

ATTACHMENT 10

NETCO Report NET-332-01, Revision 1 "Inspection and Testing of BORAL[®] and Fast Start Surveillance Coupons from the LaSalle County Units 1 & 2 Stations"

**Inspection and Testing of BORAL® and
Fast Start Surveillance Coupons from the
LaSalle County Units 1 & 2 Stations**

September 2009

Prepared
for
Exelon Generation

under
Contract No.: 00447725

by
NETCO, a division of Scientech, a business unit
of Curtiss-Wright Flow Control Service Corporation
108 North Front Street - UPO Box 4178
Kingston, New York 12402

Review/Approval Record

Revision	Date	Prepared by:	Reviewed by:	Approved (QA):
0	9/10/2009	K. L. L. L.	[Signature]	L. P. Mariani
1	6/18/2012	K. L. L. L.	[Signature]	L. P. Mariani

Table of Contents

1.0	Introduction.....	1
2.0	Scope of Surveillance Coupon Testing.....	2
3.0	Test Results.....	5
3.1	Visual Inspection.....	5
3.2	Coupon Dimensions	13
3.3	Dry Weight	16
3.4	Specific Gravity and Density	18
3.5	Neutron Attenuation Measurements and B-10 Areal Density.....	20
3.6	Rockwell Hardness	21
3.7	Radioassay	21
3.8	Corrosion Rates of the Fast Start Coupons	22
4.0	Conclusions	23
5.0	References	24
Appendix A: Procedures for Measuring and Recording BORAL Surveillance Coupon Physical Attributes, SEP-235-01, Northeast Technology Corp.; 4/5/04		
Appendix B: Procedure for Post-Test Characterization of the Fast Start Surveillance Coupons, SEP-259-13, 6/30/09		
Appendix C: NIST Traceable Calibration Certificates for the Gage Blocks, Masses and Rockwell Hardness Tester		

1.0 Introduction

One BORAL coupon from Exelon's LaSalle County Unit 1 Station (LC1) and two ALCAN composite coupons from the Fast Start Surveillance program at Unit 2 were shipped to Sciencetech's laboratory facilities at the Pennsylvania State University. The BORAL coupon was identified as CL205700-2-3; and the Alcan Coupons were identified as No. 23 and 24. The coupons were subjected to testing and inspection as described in Section 2.0 of this report. The test results are contained in Section 3.0 and Sciencetech's conclusions with respect to test results are contained in Section 4.0. Appendix A contains a copy of SEP-235-01, "Procedures for Measuring and Recording BORAL Surveillance Coupon Physical Attributes," which was used as the test procedure for this work. Appendix B contains a copy of SEP-259-13, "Procedure for Post-Test Characterization of the Fast Start Surveillance Coupons," the procedure used to test the Alcan composite coupons. Appendix C contains copies of the NIST traceable calibration certifications for the gage blocks, the standard masses and the Rockwell Hardness Tester. The gage blocks and the standard masses were used to verify the dimension and weight measuring instruments.

All coupons were in good overall condition when received by Sciencetech. No significant deterioration or degradation was evident. Comparison with measurements made prior to placement in the pool (pre-irradiation) confirm this. No surface pitting was observed. Corrosion of the BORAL was limited to a more or less uniform, light oxide film on the coupon surfaces. No visible evidence of corrosion was noted on the surfaces of the Alcan coupons.

2.0 Scope of Surveillance Coupon Testing

The coupons received by Scientech were first subjected to visual inspection and photography followed by cleaning with demineralized water to remove transferable contamination. Weight measurements were then taken and the BORAL coupon was subjected to a four step drying process. After each drying step, the coupon was cooled and again weighed. After the drying sequence, the BORAL coupon was subjected to the following tests:

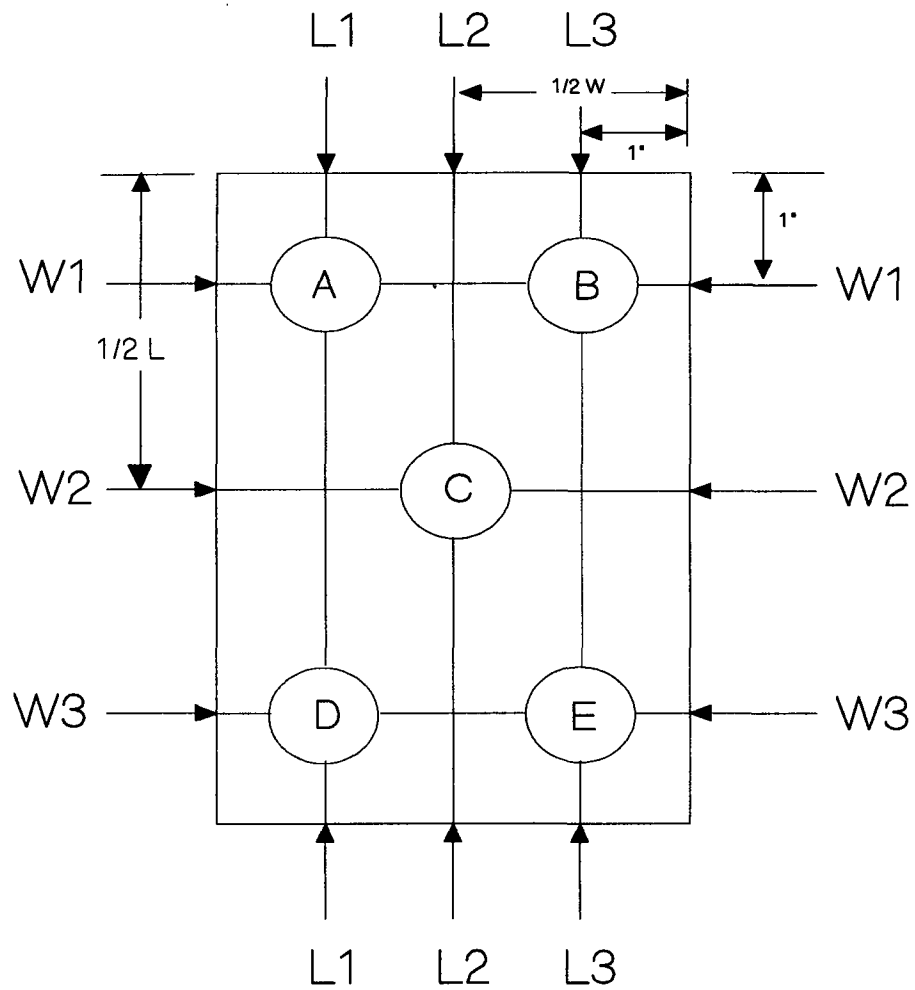
- Dimension measurements
- Specific gravity and density measurements
- Neutron attenuation testing and B-10 areal density measurements
- Rockwell E hardness measurements
- Radioassay measurements.

The locations for the dimension, attenuation and hardness tests are shown in Figure 2-1. All testing was conducted in accordance with SEP-235-01, which is included as Appendix A to this report.

The Alcan coupons were dried for 1 hour at 220° F to remove surface moisture. After drying, the coupons were subject to:

- Dimension measurements
- Specific gravity and density measurements
- Neutron attenuation testing and B-10 areal density measurements
- Acid cleaning and corrosion rate determination

The locations for the dimension measurements and neutron attenuation test are shown in Figure 2-2. All testing was conducted in accordance with SEP-259-13, which is included as Appendix B to this report.



Thickness, Neutron Attenuation and Hardness Testing at Locations A, B, C, D and E

Figure 2-1: Locations for the BORAL Coupon Measurements

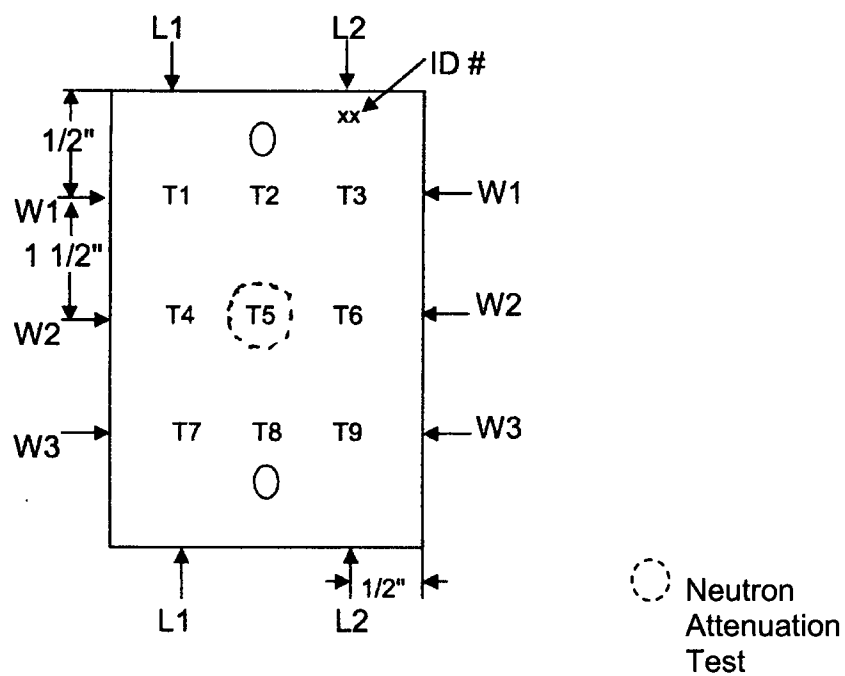


Figure 2-2: Locations for Fast Start Coupon Tests

3.0 Test Results

3.1 Visual Inspection

In this section, the condition of the front and back sides of the coupons are described. The front side is defined as the side of the coupon with the ID number on it.

