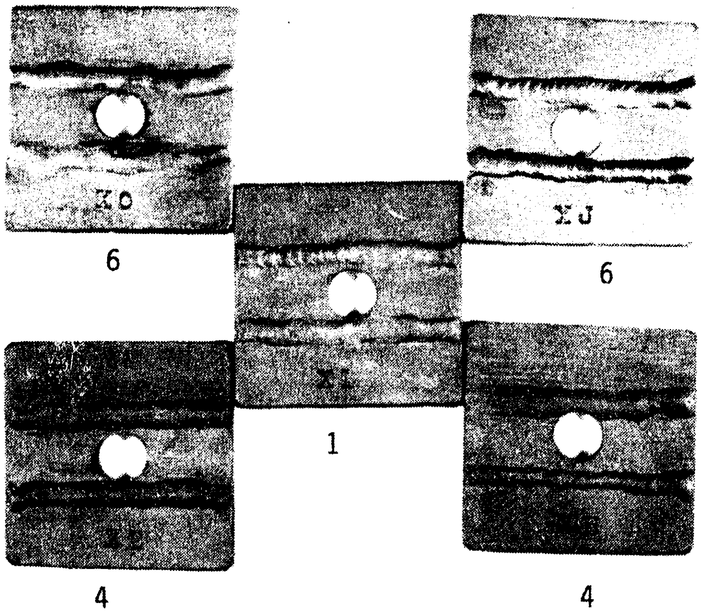


Figure 14 - Type 304L SS

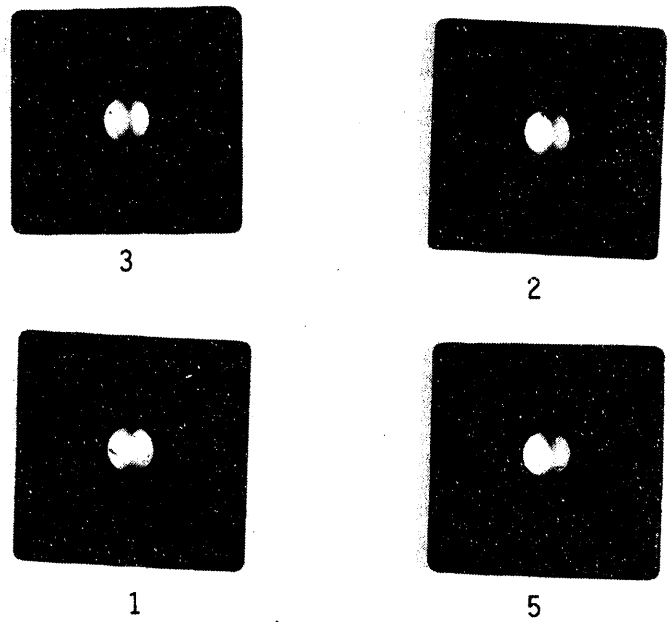


Figure 15 - Type 304L SS

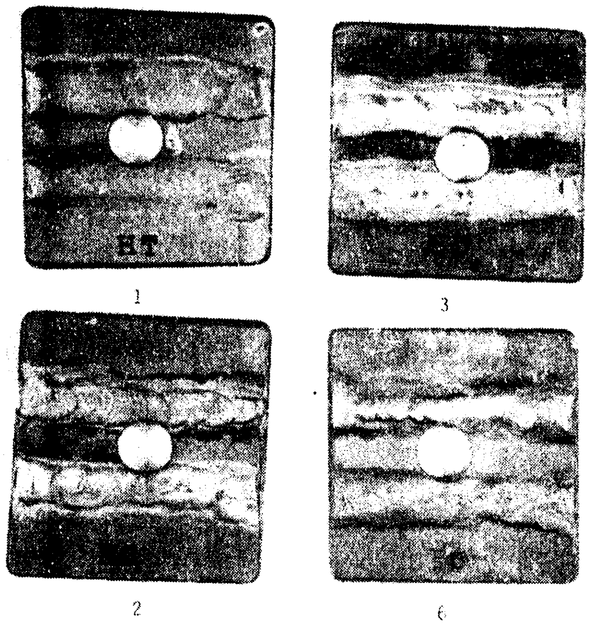


Figure 16 - Type 6061-T6 Aluminum

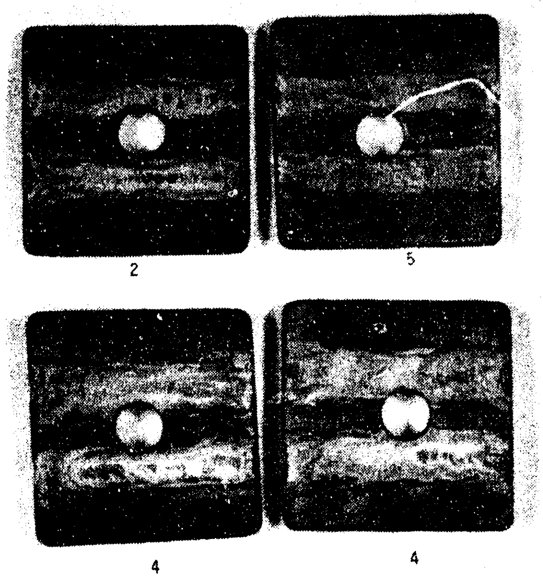


Figure 17- Type 6061 T-6 Aluminum

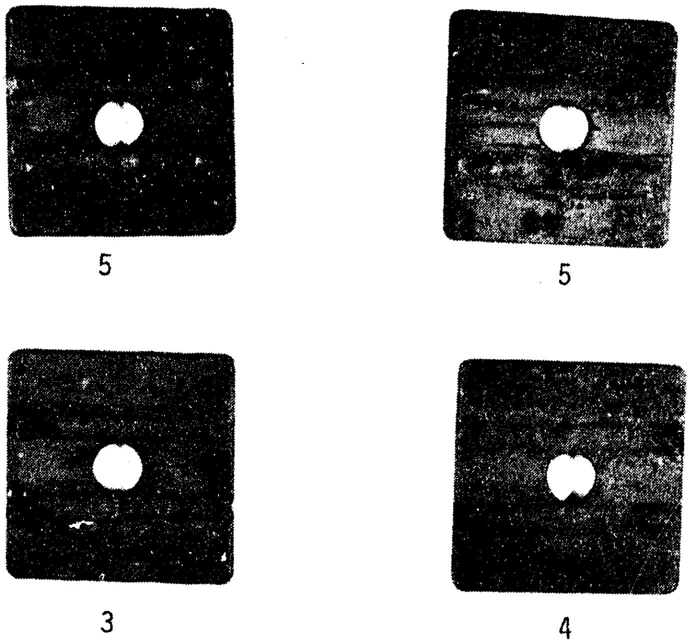


Figure 18 - Type 1025 Carbon Steel

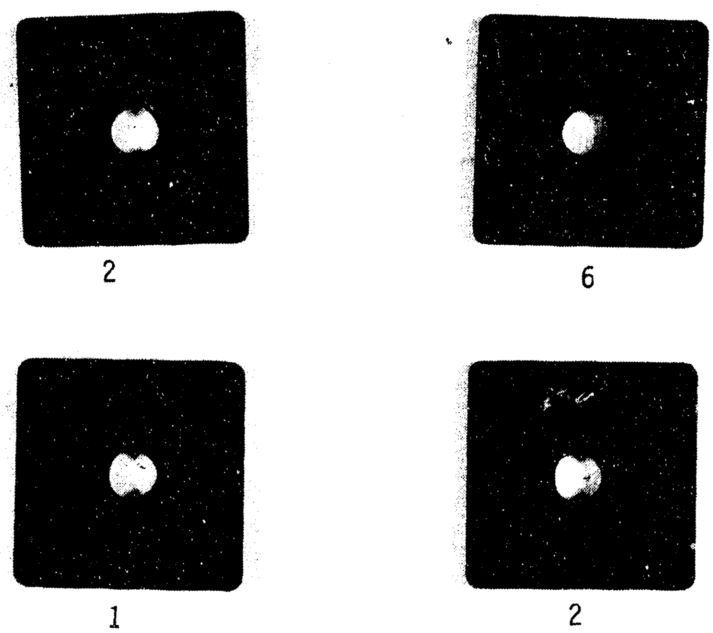


Figure 19 - Type 1025 Carbon Steel

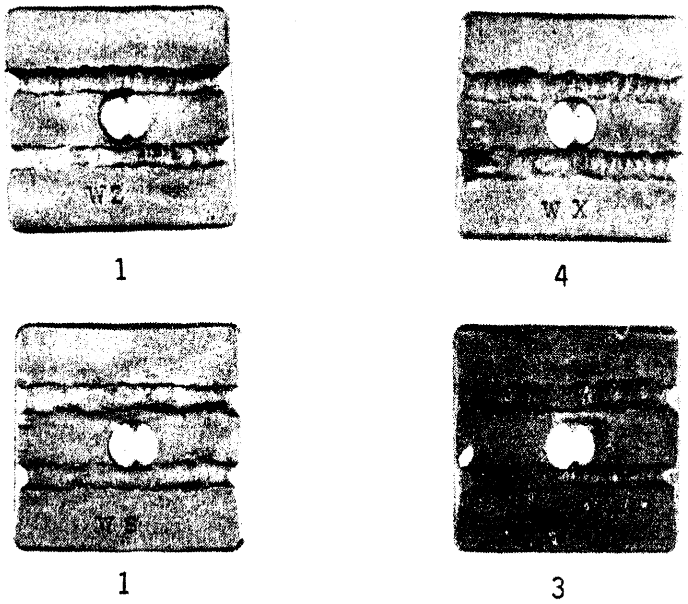


Figure 20 - Type 316L SS

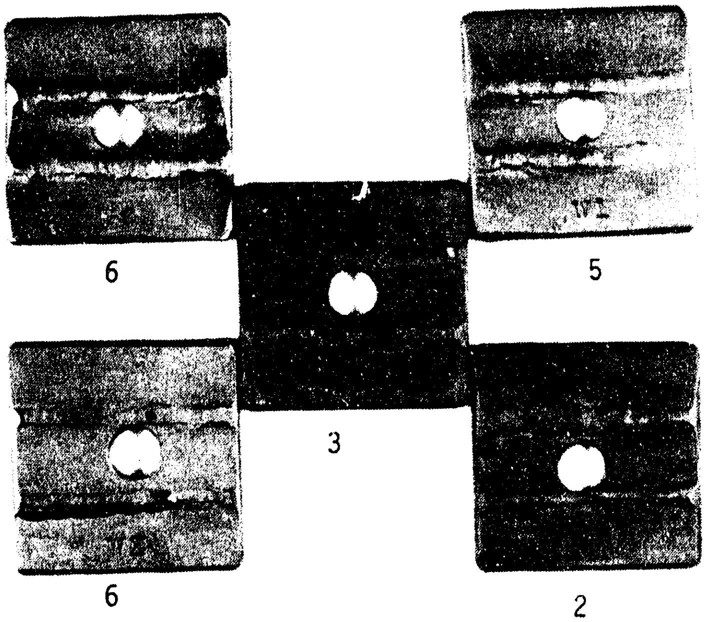


Figure 21- Type 316L SS

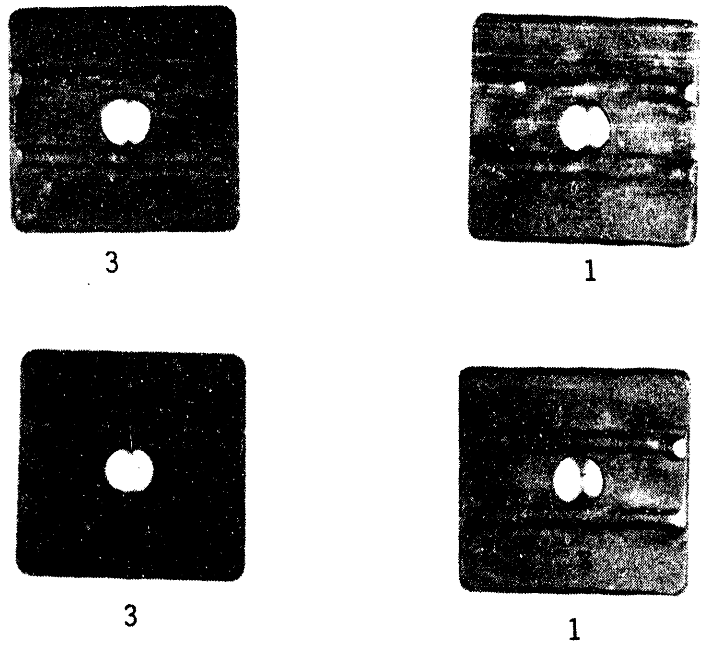


Figure 22 - Type 347 SS

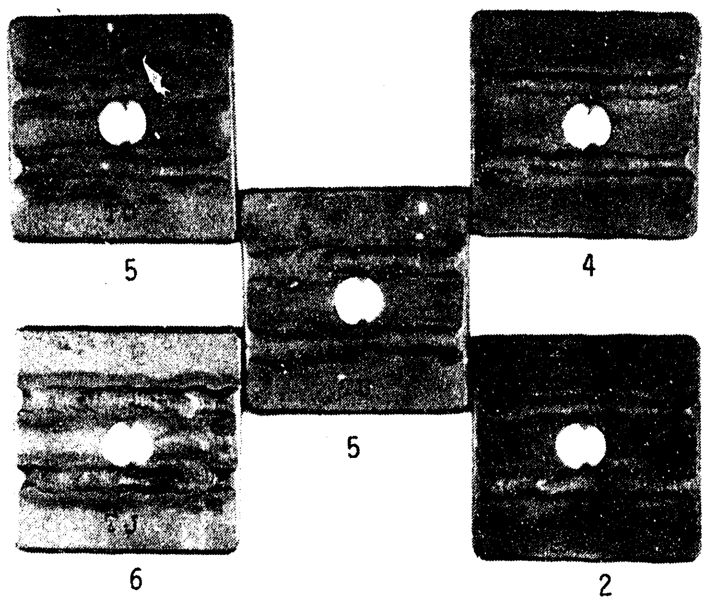


Figure 23 - Type 347 SS

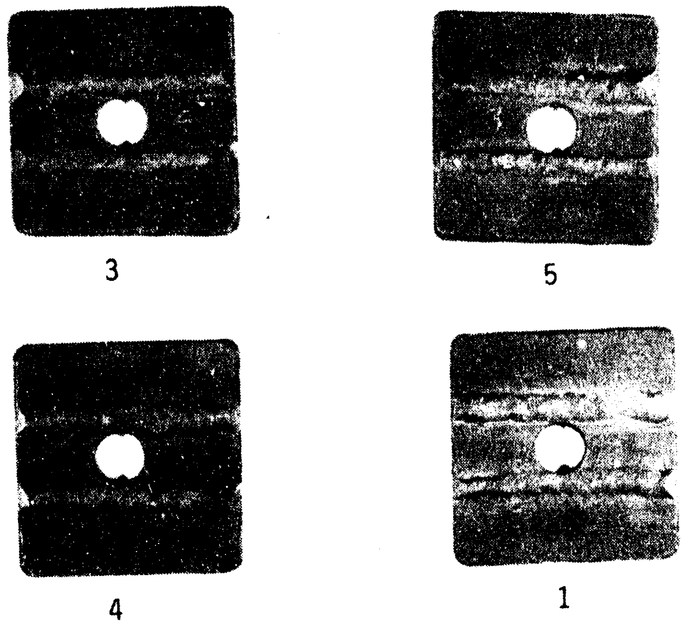


Figure 24 - Type 405 SS

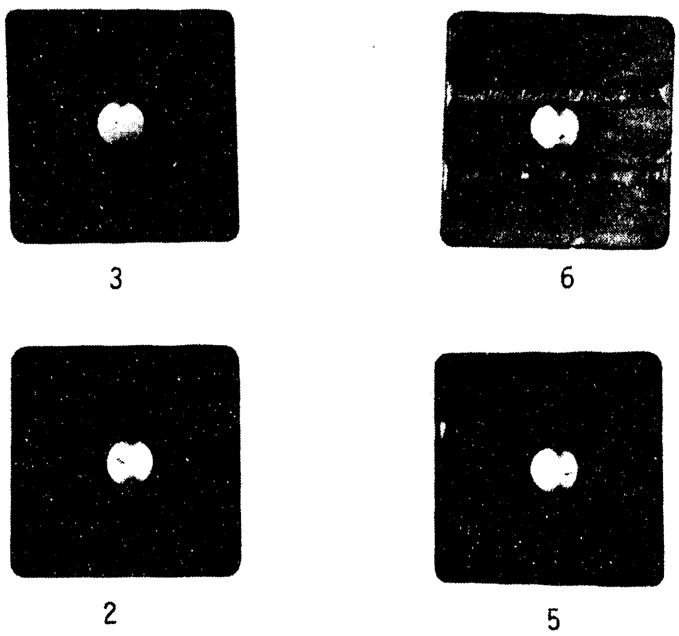


Figure 25 - Type 405 SS

III. Field Data:

Corrosion coupons were pulled from two different bins in Bin Set 2 after 6 years of exposure in alumina calcine and 2 years in zirconia calcine. These coupons were Type 1025 carbon steel, Type 304, Type 304L, and Type 405 stainless steels. Type 304 and Type 304L showed a projected corrosion rate of 7 and 5 mils respectively for 500 years at the 95% confidence level.[4]

IV. Results:

A total of sixty coupons, 7 to 9 of each alloy under test, were exposed in the laboratory over a total of seventeen years in nonradioactive zirconia calcine.

The first two years of exposure was at mid-point temperature average of 201° C. The average temperature measured at a point one quarter of the way down the test vessel from the top flange was 206° C while the average temperature measured at a point one quarter of the way up the test vessel from the bottom was 154° C. After