BORAL Coupon CL205700-2-3

The notes from the visual inspection of Coupon CL205700-2-3 are contained in Table 3-1. The overall condition of the coupon was good with no anomalies other than a relatively high level of radioactive surface contamination. Figures 3-1 and 3-2 contain the microphotographs of the front and back sides of the capsule identified as No. 2. Figures 3-3 and 3-4 show the front and back sides of Coupon CL205700-2-3. During drying at 300 °F, six blisters developed on each side of the coupon. Figures 3-5 and 3-6 show the condition of the front and back sides of the coupon after drying.

Fast Start Coupons Nos. 23 and 24

The fast start coupons are unclad and are attached to each other by stainless links through the two holes in each coupon. The chain of coupons are attached to a head piece that fits into the upper lead-in of a storage cell in the pool. The notes from the visual inspection of the fast start coupons are contained in Table 3-2. The fast start coupons were in good condition. There were some minor surface scratches, which likely occurred when the coupon chain was removed from the pool. The front and back sides of coupon No. 23 are shown in Figures 3-7 and 3-8, respectively. The front and back sides of coupon No. 24 are shown in Figures 3-9 and 3-10. The surface appearance of both coupons was similar to that of mill finish aluminum with no visible surface oxidation after 6 months residency in the pool. When comparing the surfaces of coupons 23 and 24 with archive material, the coupons taken from the pool appear darker than the archive material as shown in Figure 3-11.

Table 3-1

Visual Inspection of Coupon CL205700-2-3

General: LAS is inscribed in the upper left corner. Just below is the AAR ID number CL205700-2-3. The coupon was received in a stainless steel capsule, which was opened by removing the twelve machine screws that secure the capsule cover plates. On the front side of the capsule the numerals 02 were inscribed. The coupon is in good overall condition and is intact. Contamination levels are high with Co-60 at about .21mCi and Fe-55 at 0.27mCi. After drying at 300 deg F six blisters developed on each side of this coupon.

Front side: Front surface is covered by a fairly heavy oxide film and the central portion of the film is a light grey. Along the side edges and bottom edge the oxide film is a darker grey. Within these regions there are areas where it looks like the oxide is thinner.

Back side: The central region is covered by a fairly heavy oxide film of a light grey color. The bottom and side edges have a darker color oxide film. Along the top edge there is a region where it looks like the oxide film is not formed. Along the top edge there are brown, rust-colored deposits, which could be the source of the high level of contamination. There is no evidence of pitting corrosion on either side of this coupon.

Table 3-2

Visual Inspection of Fast Start Coupons Nos. 23 and 24

General: The numerals F23 are inscribed in the upper right corner of the front side. The appearance of the surface of the coupon is similar to a mill finish. There is no visible oxide film on any of the surfaces. Inspection of the surface with a microscope confirms there is no oxide film.

Front side: There are random scratches on this side. There are no other anomalies.

Back side: The appearance is similar to the front.

General: The numerals F24 are inscribed in the upper right corner of the front side. The surfaces of the coupon appear as mill finish; that is, there are no visible signs of an oxide film. Inspection of the surface with a microscope confirms the absence of an oxide film.

Front side: There are various scratches, which most likely occurred when the coupons were removed from the pool. There are no anomalies on the front side. There appears to be some marking ink on the upper left edge from when the sheet was rolled.

Back side: Appearance of the back side is similar to the front.