this initial two year exposure period, coupons of all seven alloys under test showed a weight gain. This was presumed to be due to the formation of an oxide film on the coupons. The coupons generally demonstrated a discoloration which can be indicative of oxide formation.

During the following fifteen years of exposure the test vessel mid-point temperature was increased to an average of 301° C. The temperature one quarter of the way down from the top averaged 343° C and the temperature one quarter of the way up from the bottom averaged 204° C. The temperature was increased to model projected thermal increases in future zirconia fuel processing. After seventeen years of exposure, only Type 304, 304L, and 347 stainless steels showed a coupon weight loss at 301 °C. The weight loss of Type 304 and 304L stainless steel generally occured near the mid-point of the test vessel, while the weight loss of Type 347 stainless steel appeared to be scattered with relation to temperature. Coupons of Type 405 and 316L stainless steels, carbon steel, and aluminum showed weight gain after seventeen years of testing. Oxide layers on the test coupons were found to be tightly bound to the coupons and were not easily removed during the cleaning process prior to weighing. Table VI indicates the amount of time that a coupon either lost weight or gained weight before it changed direction. Table VII shows the final weight gain or loss after 17 years of exposure.

The coupons were cleaned each year after exposure to remove any loose oxide scale. They were ultrasonically cleaned in distilled water for 15 minutes, rinsed in distilled water, dipped in isopropyl alcohol, and dried in a heated oven at 100° F for 30 minutes, air cooled and weighed.

Table VI

Coupon Exposure (Yrs) in Heated Zirconium Calcine Prior to Weight Reversal

Alloy	Bar 1	Bar 2	Bar 3	Bar 4	Bar 5	Bar 6
405 SS	9	7[a]	8[a]	8[a]	8[a]	8
304 SS	9[a]	6	4	6[a]	6	8[a]
304L SS	6[a]	4	4[a]	6	6	7[a]
316L SS	6[a]	6	6[a]	6	6[a]	6
347 SS	9			6	6[a]	8
Carbon Steel	9[a]	8[a]	13	8[a]	6	8
Aluminum 6061-T6	9	17	17	17	8	8

(a) Average of two coupons

Table VII

Weight Change (g) of Corrosion Coupons After Seventeen Years Exposure

Alloy	Bar 1	Bar 2	Bar 3	Bar 4	Bar 5	Bar 6
405 SS	+0.0315	+0.1159	+0.1464[c]	+0.1245	+0.0602[c]	+0.0066
304 SS	+0.0041	0.0000[c]	-0.0082	-0.0159[c]	-0.0840	+0.0009[c]
304L SS	-0.0252[c]	-0.0157	-0.0295	-0.0098[c]	+0.0121	+0.0021[c]
316L SS	+0.0019[c]	+0.0186	+0.0188[c]	+0.0100	+0.0026	+0.0009[c]
347 SS	+0.0005[c]	+0.0060	-0.0101[c]	+0.0047	-0.0123[c]	+0.0027
Carbon Steel	+0.0541	+0.1894[c]	+0.2605	+0.0796	+0.0514[c]	+0.0047
Aluminum 6061-T6	+0.0152	+0.0300[c]	+0.0411	+0.0316[c]	+0.0120	+0.0012

Note: The first two years of exposure were at lower temperatures (Reference Table III)

Average temperature for thermocouple readings between Bar 2 - Bar 3 was 343° C, Bar 3 - Bar 4 was 301° C, and Bar 4 - Bar 5 was 204° C.
Average of 2 coupons (a)

One of the Type 304L stainless steel samples exposed at the nominal temperature of 350° C for 17 years was sectioned, mounted, and measured using an optical comparator to determine the oxide thickness. One of the original unexposed coupons of Type 304L stainless steel was sectioned, and the original oxide layer thickness was determined using an Auger Electron Spectroscopy (AES) sputtering technique. This provided a composition of the oxide layer after exposure. A second unexposed Type 304L stainless steel coupon from the original sample set was heated at 350° C in a laboratory oven exposed to the air for 30 days. The sample oxide thickness was then measured using the AES sputtering technique. This allowed a comparison between the oxide formed in contact with the zirconia calcine and the oxide formed when heated in air.

It appears that there is little difference in thickness between the air exposure and the zirconia calcine exposure. If you extrapolate the 30 day air data out to 17 years the comparison is 1.5 X 10^5 Å vs 1.2 X 10^5 Å in thickness. Table VIII lists the oxide layer thicknesses. Figure 26 shows the cross section of the oxide layer formed on Type 304L stainless steel after 17 years exposure to simulated zirconia calcine. Analysis of the calcine exposed oxide layer showed no incorporation of any of the elements found in the zirconia calcine composition. This indicates that the oxide layer is the same as one would find if you exposed Type 304L at 350° C in air for 17 years.

Table VIII

Comparison of Laboratory Coupon Oxide Thickness

Type 304L Coupon #	Exposure Period	Temperature	Oxide Thickness
XA As Received	N. A.	20° C	52.1 Å
XU	17 years	350 °C Zr Calcine	1.2 X 10^5 Å
XV	30 days	350 °C Air	719 Å

Figure 26 -Oxide layer of Type 304L SS (600X)
after 17 years exposure at 350° C- $\approx 1.2 \times 10^7$ - 1.5×10^7 Å Thick

Typical scans of sputtering profiles and oxide compositions for Type 304L stainless steel in the as received condition, exposed at 350° C in air for 30 days and exposed in synthetic zirconia calcine for 17 years are included in the appendix.

Area measurements were made on the remaining portions of the Type 304L stainless steel coupons to determine oxide thickness. One coupon was then decontaminated in the same manner as the coupons pulled from Bin Set 2 in 1975.[5] The decontamination procedure consisted of boiling the coupon in a propriatary alkaline permanganate for 45 minutes, dipping the coupon in oxalic acid at 80° C to reduce the permanganate, rinsing in distilled water and then placing in boiling 4 M HNO_3 for 15 minutes. The coupon was then rinsed in distilled water, ultrasonically cleaned in distilled water, oven dried and weighed. A Type 304L stainless steel blank was also decontaminated to determine a weight loss caused by the decontaminating solution which was then subtracted from the final weight loss.

After the normalized weight loss was determined, corrosion rates were calculated and extrapolated for 500 years exposure. This data was then compared with the actual data derived from corrosion rates determined for Bin Set 2. These extrapolated rates compared very closely. Bin Set 2 after 2 years exposure to radioactive zirconia calcine showed a projected 5 mils total loss for 500 years and the laboratory coupons exposed for 17 years showed a projected loss of 4.48 mils for 500 years. While it may be argued that you can not project corrosion rates for 500 years, the data generated in the laboratory was run at a higher temperature than that reached in the calcine storage bins and is therefore considered conservative.

The corrosion rate of the laboratory coupons is based upon the stripping of the oxide layer. This layer is protective in nature and will most likely not spall from the surface when in contact with the calcine. In order to determine the benifical effect of the oxide layer one of the laboratory Type 304L test coupons was cleaned ultrasonically to remove loosely adhearing particles. The projected loss after 500 years was 0.79 mils. This would indicate that the decontamination solution used to remove the oxide layer increased the expected corrosion rate. This data is compared in Table IX.

Table IX

Comparison of Bin Set 2 Coupons with Laboratory Test Coupons

Coupon Location	Coupon Condition	Exposure Period	Corrosion Loss Estimated for 500 Yrs
Bin Set 2 Type 304L SS	Decontaminated Scale Removed	2 years	5 mils
Laboratory Type 304L SS	Decontaminated Scale Removed	17 years	4.48 mils
Laboratory Type 304L SS	Ultrasonically Cleaned	17 years	0.79 mils

Coupons were decontaminated in a propriatary alkaline permanganate for 45 minutes, dipped in Oxalic Acid for 2 minutes, water rinsed, and placed in 4 M HNO_3 for 15 minutes. A blank was used for rate calculations.

Laboratory corrosion rates and projected 500 year material loss for Type 316L, Type 304, Type 347, Type 405 stainless steel, 1025 carbon steel, and 6061-T6 aluminum are presented in Table X. These coupons received the same exposure as the Type 304L stainless steel coupon. Oxide thickness measurements were not made on these coupons since it was determined early on that Type 304L stainless steel would be the material of choice for all future calcine bins. Figure 27 shows the coupons prior to decontamination. Figure 28 shows the coupons after decontamination.

Table X

Corrosion Rates of Other Alloys Tested

Alloy	Exposure Period Years	Corrosion Rate MPY	Estimated 500 Year loss (Mils)
316L SS	17	0.00548	2.74
304 SS	17	0.00937	4.69
347 SS	17	0.01280	6.38
405 SS	17	0.000879	0.44
1025 Carbon Steel	17	0.01671	8.36
6061-T6 Aluminum	17	0.02220	11.10

Note: Type 405 SS, Type 1025 carbon steel, and Type 6061-T6 aluminum did not have blanks subtracted in determination of corrosion rates. The unexposed blanks had a high corrosion rate, which if subtracted would have resulted in a less conservative corrosion rate for the exposed samples. These high corrosion rates for the blanks indicate that the oxide layers on the exposed coupon were protective in the decontamination solutions used. Exterior bin corrosion was not tested, however, should the metal temperature drop to ambient in a humid vault environment the above three alloys would see an increased corrosion rate. The austinetic stainless steels would have a very low corrosion rate under these conditions.

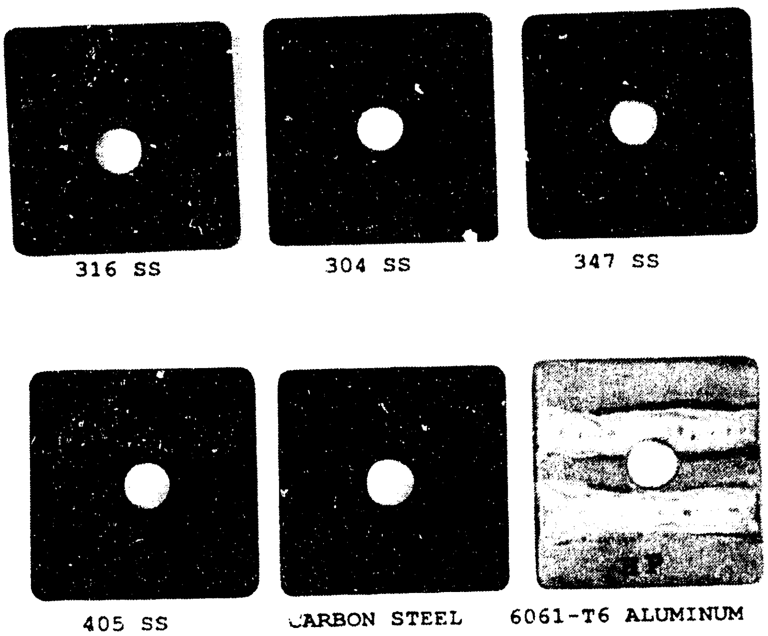


Figure 27 - Corrosion Coupons Prior to Decontamination

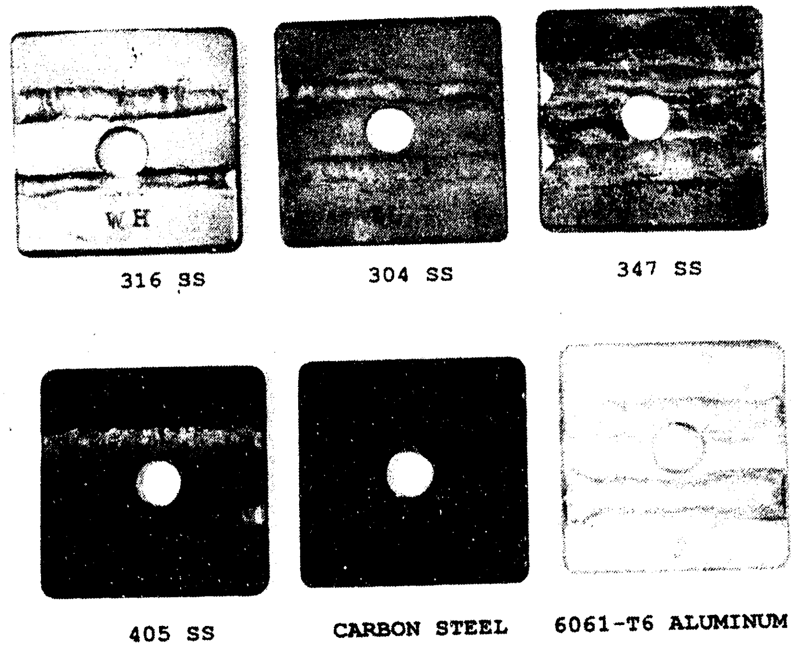


Figure 28 - Coupons after Decontamination

Historical data on the wall temperatures of Bin Sets 2 through 5 were researched. The wall temperatures measured with thermocouples through the year 1990 show the highest temperatures recorded to be approximately 100° C. This means the laboratory tests are very conservative since they were run at 350° C. Thermocouple data for the bin sets are presented in Figures 29-35.