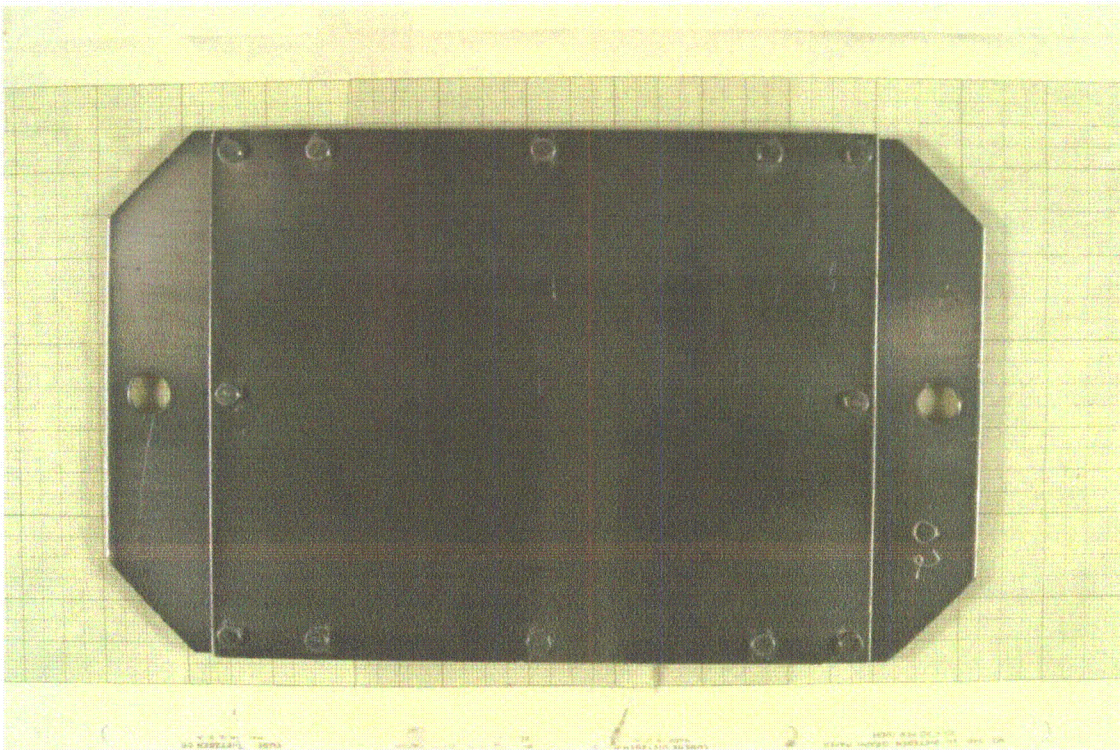


Figure 3-1: Capsule Designated as 02 As-Received: Front Side

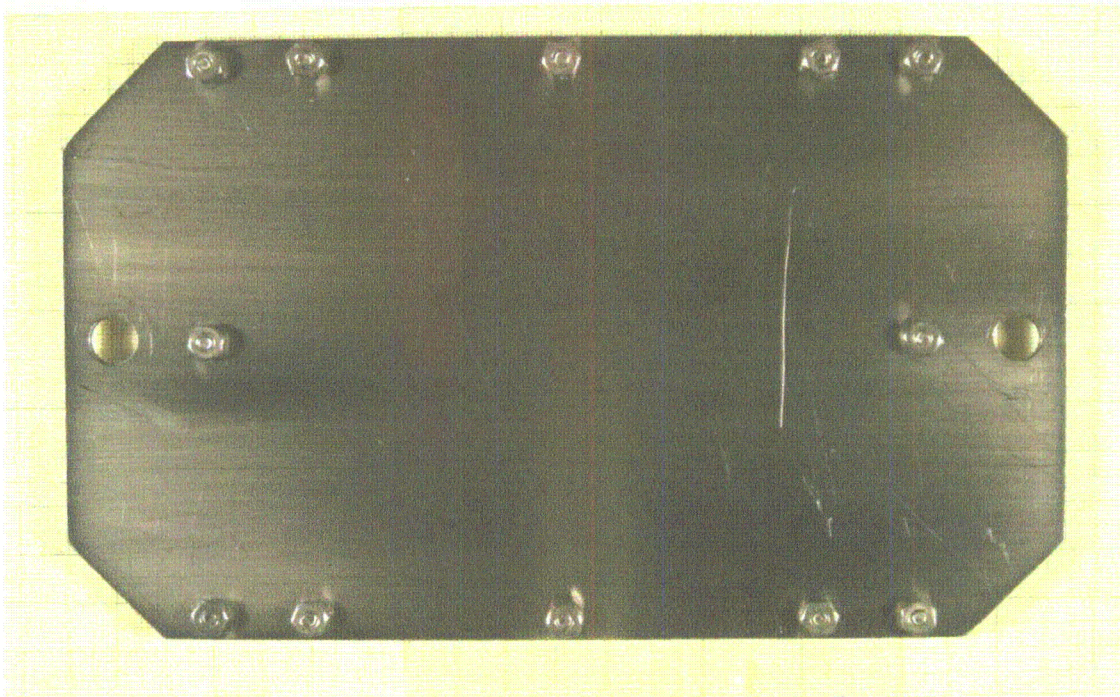


Figure 3-2: Capsule Designated as 02 As-Received: Back Side



Figure 3-3: Front Side of Coupon CL205700-2-3

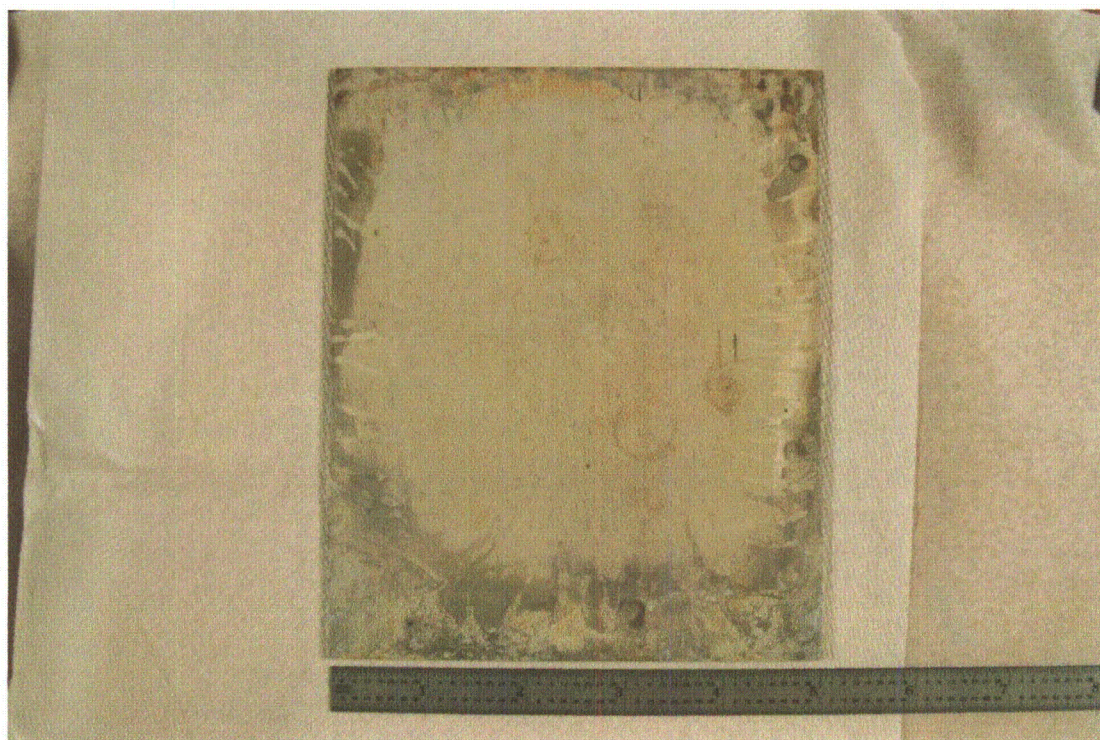


Figure 3-4: Back Side of Coupon CL205700-2-3



Figure 3-5: Front Side of Coupon CL205700-2-3: As Dried

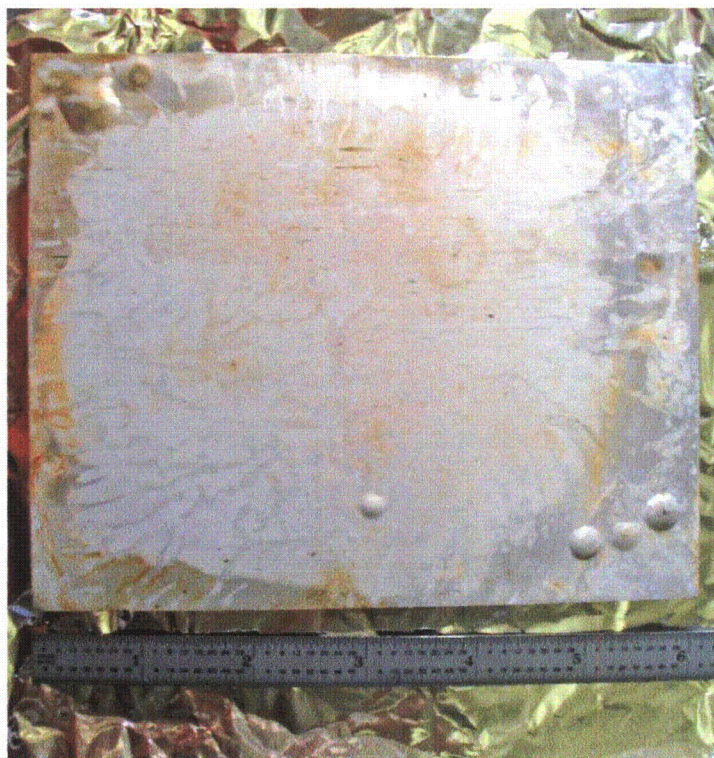


Figure 3-6: Back Side of Coupon CL205700-2-3: As Dried

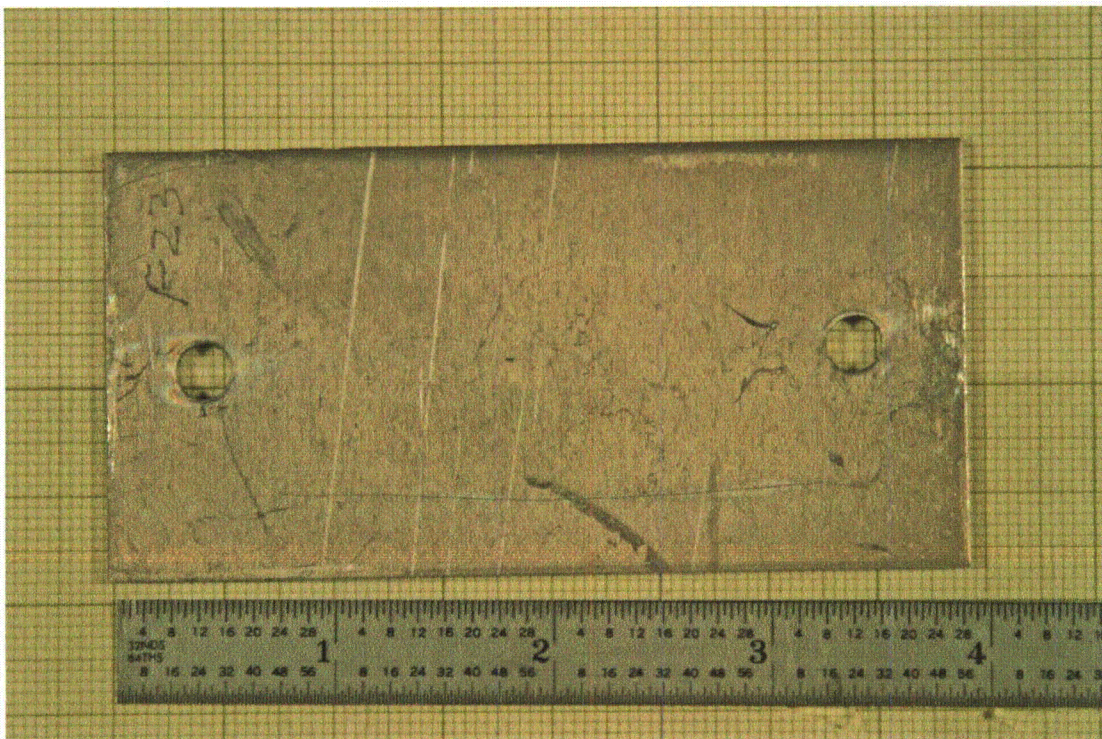


Figure 3-7: Fast Start Coupon 23 Front Side

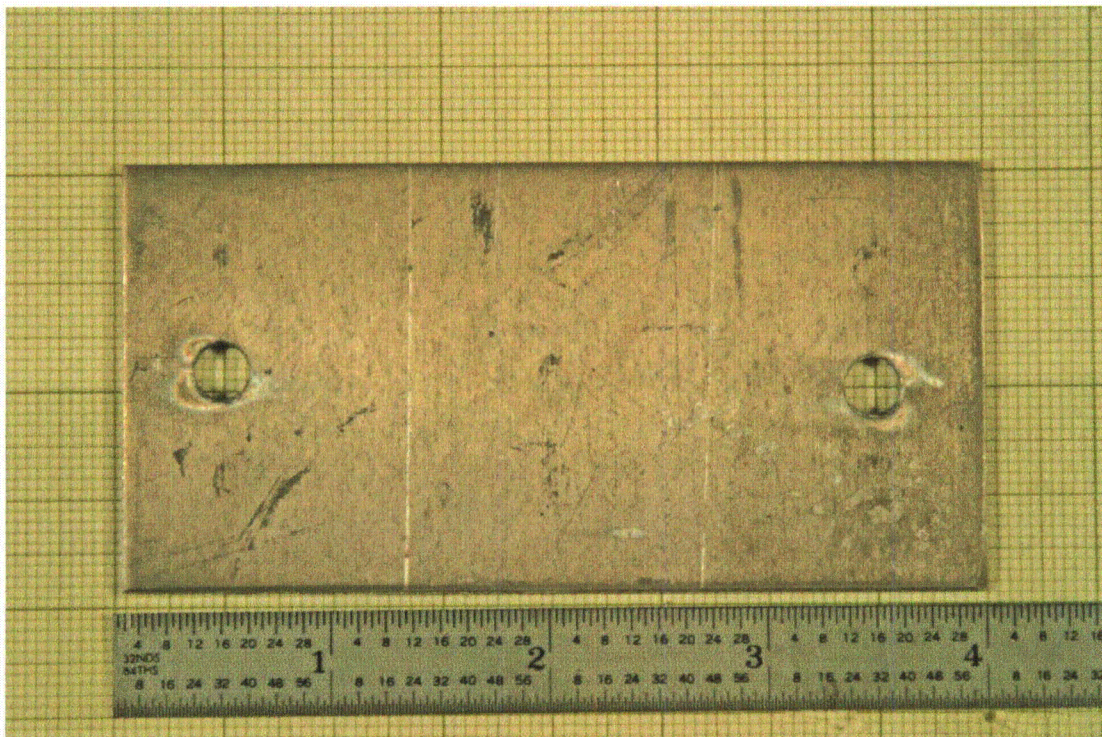


Figure 3-8: Fast Start Coupon 23 Back Side

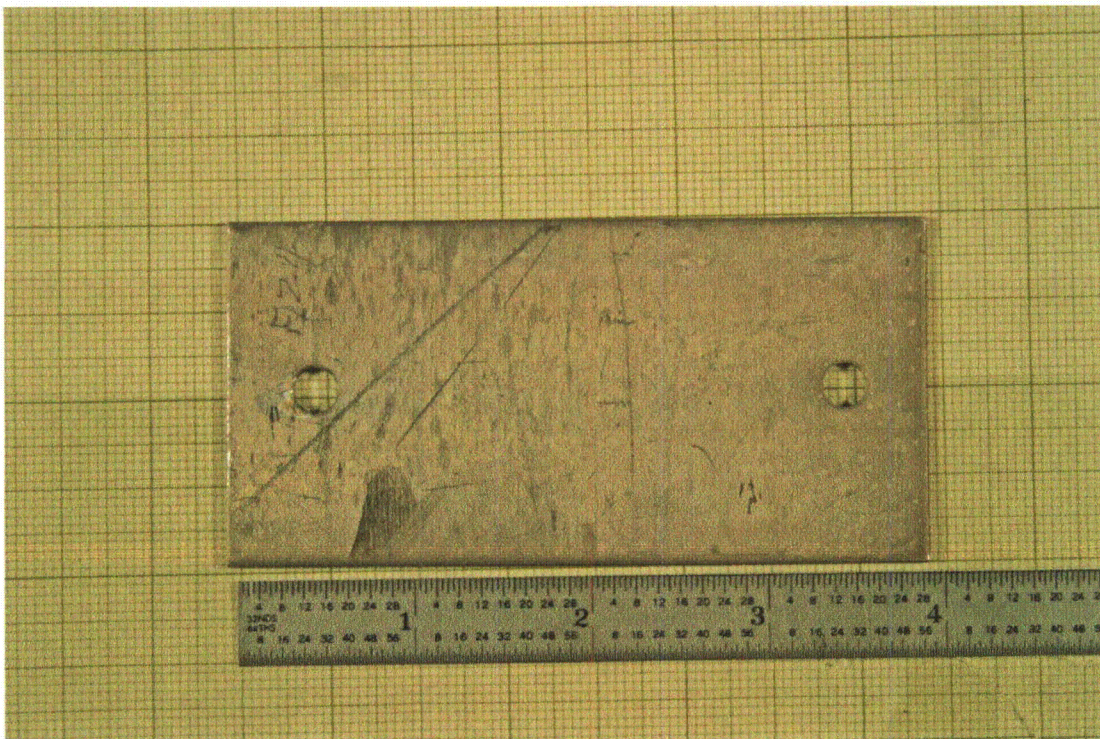


Figure 3-9: Fast Start Coupon 24 Front Side