BIN SET II WALL TEMPERATURES
TYPE 304 STAINLESS STEEL

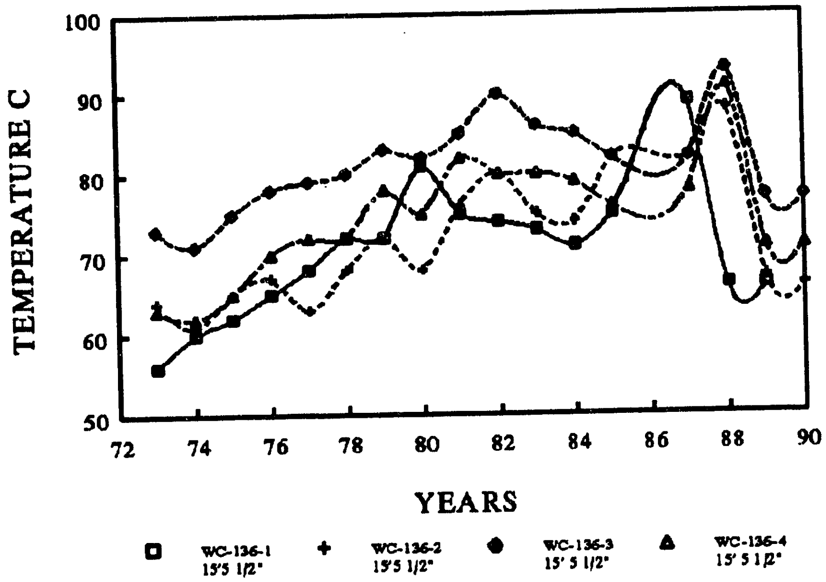


Figure 29

BIN SET II WALL TEMPERATURES
TYPE 304 STAINLESS STEEL

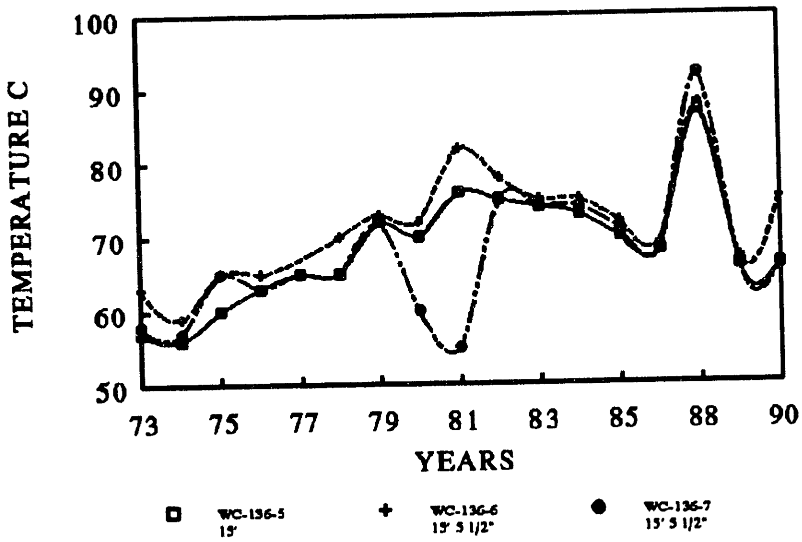


Figure 30

BIN SET III WALL TEMPERATURES
TYPE 304 STAINLESS STEEL

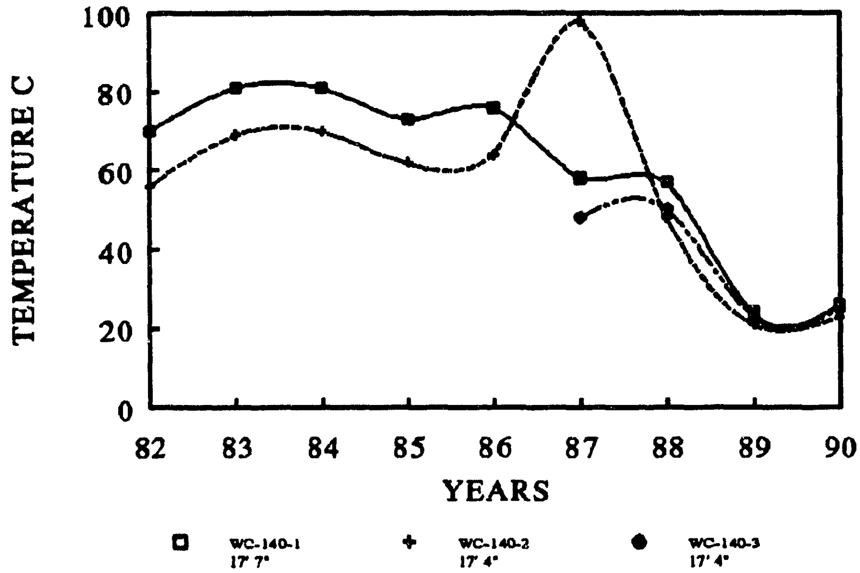


Figure 31

BIN SET III WALL TEMPERATURES
TYPE 304 STAINLESS STEEL

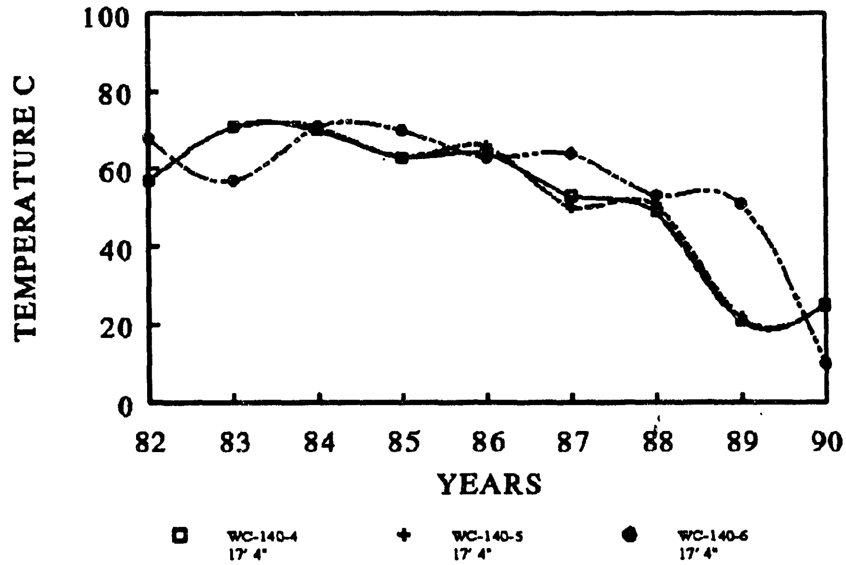


Figure 32

BIN SET IV WALL TEMPERATURES
TYPE 304 STAINLESS STEEL

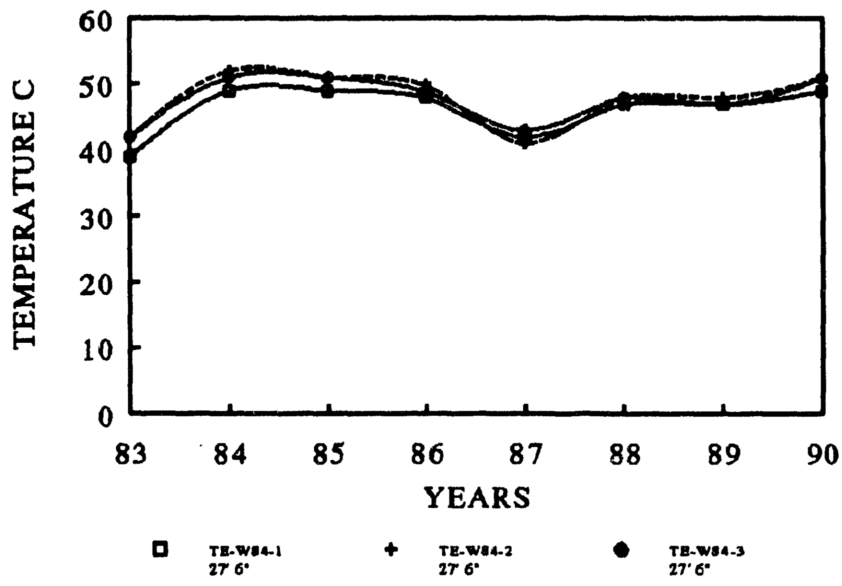


Figure 33

BIN SET V WALL TEMPERATURES
TYPE 304L STAINLESS STEEL

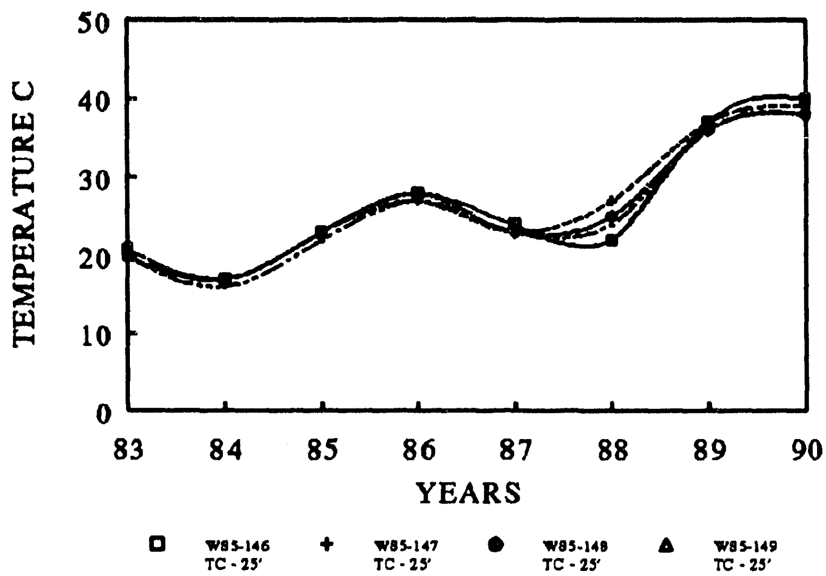


Figure 34

BIN SET V WALL TEMPERATURES
TYPE 304L STAINLESS STEEL

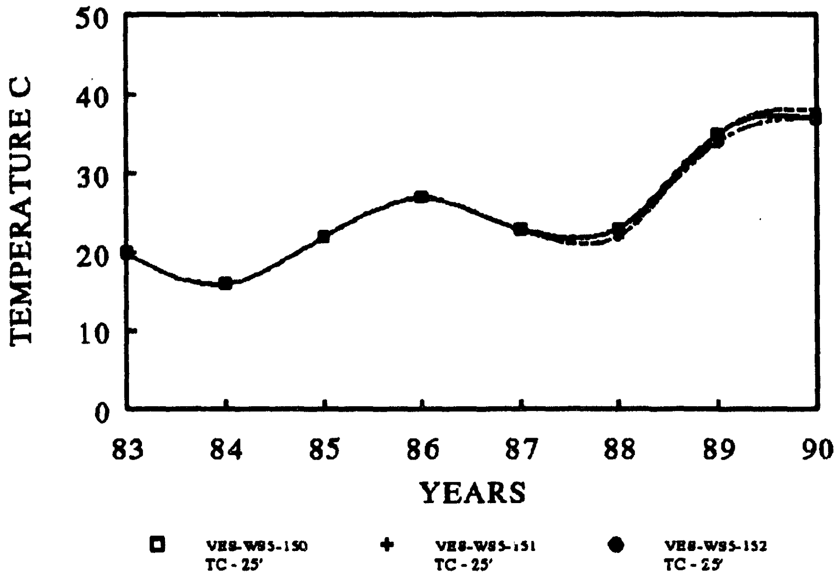


Figure 35

Conclusion:

Long term laboratory corrosion tests of carbon steel, aluminum 6061-T6, Type 405, 304, 304L, 316L, and 347 stainless steel show low corrosion rates for zirconia calcined storage wastes. Type 304 and 304L stainless steel show low corrosion rates which can extend the time interval for the first coupon pull from 10 years to 50 years of exposure.

REFERENCES

(1) J. R. Berreth, "Inventoried and Properties of ICPP Calcined High-Level Waste", WINCO -1050 page 4-2, February 1988.

(2) The Phillips Petroleum Company, Calcine Storage Bin Architectural Drawing CPP-E-4068, 054068, "Second Solids Storage Corrosion Coupon Assembly Details & Identification", dated June 26,1966.

(3) C. A. Zimmerman, "Laboratory Note Book -U-1211", page 11 , dated September 7, 1966, and Phillips Petroleum Company, "Request for Analysis by Analytical Section-Log # 66-5030", dated August 4, 1966.

(4) T. L. Hoffman, "Corrosion Monitoring of Storage Bins for Radioactive Calcines", ICP-1071, UC-70, October 1975.

(5) W. J. Dirk, Letter Dirk-04-91, to R. E. Mizia, , "Run Plan for Removing Oxide Coating from a Type 304L Cold Calcine Corrosion Coupon", dated March 4, 1991.

APPENDIX

A. Sample XU Type 304L stainless steel exposed in zirconia calcine for 17 years

1. AES survey of kinetic energies of oxide in the as received condition.
2. AES profile of oxide layer after sputtering for 120 minutes.
3. Atomic concentration after initial sputtering for 120 minutes.
4. AES survey of kinetic energies after sputtering for 200 minutes.
5. Atomic concentration after sputtering for 200 minutes.

B. Sample XA Type 304L stainless steel in the as received condition (native oxide) exposed only to ambient air.

1. AES survey of kinetic energies of the oxide in the as received condition.
2. Oxide thickness profile determination.
3. Atomic concentrations of oxide layer in the as received condition.

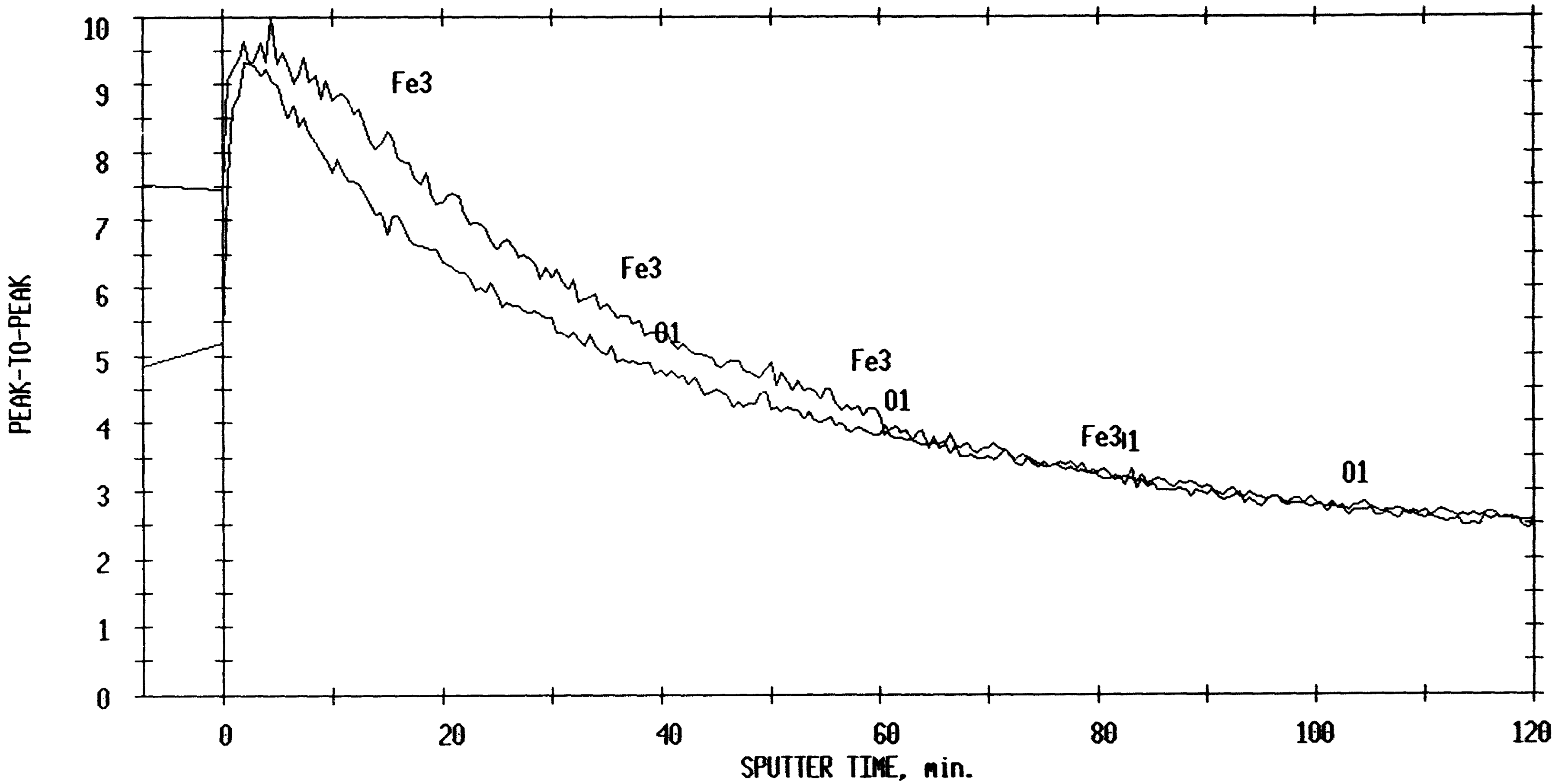
C. Sample XV Type 304L stainless steel exposed in air at 350° C for 30 days.

1. AES survey of kinetic energies of the oxide layer in the as received condition.
2. Oxide thickness profile determination.
3. Graphic view of oxide thickness profile.

AES PROFILE V/F ALT. 12/20/90 EL=Fe3 REG 2 AREA 1 SPUTTER TIME=120.00 MIN.

FILE: CPP131 CPP sample XU spp. at 3 kV, 10% raster, 30 deg. angle.

SCALE FACTOR= 174.106 k c/s, OFFSET= 0.000 k c/s   BV=5.00kV BI=0.1134uA



Atomic Concentration

a User Settings Previous New

b Element Name
d Massaged Yes No

c Area 1

e Regions C1 O1 Fe3 N1 S1 Cl1 Cr2 Al2 Cu1 Ni2 Ca1

Atomic Concentration Table

File: CPP130
Area: 1

CPP Sample XU as received.