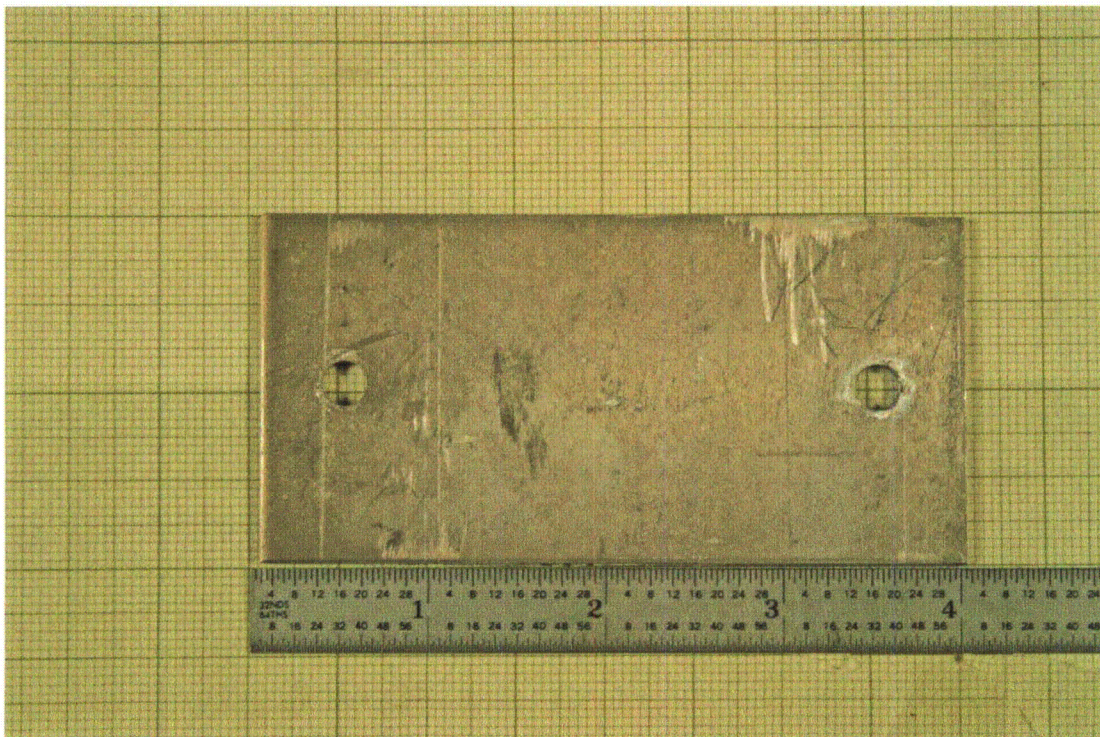


Figure 3-10: Fast Start Coupon 24 Back Side

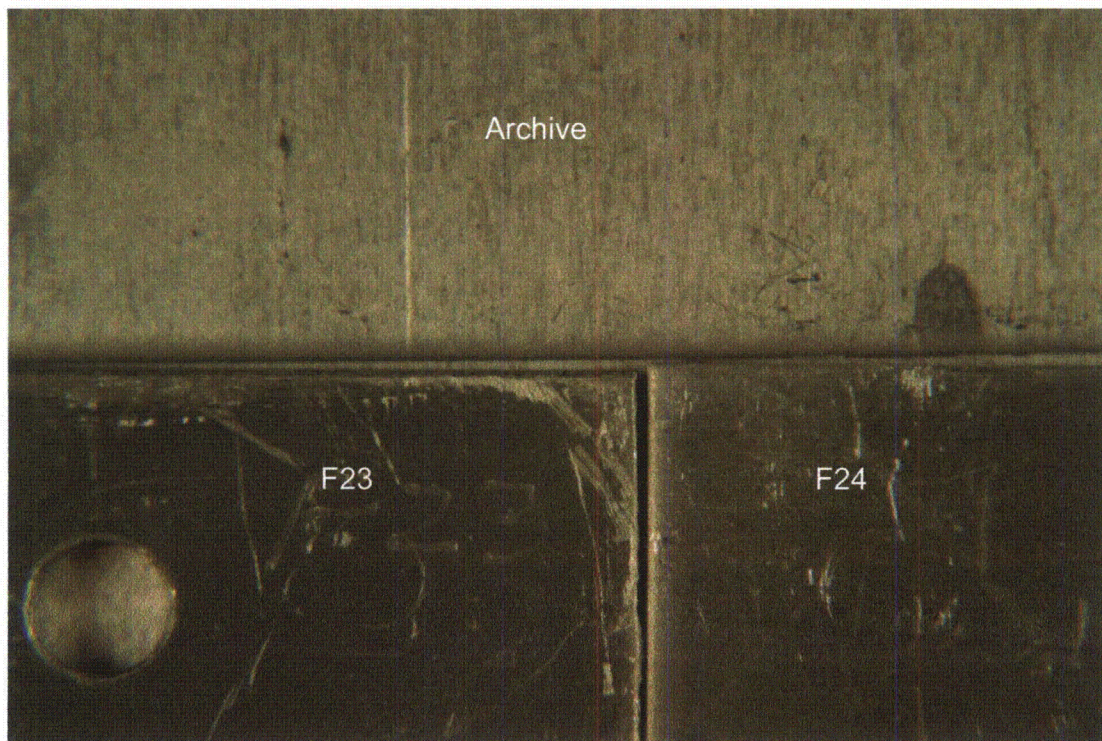


Figure 3-11: Microphotograph of Coupons 23, 24 and Archive Material
(8X Magnification)

3.2 Coupon Dimensions

The dimensions of the BORAL coupon were measured at the locations shown in Figure 2-1. The locations of the coupon dimension measurement for the fast start coupons are shown in Figure 2-2. The measured length, width and thickness of the BORAL coupon are summarized in Tables 3-3 and 3-4. The post irradiation data in these tables are compared with measurements taken when the coupon was prepared and pre-characterized. Comparison of the pre and post irradiation measurements indicate no change in dimensions.

The measured dimensions of the fast start coupons are summarized in Tables 3-5 and 3-6. The post irradiation measurements are compared with the measurements taken when the coupons were prepared and pre-characterized in these tables. Comparison of pre and post irradiation measurements shows no significant change in coupon dimensions.

Table 3-3

Length and Width Measurements of Coupon CL205700-2-3

Coupon ID No.		Length, inches				Width, inches			
		Length 1	Length 2	Length 3	L-avg	Width 1	Width 2	Width 3	W-avg
CL205700-2-3	As Measured	6.084	6.084	6.083	6.084	5.010	5.016	5.016	5.014
		As Manufactured Length				As Manufactured Width			
		6.083	6.084	6.078	6.082	5.02	5.016	5.016	5.017
		Change In Length				Change in Width			
		0.001	0.000	0.005	0.002	-0.010	0.000	0.000	-0.003
		Percent Change in Length				Percent Change in Width			
		0.02	0.01	0.08	0.04	-0.19	0.01	0.01	-0.06

Table 3-4

Thickness Measurements of Coupon CL205700-2-3

Coupon ID No.	Thickness, Inches					
	Thickness-A	Thickness-B	Thickness-C	Thickness-D	Thickness-E	T-Avg
CL205700-2-3	0.0799	0.0800	0.0785	0.0790	0.0788	0.0792
	As Manufactured Thickness					
	0.0800	0.0800	0.0780	0.0790	0.0790	0.0792
	Change In Thickness					
	-0.0001	0.0000	0.0005	0.0000	-0.0002	0.0000
	Percent Change In Thickness					
	-0.19	0.06	0.58	0.06	-0.25	0.05

Table 3-5
Length and Width Measurements of Fast Start Coupons 23 and 24

Coupon ID		Length-1	Length-2	Width-1	Width-2	Width-3
F23	Pre-Irradiation	3.953	3.975	1.975	1.984	1.982
	Post-Irradiation	3.953	3.974	1.974	1.983	1.982
	Change Δ	0.000	-0.001	-0.001	-0.001	0.000
	Change %	-0.01%	-0.03%	-0.03%	-0.05%	0.03%
F24	Pre-Irradiation	3.977	3.975	1.975	1.982	1.977
	Post-Irradiation	3.977	3.974	1.974	1.992	1.977
	Change Δ	0.001	0.000	-0.001	0.010	0.000
	Change %	0.03%	-0.01%	-0.03%	0.53%	-0.03%

Table 3-6
Thickness Measurements of Fast Start Coupons 23 and 24

Coupon ID			Thickness-1	Thickness-2	Thickness-3	Thickness-4	Thickness-5	Thickness-6	Thickness-7	Thickness-8	Thickness-9	Average
F23	Pre-Irradiation		0.0670	0.0670	0.0670	0.0670	0.0670	0.0670	0.0670	0.0670	0.0670	0.0670
	Post-Irradiation, As Dried		0.0658	0.0695	0.0663	0.0663	0.0667	0.0670	0.0661	0.0686	0.0669	0.0667
		Change Δ	-0.0011	0.0025	-0.0006	-0.0006	-0.0003	0.0000	-0.0009	0.0016	-0.0001	-0.0003
		Change %	-1.68%	3.77%	-0.93%	-0.93%	-0.48%	0.04%	-1.38%	2.43%	-0.11%	-0.38%
	Post-Irradiation, After Acid Wash		0.0675	0.0680	0.0670	0.0671	0.0670	0.0665	0.0664	0.0663	0.0664	0.0669
		Change Δ	0.0005	0.0010	0.0000	0.0002	0.0000	-0.0005	-0.0006	-0.0007	-0.0006	-0.0001
		Change %	0.79%	1.53%	-0.04%	0.26%	-0.04%	-0.71%	-0.86%	-1.01%	-0.86%	-0.10%
F24	Pre-Irradiation		0.0670	0.0670	0.0670	0.0670	0.0670	0.0670	0.0670	0.0670	0.0670	0.0670
	Post-Irradiation, As Dried		0.0666	0.0688	0.0668	0.0670	0.0668	0.0675	0.0662	0.0679	0.0668	0.0672
		Change Δ	-0.0004	0.0018	-0.0001	0.0000	-0.0001	0.0005	-0.0007	0.0009	-0.0001	0.0002
		Change %	-0.63%	2.73%	-0.19%	0.04%	-0.19%	0.71%	-1.08%	1.31%	-0.19%	0.28%
	Post-Irradiation, After Acid Wash		0.0667	0.0693	0.0667	0.0667	0.0674	0.0671	0.0672	0.0674	0.0673	0.0673
		Change Δ	-0.0003	0.0023	-0.0003	-0.0002	0.0004	0.0001	0.0002	0.0004	0.0003	0.0003
		Change %	-0.48%	3.40%	-0.41%	-0.33%	0.56%	0.11%	0.34%	0.64%	0.49%	0.48%

Note: The value of pre-irradiation thickness (0.0670") for coupons F23 and F24 was determined based on the average value of eighty-one thickness measurements made on archive coupons taken from the same sheet of rolled ALCAN material.

3.3 Dry Weight

The as-received dry weight of the BORAL coupon is given in Table 3-7. Also given in this table is the weight after each 4 hour drying step, and the change in weight as a result of each drying step. The purpose of the 4-step drying process is to remove moisture entrained in porosity in the core of the BORAL. The coupon showed a gain in weight of ~ 0.92%, relative to the coupon's as-manufactured weight. This is likely due to the presence of an oxide film on most of the coupon surfaces.

The dried weights of fast start coupons 23 and 24 are given in Table 3-8. Since the fast start coupons are fully dense (negligible porosity), they are dried for 1 hour at 220° F to remove surface moisture. The dried coupon weights are compared with the weight measurements made when the coupons were prepared and pre-characterized in Table 3-8. This shows there has been an insignificant change in coupon weight.

Also, shown in Table 3-8 are the weight losses after two cleanings with nitric acid. There was no further weight loss after the second acid wash and it is concluded that any corrosion products present were removed by the first wash. The data in this table show there is very little weight change due to acid cleaning (-0.01%) for each coupon. This confirms the visual and microscopic inspections, which concluded there were little or no corrosion products on the coupons.

Table 3-7
Weight Measurements of Coupon CL205700-2-3

Coupon ID No.		Weight, Grams					
		As	After Four Hours Of Drying At				As
		Opened	Specified Temperature				Manufactured
			175° F	300° F	500° F	500° F	
CL205700-2-3	Weight, grams	98.63	98.55	98.26	97.89	97.87	96.98
	Step Change, gms		0.08	0.29	0.37	0.02	
	Step Change, %		0.08%	0.29%	0.38%	0.02%	

Table 3-8
Weight Measurements of Fast Start Coupons 23 and 24

Coupon ID		Dry Weight (Grams)
F23	Pre-Irradiation	22.6870
	Post-Irradiation, As Dried	22.6800
		Change Δ
		Change %
	Post-Irradiation, After Acid Wash	22.6770
		Change Δ
		Change %
F24	Pre-Irradiation	22.8060
	Post-Irradiation, As Dried	22.8000
		Change Δ
		Change %
	Post-Irradiation, After Acid Wash	22.7960
		Change Δ
		Change %

3.4 Specific Gravity and Density

The coupons were also subjected to specific gravity and density measurements via immersion weighing. The results of the measurements for the BORAL coupon are summarized in Table 3-9. The pre-irradiation density of the coupon was reported⁽¹⁾ as 2.49 gms/cm³. The measured post irradiation coupon density is 2.53 gms/cm³. The difference between the pre and post irradiation measurements is not significant.

The results of the density measurements for the fast start coupons are summarized in Table 3-10. Comparison of the densities measured when the coupons were prepared and pre-characterized with the post irradiation values indicates essentially no change in coupon densities.

Table 3-9

Specific Gravity and Density Measurements of Coupon CL205700-2-3

Coupon ID No.	Dry Weight, gms	Immersed Weight, gms	Specific Gravity	Density g/cm ³	Coupon ID No.	Dry Weight, gms	Immersed Weight, gms	Specific Gravity	Density g/cm ³
CL205700-2-3	97.89	59.25	2.53	2.53	As Built	96.98	58.12	2.50	2.49
Water Properties					Water Properties				
Temperature	23 deg C				Temperature	21 deg C			
Density	0.997567 g/cm ³				Density	0.99802 g/cm ³			

Table 3-10

Specific Gravity and Density Measurements of Fast Start Coupons 23 and 24

Coupon ID		Immersed Weight, (Grams)	Specific Gravity gm/cm ³	Water Temp (° C)	Density gm/cm ³
F23	Pre-Irradiation	14.2700	2.70	19.0	2.69
	Post-Irradiation	14.2800	2.70	19.0	2.70
		Change Δ	0.0100	0.005	0.0046
		Change %	0.07%	0.17%	0.17%
F24	Pre-Irradiation	14.3500	2.70	19.0	2.69
	Post-Irradiation	14.3700	2.70	19.0	2.70
		Change Δ	0.0200	0.0076	0.0076
		Change %	0.14%	0.28%	0.28%

3.5 Neutron Attenuation Measurements and B-10 Areal Density

The Boron-10 areal density of the coupon was measured via neutron attenuation testing. The test was conducted in the Beam Hole Laboratory at the Pennsylvania State University using the PSU Triga Reactor as a source of thermal neutrons. Table 3-11 contains the results of the B-10 areal density measurements.