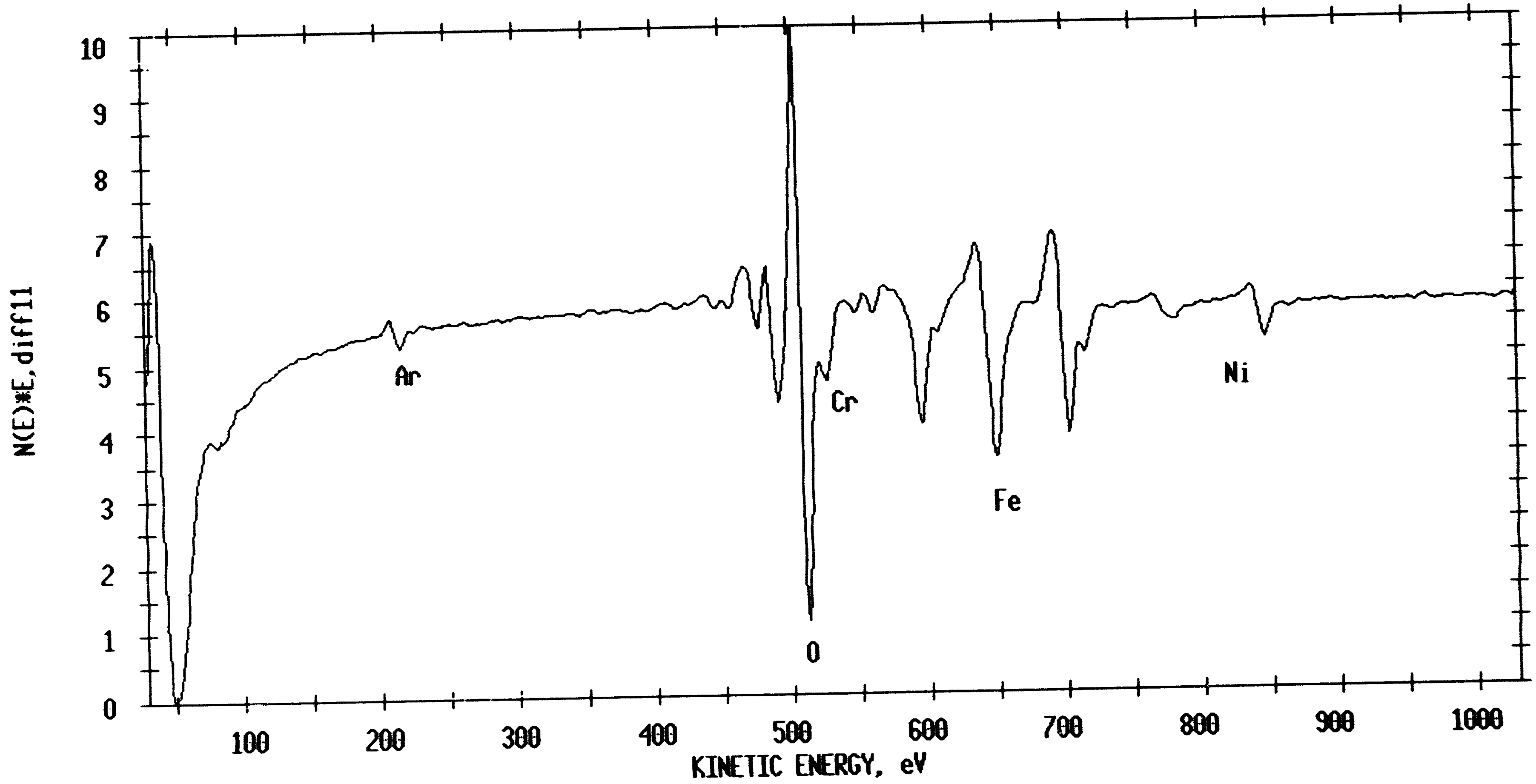
Element	Peak-to-peak	Sensitivity Factor	Concentration (%)
C1	6005450	0.140	32.77
O1	18569806	0.400	35.46
Fe3	4603761	0.220	15.99
N1	423583	0.230	1.41
S1	1477168	0.750	1.50
Al2	581731	0.070	6.35
Cu1	587509	0.230	1.95
Ni2	256561	0.107	1.83
Ca1	1434428	0.400	2.74

AES SURVEY P-C 12/21/90 AREA 1 ACQ TIME=11.26 MIN.

FILE: CPP132 CPP Sample after spp. for 200 min at 137 A/min.

SCALE FACTOR= 2051.075 k c/s, OFFSET= 0.000 k c/s

BV=5.00kV BI=0.1098uA



Atomic Concentration

a User Settings Previous New

c Area 1

b Element Name
d Massaged Yes No

e Regions C1 O1 Fe3 N1 S1 Cl1 Cr2 Ni2 Cal

Atomic Concentration Table

CPP Sample after spp. for 200 min at 137 A/min.

File: CPP132
Area: 1

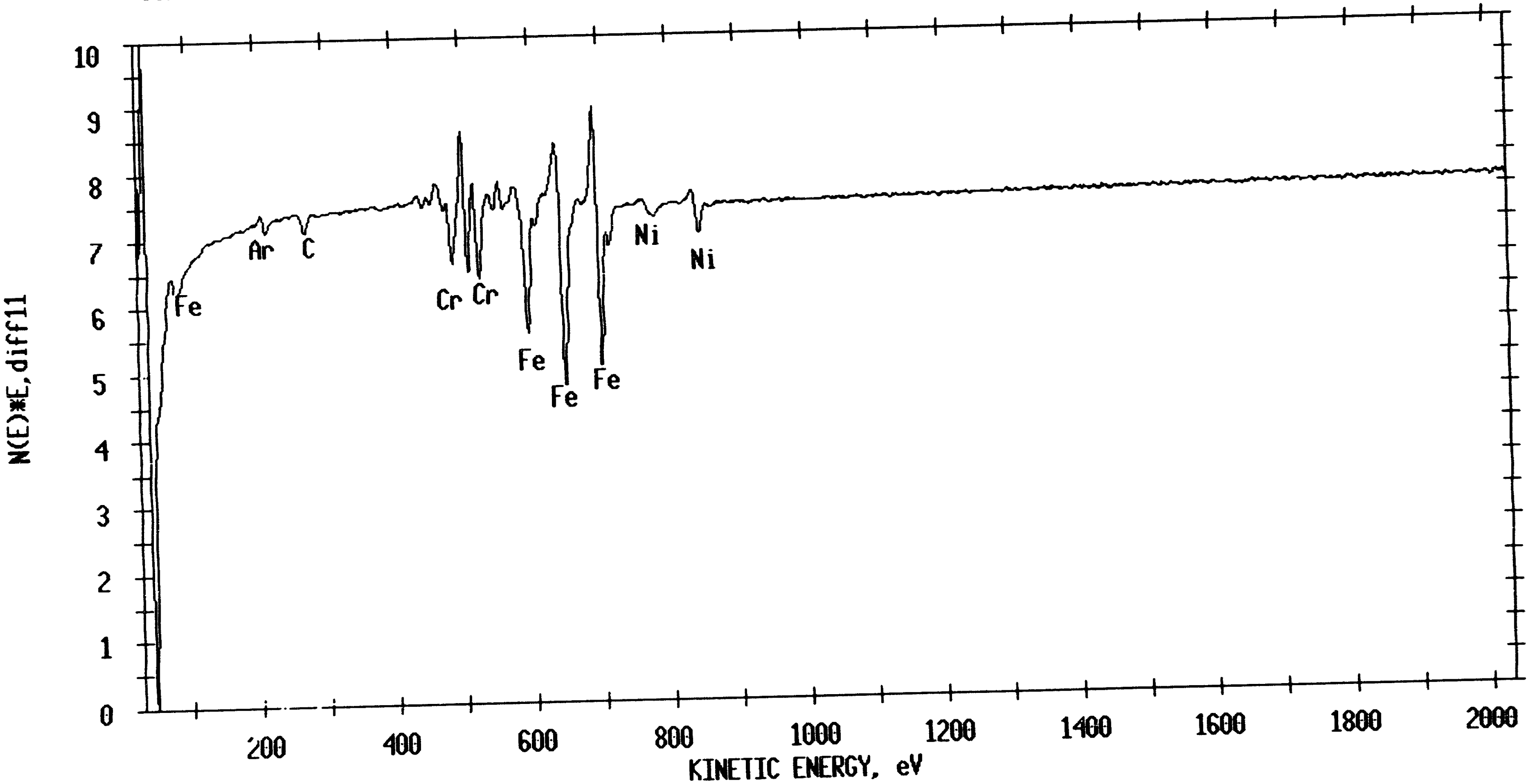
Element	Peak-to-peak	Sensitivity Factor	Concentration (%)
O1	629744512	0.400	61.60
Fe3	142875456	0.220	25.41
Cr2	65838724	0.310	8.31
Ni2	12806354	0.107	4.68

AES SURVEY V/F 12/18/90 AREA 1 ACQ TIME=22.51 MIN.

FILE: CPP129 CPP Sample XA after sputter profile.