The Boron-10 areal density was measured at five locations on the BORAL coupon as shown in Figure 2-1. The average areal density for the coupon is 0.0256 ± 0.0003 grams B-10/cm². The pre-irradiation areal density for coupon CL205700-2-3 is reported as 0.0250 gms B-10/cm². There is no difference between the pre and post-irradiation values.

The Boron-10 areal density of the fast start coupons was measured at the geometric center of each fast start coupon. The areal densities of coupons 23 and 24 are 0.0093 ± 0.0003 and 0.0095 ± 0.0003 grams B-10/cm², respectively. During pre-characterization measurements, the areal density for coupons 23 and 24 were each determined to be 0.0092 ± 0.0006 grams B-10/cm². Accordingly, it is concluded there has been no change in areal density.

Table 3-11
Neutron Attenuation

Measurement ID	Attenuated Beam, counts	Count Time, s	Power Beam, counts	Count Time, s	Unattenuated Beam, cps	Bkgrd, cps	Attenuated Beam, cps	Power Beam, cps	Areal Density	Uncertainty
CL205700-2-3:A	37434	30	20321	30	33899	74	1248	677	0.0253	0.0003
CL205700-2-3:D	38952	30	20146	30	33899	74	1298	672	0.0248	0.0003
CL205700-2-3:B	37911	30	20231	30	33899	74	1264	674	0.0251	0.0003
CL205700-2-3:E	38316	30	20022	30	33899	74	1277	667	0.0249	0.0003
CL205700-2-3:C	39039	30	20128	30	33899	74	1301	671	0.0248	0.0003
F23	270841	30	20022	30	33899	74	9028	667	0.0093	0.0003
F24	267310	30	20152	30	33899	74	8910	672	0.0095	0.0003

3.6 Rockwell E Hardness

The Rockwell E Hardness was measured at five locations on the BORAL coupon as shown in Figure 2-1. The measured values are summarized in Table 3-12. The RHE for as-manufactured BORAL is typically 50 to 60 RHE depending on the boron carbide loading. All measured values are within this range indicating acceptable material performance.

Table 3-12
Rockwell E Hardness

Location	RHE
A	52.0
B	55.0
C	55.0
D	54.0
E	55.0
Average	54.2

3.7 Radioassay

The BORAL coupon was subjected to radioassay along the top edge, center and bottom edge. The measurements are recorded in Table 3-13. The relatively high activity on the top edge is likely due to crud which has settled out from the pool water. The measurements indicate some water permeation along the porous bottom edge and radioactive pool contamination at that location. The lower level of activity at the center of the coupon is likely due to residual surface contamination.

Table 3-13
Radioassay

Beta & Gamma, cpm			Gamma, cpm			Beta, cpm		
Top	Center	Bottom	Top	Center	Bottom	Top	Center	Bottom
2800	400	1950	2000	300	1550	800	100	400

3.8 Corrosion Rates of the Fast Start Coupons

The corrosion rates of Coupons 23 and 24 were calculated based on ASTM-G31-72 (Reapproved 2004) and the weight loss data in Table 3-8 that resulted from acid cleaning. The corrosion rates so calculated are 0.03 mils/year for both Coupons 23 and 24. This corrosion rate is extremely low and confirms the visual observation that both coupons were essentially devoid of corrosion products. The measured corrosion rates are consistent with the corrosion rates as determined from accelerated corrosion testing at 195° F.⁽²⁾

1

4.0 Conclusions

BORAL surveillance coupon CL205700-2-3 from the LaSalle Unit 1 spent fuel pool was subjected to non-destructive testing. The tests included measurement of dimensions, dry weight, density, boron-10 areal density, Rockwell E hardness and radioassay. The results of these tests are indicative of satisfactory material performance. Comparison of the post-irradiation areal densities with pre-irradiation values further confirms acceptable coupon performance and no loss of boron carbide.

The coupon showed a weight gain of ~ 1.0% relative to the pre-irradiation weight. This is likely due to a light oxide film which had formed on the coupon surfaces during residency in the spent fuel pool. A small increase in the coupon's dimension tends to confirm this conclusion. The development of small blisters in the clad of the BORAL during drying is inconsequential.

The two fast start coupons, Nos. 23 and 24, from the LaSalle 2 pool exhibited excellent dimensional stability, insignificant weight or density change, and no change in boron-10 areal density. Corrosion rates were determined per ASTM-G31-72 (Reapproved in 2004). Any corrosion products present were removed by successive washes in nitric acid and weight changes measured. The corrosion rate for each coupon so determined was 0.03 mils/year, which is very low and consistent with the rates measured under accelerated corrosion conditions.⁽²⁾

5.0 References

- 1) Certificate of Compliance and Data Sheets for LaSalle BORAL Surveillance Coupon, Holtec Project Number 11240, 6/25/93.
- 2) "Material Qualification of Alcan Composite for Spent Fuel Storage," NET-259-03, Rev 5, Northeast Technology Corp., 7/30/09.

Appendix A

SEP-235-01

Procedure for Measuring and Recording BORAL Surveillance
Coupon Physical Attributes

NORTHEAST TECHNOLOGY CORP.

SPECIAL ENGINEERING PROCEDURE NO. SEP 235-01

**PROCEDURES FOR MEASURING AND RECORDING BORAL
SURVEILLANCE COUPON PHYSICAL ATTRIBUTES**

Prepared for:

Exelon Generation Co., LLC.

LaSalle County Station

Revision/Approval Record

Revision	Date	Prepared by:	Reviewed by:	Approved (QA):
0	4/5/04	K. Longest	M. Harris	L.P. Maurini

Table of Contents

1.0	Scope	1
2.0	Applicability.....	1
3.0	Related Documents	1
4.0	Required Test Equipment and Material	2
5.0	Coupon Description	3
6.0	Procedure for Non-Destructive Testing of Boral Coupons.....	3
6.2	Procedure for Visual Inspection/Permanent Photo Record.....	3
6.2	Procedure for Coupon Drying	3
6.3	Procedure for Coupon Thickness Measurements.....	4
6.4	Procedure for Measuring Specific Gravity and Density.....	4
6.5	Procedure for Neutron Attenuation Testing (Performed in the Beam Hole Laboratory of the Pennsylvania State University Triga Reactor Facility).....	5
6.6	Procedure for Rockwell Hardness Test.....	6
6.7	Procedure to Calibrate hardness Tester Using NIST Traceable Test Blocks	6
6.8	Procedure for Coupon Radioassay	7
6.9	Procedure for Surface Pit Characterization.....	7
7.0	Control of Material	8

SPECIAL ENGINEERING PROCEDURE SEP-235-01

PROCEDURE FOR MEASURING AND RECORDING
BORAL SURVEILLANCE COUPON PHYSICAL ATTRIBUTES1.0 SCOPE:

This procedure specifies methods for measuring and recording the physical attributes of Boral coupons in the post-irradiation condition that will be performed at Penn State University, University Park, PA by NETCO.

2.0 APPLICABILITY:

This procedure applies to the characterization measurements performed on BORAL surveillance coupons from the LaSalle County Station.

3.0 RELATED DOCUMENTS:

- Procedure for Boral Surveillance Samples, BPS 454 Rev. 1, 10/12/82, Brooks and Perkins, Inc.

ASTM Standards:

- C992 Specification for Boron-Based Neutron Absorbing Material Systems for Use in Nuclear Spent Fuel Storage Racks.
- E6 Definitions of Terms Relating to Methods of Mechanical Testing.
- G1 Recommended Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens.
- G4 Guide for Conducting Corrosion Coupon Tests in Plant Equipment.
- G15 Definitions of Terms Relating to Corrosion and Corrosion Testing.
- G16 Recommended Practice for Applying Statistics to Analysis of Corrosion Data.
- G46 Recommended Practice for Examination and Evaluation of Pitting Corrosion.

- G69 Practice for Measurement of Corrosion Potentials of Aluminum Alloys.
- ASTM Proposed Standard Guide for Establishing Surveillance Test Progress for Boron Based Neutron Absorbing Materials for Use in Nuclear Fuel Storage Racks.

4.0 REQUIRED TEST EQUIPMENT AND MATERIAL:

Test equipment required:

1. Precision Micrometer with minimum of 3½" throat (Starrett Model No. 222BR-1 or equivalent).
2. Vernier Caliper with range 0-8", .001" graduation (Mitutoyo Model No. 531-144 or equivalent).
3. Gage blocks, steel, Weber Grade 2, .050" to 12.00" with Certification (NIST Traceable).
4. Balance (Ohaus TS400D or equivalent).
5. Standard Masses Calibrated and Certified (NIST Traceable).
6. High Resolution 35mm Camera (Minolta XJ7 or equivalent).
7. Thermometer, range 0 to 110°C (1°C graduation), (Scientific Products Model T2050-1 or equivalent).
8. Geiger Mueller Detector
9. Desiccator
10. Metallurgical Microscope with 50x to 500x magnification range and fine focus calibration knob.
11. Rockwell Hardness Tester with indenter ball for E scale.

To assure that tools, gages, instruments, and other measuring and test equipment for the project are properly controlled, calibrated and adjusted at specified periods to maintain the required accuracy the following shall apply:

The calibration standards listed above (#3 and #5) shall have certification and/or

a sticker indicating the last date of calibration and the date when further calibration is required. At the time of the tests, the calibration of Items 1, 2, and 4 will be completed and entered on the appropriate Data Sheets using Items #3 and #5 as appropriate.

Other measurements, such as neutron attenuation, are compared with measurements on an archive sample of Boral or reference standards. Since these measurements are relative, no further calibrations are required.

Materials required:

1. Deionized water
2. 35mm color film (ASA 200 or slower)
3. Kimwipes or paper towels
4. Disposable latex gloves
5. Roll of masking tape
6. Small adhesive labels

5.0 COUPON DESCRIPTION:

The Boral coupons are approximately 4" x 8" and rectangular in configuration.

6.0 PROCEDURE FOR NON-DESTRUCTIVE TESTING OF BORAL COUPONS:

6.1 Procedure for Visual Inspection/Permanent Photo Record:

- a. Inspect coupon and note any anomalies such as corrosion films or pitting, blisters, discolorations, and record on Page 1 of the attached Data Sheets. Record location of anomalies on Page 2 of the attached Data Sheets.
- b. Take a minimum one high resolution color macro photograph of each side of each coupon. Adjust magnification of the lens to clearly show any anomalies and take additional photographs as required. Note the location of any anomalies on Page 2 of the attached Data Sheets for possible further characterization.

6.2 Procedure for Coupon Drying:

- a. Weigh coupon and record dry weight on Page 3 of the attached Data Sheets.
- b. Dry coupons at 175• F for 4 hours, cool to room temperature in a desiccator and measure dry weight. If weight shows a decrease after drying, continue drying and weighing per steps c and d below.
- c. Dry capsules again at 300• F for 4 hours and cool to room temperature in

a desiccator. Weigh and record weight on Page 3 of the attached Data Sheets. If there is no weight loss discontinue drying, otherwise continue drying as per d. below.

- d. Dry coupon at 500• F for 4 hours, cool capsule in desiccator and weigh at room temperature. Determine weight loss, if any, from weight in paragraph c above. If no further weight loss is observed discontinue drying. If weight loss is observed, further dry coupon per this step until no further weight loss is observed.

6.3 Procedure for Coupon Dimension Measurements:

- a. Check calibration of measuring instruments with certified gage block and record on Page 4 of the attached Data Sheets. Attach a copy of the NIST Certification to this procedure.
- b. Measure coupon length, width, and thickness at the locations as shown in Figure 1 using a precision micrometer. Record data on Page 4 of the attached Data Sheets. The measuring device used should provide an accuracy of ± 0.001 inches.

6.4 Procedure for Measuring Specific Gravity and Density:

- a. Set up balance over a container containing deionized water. Check balance by weighing appropriate calibration masses and record on Page 5 of the attached Data Sheets. Attach a copy of the NIST Certificates for the calibration masses to this procedure.
- b. Temporarily remove ID label from coupon noting its location and relative orientation of the coupon so that it can be replaced in the same location.
- c. Record final coupon weight in air from Paragraph 6.2 on Page 5 of the attached Data Sheets.
- d. Place an appropriate suspension yoke and wire harness on the pan of the balance. Re-zero balance to account for tare weight of yoke and harness.
- e. Place coupon in weighing harness and immerse in water.
- f. Inspect immersed coupon and verify that there are no adherent air bubbles on the coupon or harness. Air bubbles may be removed by rubbing lightly with a wetted finger.
- g. Record immersed weight of coupon on Page 5 of the attached Data Sheets.

- h. Remove coupon, dry the surface with a soft, absorbent cloth, and replace label.
- i. Measure and record temperature of the immersion water.
- j. Compute and record sample weights, specific gravity and density on Page 5 of the attached Data Sheets.

6.5 Procedure for Neutron Attenuation Testing (Performed in the Beam Hole Laboratory of the Pennsylvania State University Triga Reactor Facility):

- a. Place coupon, archive coupon or BORAL Standards and Cd plate in the xy positioning device in front of the beam hole.
- b. Confirm the counter plateau and operating high voltage of the neutron detector system.
- c. With the reactor set and locked at a steady state power level of 750 kw, obtain the following:
 - 1. Counting rate of the direct beam with no absorber in place.
 - 2. Counting rate with the Boral coupon between the neutron beam and detector such that the total counts are >10,000.
 - 3. Background count rate with a cadmium plate inserted in the neutron beam such that the total counts are >10,000.
 - 4. Steps 1 to 3 above may be performed in any desired sequence. For each of the counting rates in 1 to 3 above record the power beam count rate on Page 6 of the attached Data Sheets.
- d. Record count rates for 1 – 3 by either of two options, namely:
 - 1. Record all count rates on Page 6 of the attached Data Sheet,
 - 2. Utilize the NETCO automated data acquisition system.
- e. Repeat Step 2 for each coupon, archive sample and reference standards (if used).
- f. Compute the neutron attenuation of the coupon and archive sample (or BORAL Standards) by the following equation:

$$NA = 1 - (C_{ATTN} - C_{BKG}) / (C_{INC} - C_{BKG})$$

where:

NA = Neutron Attenuation

C_{ATTN} = Count rate (cpm) of the attenuated beam
 C_{INC} = Count rate (cpm) of the incident beam
 C_{BKG} = Background count rate (cpm) - with cd plate

6.6 Procedure for Rockwell Hardness Test

Note: For Rockwell E scale hardness test use 1/8" ball indenter.

- a. Assure that flat anvil is thoroughly clean and is securely attached to tester.
- b. Inspect coupon and ascertain that surface is clean and free of any foreign matter and assure that the indenter will not contact the coupon where its ID number has been inscribed.
- c. Place coupon on tester anvil such that ball indenter will contact coupon at location A in Figure 1.
- d. Raise coupon slowly with the aid of elevating screw gently easing the coupon against the indenter.
- e. After the coupon contacts the indenter apply the minor load by carefully forcing the coupon against the indenter until the small dial handle goes slightly past the minor load set point on appropriate scale.
- f. Set the large dial handle on appropriate scale to zero and apply the major load by releasing the crank.
- g. Be careful not to touch the coupon until the large dial handle has completely come to rest.
- h. After the dial handle comes to rest, retract the load by reversing the crank and returning to set point.
- i. Take the reading from the dial and record on page 7 of the attached data sheet.
- j. Inspect under side of coupon to determine if surface is in anyway disturbed.
- k. In a similar manner, take four more readings along the coupon at locations B, C, D and E shown on Figure 1.
- l. Take all five readings with the indenter contacting the same side of the coupon.

6.7 Procedure to Calibrate Hardness Tester Using NIST Traceable Test Blocks

- a. Assure that flat anvil is thoroughly clean and is securely attached to tester and that the tester is closed tight enough to hold test block in place.
- b. Inspect test block and ascertain that surface is clean and free of any foreign matter.
- c. Place test block on tester anvil such that ball indenter will contact coupon no less than 1/4" from edge, no closer than 1/2" from previous indentation and that the test block surface is perpendicular to the direction of indenter force.
- d. Raise test block slowly with the aid of elevating screw gently easing the

- coupon against the indenter.
- e. After the test block contacts the indenter apply the minor load by carefully forcing the test block against the indenter until the small dial handle on appropriate scale goes slightly past the minor load set point.
- f. Set the large dial handle on appropriate scale to zero and apply the major load by releasing the crank.
- g. Be careful not to touch the test block until the large dial handle has completely come to rest.
- h. After the dial handle comes to rest retract the load by reversing the crank.
- i. Take the reading from the dial and record on the data sheet.
- j. Repeat three times.
- k. Confirm that average reading is within tolerance of Rockwell units of stated hardness of test block.
- l. If specified tolerance is not achieved replace indenter ball with a new ball.

6.8 Procedure for Coupon Radioassay:

- a. Place coupon on a flat surface with coupon oriented as in Figure 1.
- b. Measure count rate with the G-M tube positioned at the top edge, center and bottom edge of the coupon.
- c. Record count rates on Page 7 of the attached Data Sheet.