SCALE FACTOR= 141.362 k c/s, OFFSET= 0.000 k c/s

BV=5.00kV BI=0.0967uA

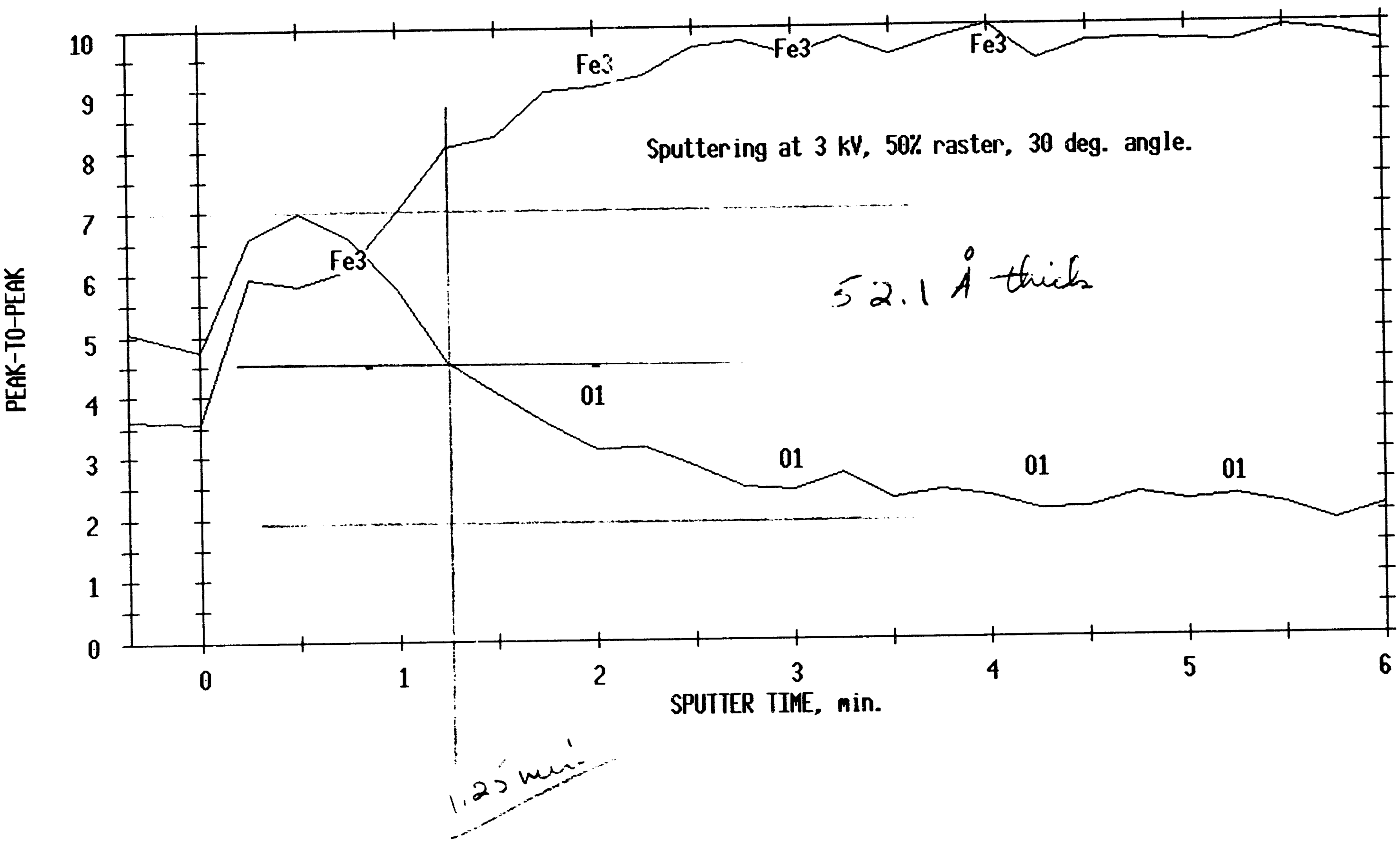


A-7

AES PROFILE V/F ALT.   12/18/90   EL=Fe3   REG 2 AREA 1   SPUTTER TIME=6.00 MIN.

FILE: CPP128   CPP Sample XA as received

SCALE FACTOR= 221.819 k c/s,   OFFSET= 0.000 k c/s   BV=5.00kV BI=0.0967uA



Atomic Concentration

a User Settings Previous New

b Element Name

c Area 1

d Massaged Yes No

e Regions C1 O1 Fe3 N1 S1 Cl1

Atomic Concentration Table

CPP Sample XA as received

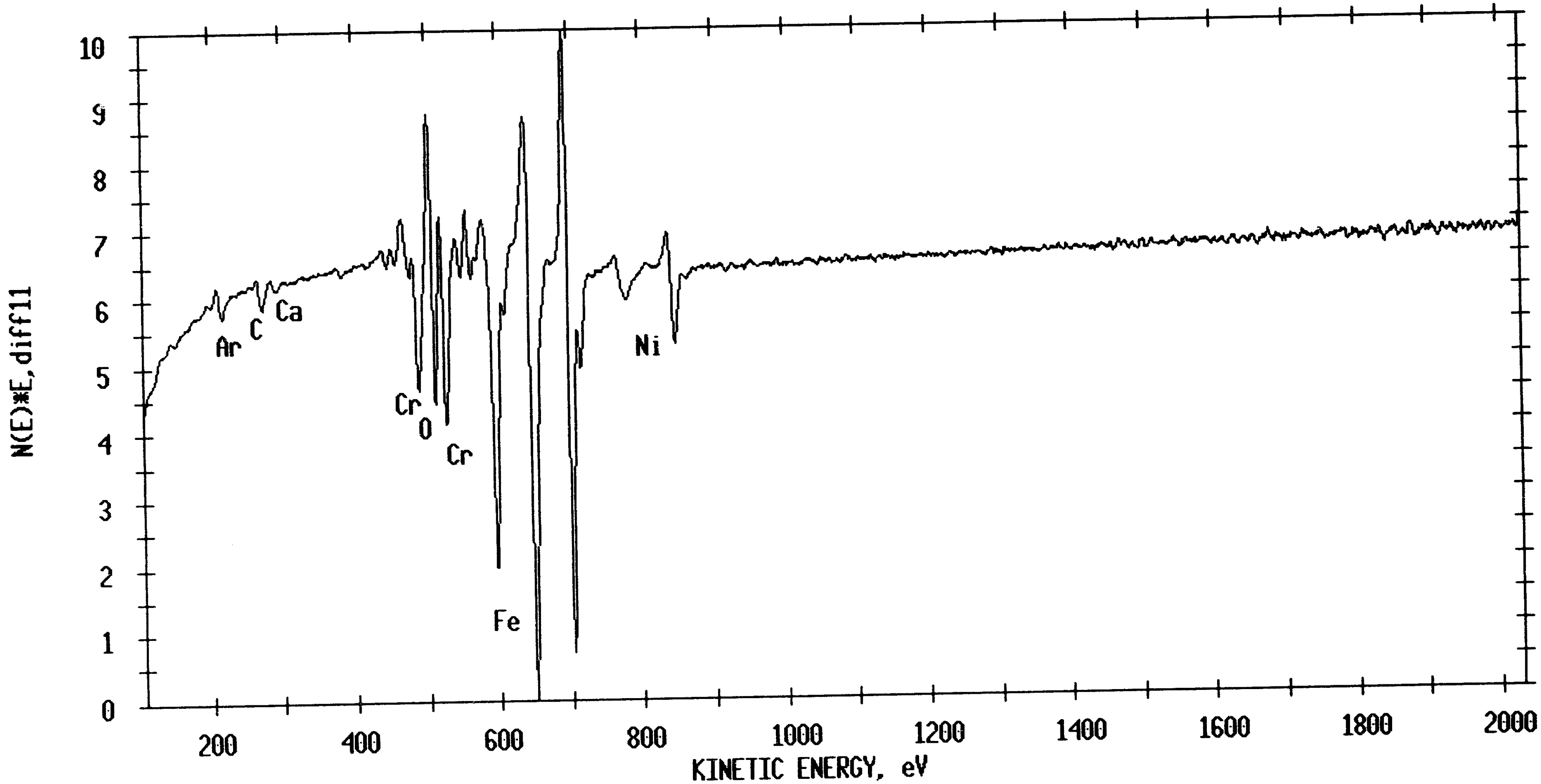
File: CPP127
Area: 1

Element	Peak-to-peak	Sensitivity Factor	Concentration (%)
C1	7938176	0.140	46.99
O1	15757495	0.400	32.65
Fe3	4301850	0.220	16.21
N1	787450	0.230	2.84
S1	657124	0.750	0.73
Cl1	744457	1.050	0.59

AES SURVEY V/F 1/25/91 AREA 1 ACQ TIME=22.51 MIN.

FILE: CPP142 CPP Sample XV after spp. 30 min.

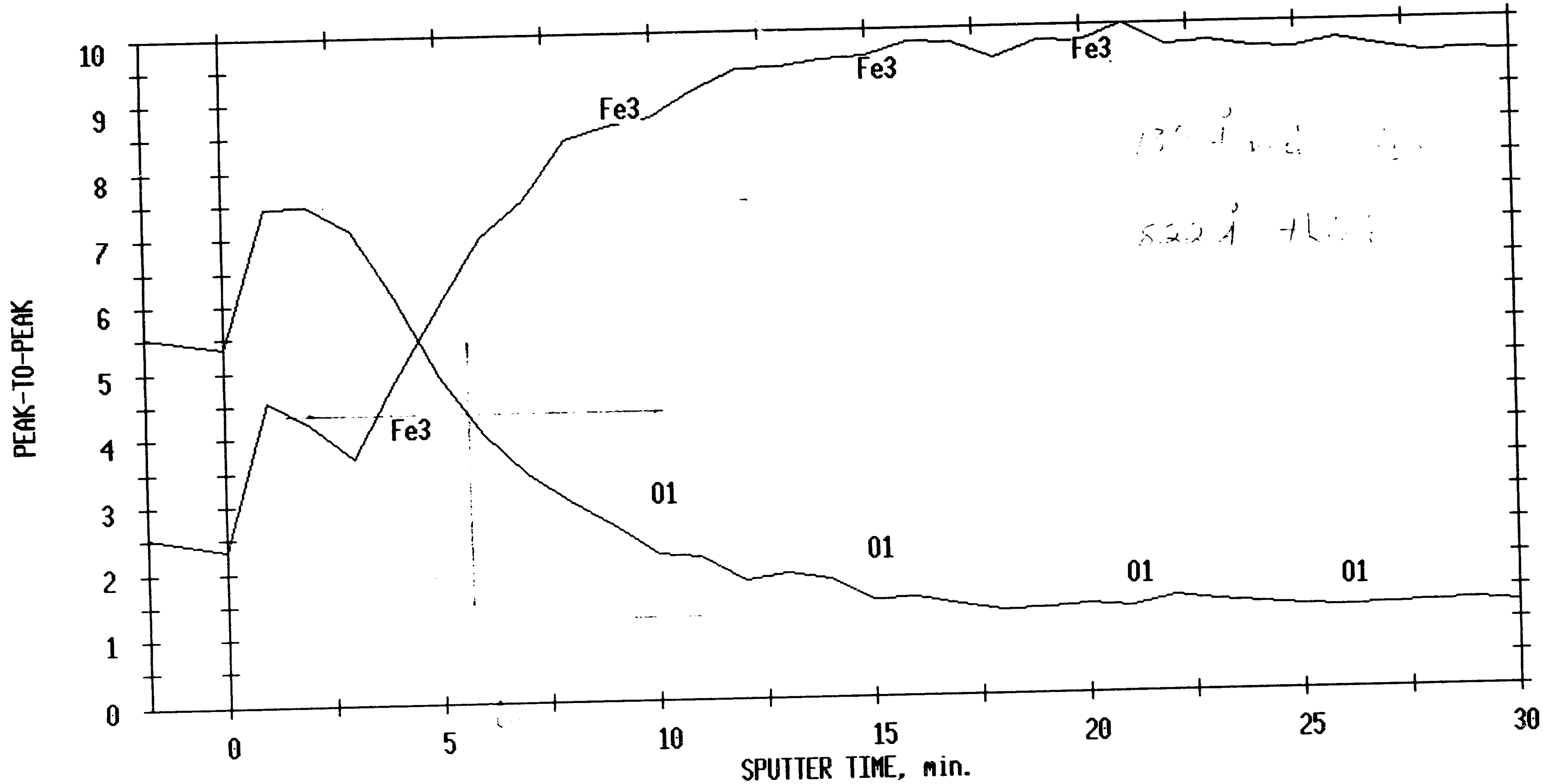
SCALE FACTOR= 64.292 k c/s, OFFSET= 940.230 k c/s BV=5.00kV BI=0.0787uA



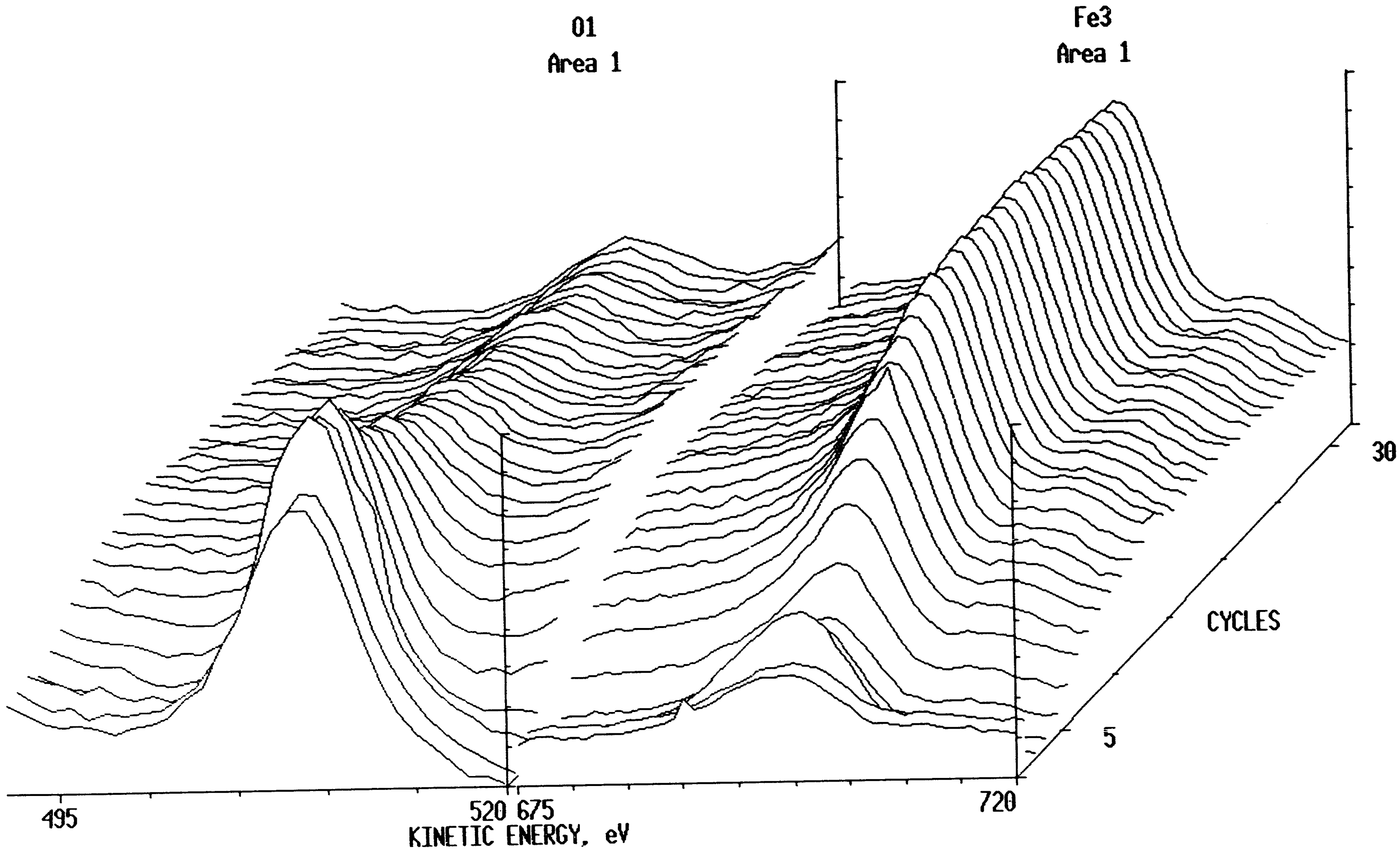
AES PROFILE P-C ALT.   1/25/91   EL=Fe3   REG 2 AREA 1   SPUTTER TIME=30.00 MIN.

FILE: CPP141 CPP Sample XV spp. at 3 kV, 10 % raster, 30 deg angle.

SCALE FACTOR= 1407.928 k c/s,   OFFSET= 0.000 k c/s   BV=5.00kV BI=0.0787uA



AES PROFILE   1/25/91 START=0, END=0, NTH=1
FILE: CPP141 CPP Sample XV spp. at 3 kV, 10 % raster, 30 deg angle.

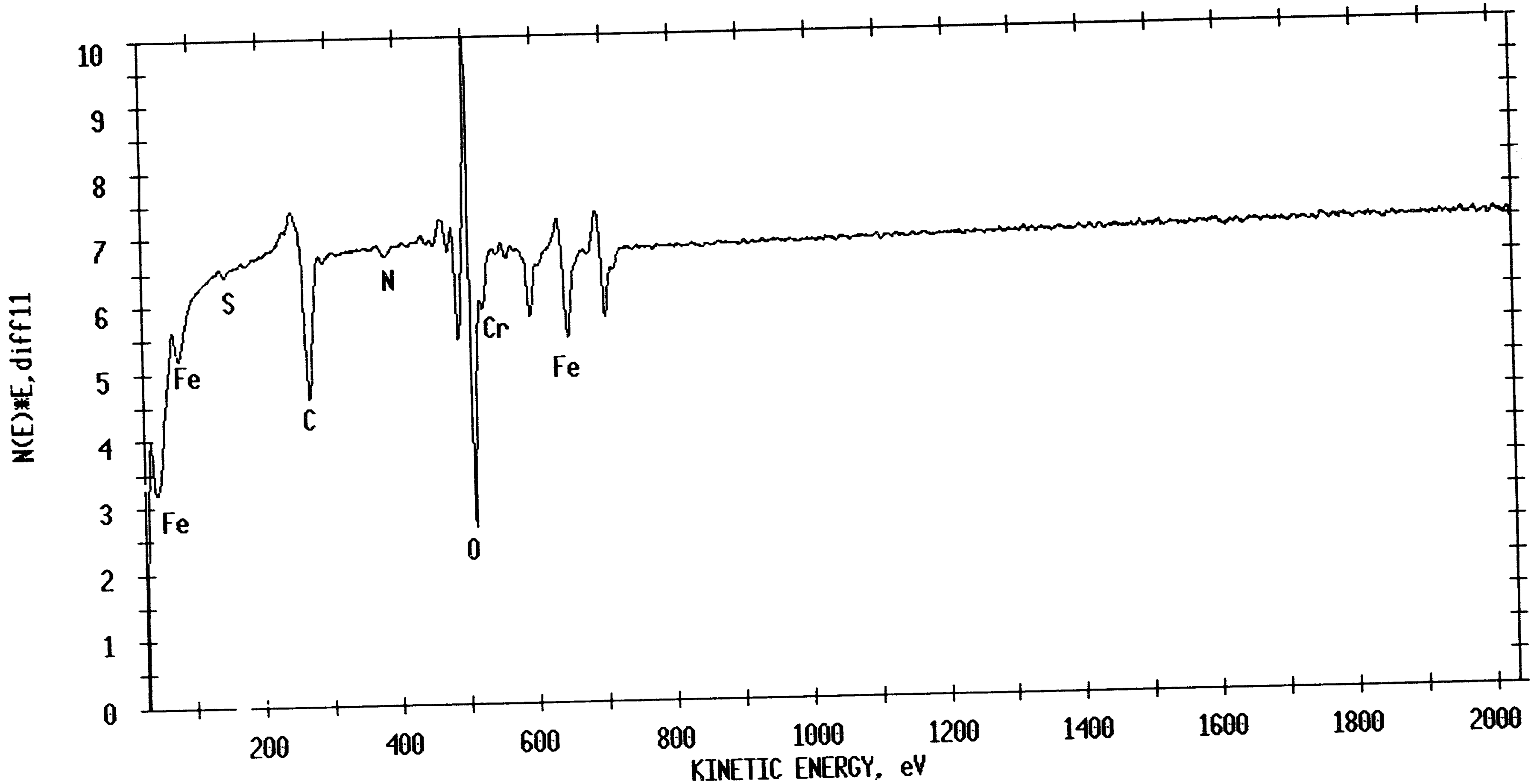


AES SURVEY V/F 1/25/91 AREA 1 ACQ TIME=22.51 MIN.

FILE: CPP140 CPP Sample XV as received.

SCALE FACTOR= 103.059 k c/s, OFFSET= 0.000 k c/s

BV=5.00kV BI=0.0779uA



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DATE FILMED

8/30/94

END