6.9 Procedure for Surface Pit Characterization:

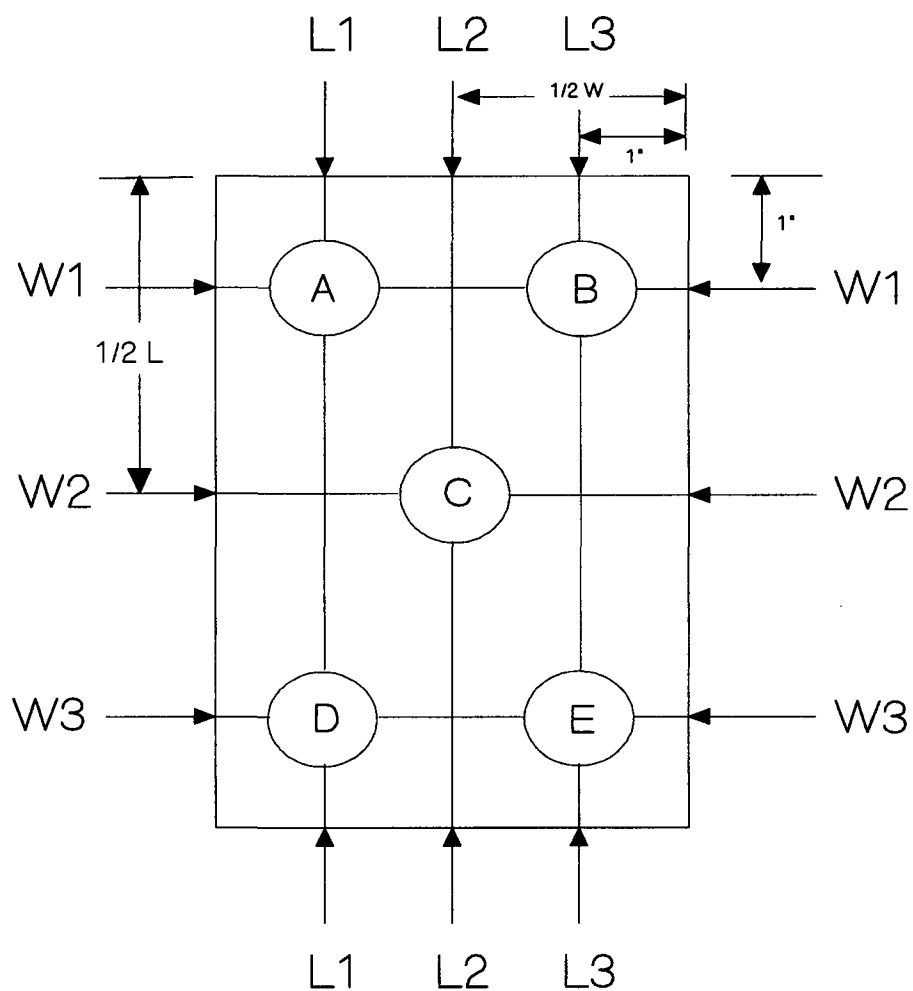
If surface pitting is noted on the BORAL Coupons, and the contract specifies pit characterization, the pit depth and width shall be measured via methods which conform with ASTM G16-88.

- a. If the surface of the BORAL Coupon has a heavy uniform oxide film, it may be advisable to clean the coupon to remove corrosion products with a cleaning solution. This will be determined when the coupons are subjected to visual inspection.
- b. If cleaning is determined to be advisable, wash the coupon in a mild detergent and use a soft brush to clean the surface. Rinse coupon in deionized water and dry. Immerse in 25% (by volume) nitric acid at room temperature for 10 to 15 minutes. Rinse with deionized water followed by an acetone wipe. Weigh sample and record weight. Take a high resolution micro-photo after cleaning.
- c. Use a metallurgical microscope with magnification range from 50 to 500x and a calibrated fine-focus knob. If the latter is not available, a dial micrometer can be attached to the microscope in such a way that it will show movement of the stage relative to the microscope body.

- d. Locate a single pit on the metal surface and center under the objective lens of the microscope at low magnification (for example, 50x). Increase the objective lens magnification until the pit area covers most of the field under view. Focus the specimen surface at the lip of the pit, using first the coarse and then the fine-focusing knobs of the microscope. Record the initial reading from the fine-focus knob and record the reading. The difference between the initial and final readings on the fine-focusing knob is the pit depth.
- e. Repeat the steps in 4 above to obtain additional measurements or until satisfactory duplications have been obtained. Record measurements on Page 8 of the attached Data Sheets.

7.0 CONTROL OF MATERIAL:

The LaSalle County Station coupons will be shipped to the Pennsylvania State University to the attention of the University Radiation Safety Officer. Upon receipt, NETCO personnel will be notified and the shipment consigned to NETCO. NETCO personnel will remove the coupons from the packaging and store them in a locked laboratory at the Breazeale Reactor Facility. The coupons will remain in the locked laboratory except for the time the procedures specified in Paragraph 6.5 are carried out. At the completion of testing, all coupons will be suitably repackaged and returned to Exelon if required. Those not returned will be disposed of as low level radioactive waste.



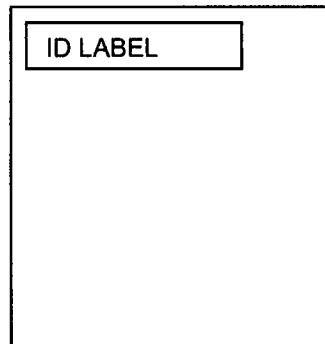
Thickness and Neutron Attenuation Testing at Locations A, B, C, D and E

Figure 1: Locations for the Coupon Measurements

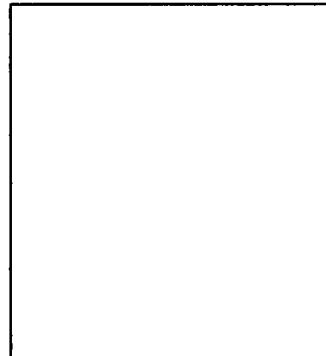
Data Sheet Page 2

Visual Inspection of Coupon (Provide Separate Sheet for Each Coupon)
 Note Location and Nature of Anomalies Below:

Coupon ID No. _____



FRONT SIDE OF COUPON



BACK SIDE OF COUPON

Inspected by: _____	Date: _____
Verified by: _____	Date: _____

Data Sheet Page 3

Dry Weight of Boral Coupons

[illegible]

Inspected by: _____ Date: _____

Verified by: _____ Date: _____

Data Sheet Page 4

Coupon Dimension Measurements:

Coupon ID No.	Inches:										
	L1	L2	L3	W1	W2	W3	TA	TB	TC	TD	TE

Length/Width Measurements	Thickness Measurements
Instrument: _____	Instrument: _____
Manufacturer _____	Manufacturer _____
Model/Serial No. _____	Model/Serial No. _____
Gage Block No: _____	Gage Block No: _____
Gage Size: _____	Gage Size: _____
Instrument: _____	Instrument: _____

Inspected by: _____ Date: _____

Verified by: _____ Date: _____

Data Sheet Page 5

Specific Gravity and Density:

Coupon ID No.	Dry Weight, gms	Immersed Weight, gms	Specific Gravity	Temperature, °C	Density of Water @ Temperature	Density, gm/cm ³

Balance:
Manufacturer:
Model/Serial No.:
Calibration Masses No.:
Mass Weight:
Balance Reading:

Inspected by: _____

Date: _____

Verified by: _____

Date: _____

Data Sheet Page 6

Neutron Attenuation Measurements:

Coupon ID Measurement Locations	Counts per ____ seconds			CP ____ S
	Attenuated Beam	Power Beam	Incident Beam	Background
Notes:				

Inspected by: _____

Date: _____

Verified by: _____

Date: _____

Data Sheet Page 7

Rockwell E Hardness Measurements

Coupon ID	A	B	C	D	E

Calibration Block ID _____
 Reading _____ Expected Hardness _____

 _____ Hardness Tester Serial/Model No. _____
 Average _____

Inspected by: _____	Date: _____
Verified by: _____	Date: _____

Radioassay Measurements

Coupon ID	Beta & Gamma, cpm			Gamma, cpm		
	Top	Center	Bottom	Top	Center	Bottom

Survey Instrument
 Serial/Model No: _____
 Manufacturer _____

Inspected by: _____	Date: _____
Verified by: _____	Date: _____

[illegible]

Microscope: Manufacturer: _____ Model No.: _____ Serial No.: _____

Appendix B

Procedure for Post-Test Characterization of the
Fast Start Surveillance Coupons, SEP-259-13, 6/30/09

Special Engineering Procedure
Procedure for Post-Test Characterization of the
Fast Start Surveillance Coupons

Prepared for:
Exelon Generation Company, LLC
4300 Winfield Road
Warrenville, IL 60555

Prepared by:
Scientech
NETCO Products and Services Division
108 North Front Street
UPO Box 4178
Kingston, NY 12402

Revision/Approval Record

Revision	Date	Prepared By:	Reviewed By:	Approved (QA):
0	6/30/09	R. L. L. L.	R. L. L. L.	L. P. Mariani

Table of Contents

1.0	Scope.....	1
2.0	Applicability.....	1
3.0	Related Documents	1
4.0	Required Test Equipment and Materials.....	2
5.0	Coupon Description	2
6.0	Schedule of Tests for Fast Start Surveillance Coupons and Test Sequence.....	3
7.0	Test Procedures.....	4
7.1	Procedure for Visual Inspection	4
7.2	Procedure for High Resolution Photography.....	4
7.3	Procedure for Coupon Drying and Dry Weight Determination	4
7.4	Procedure for Coupon Thickness Measurements.....	5
7.5	Procedure for Neutron Attenuation Testing.....	5
7.6	Procedure for Measuring Coupon Density	6

1.0 Scope

This procedure specifies the methods and techniques for measuring and recording the data associated with specified physical attributes of test coupons prior to subjecting the coupons to accelerated corrosion testing. In the implementation of this procedure, the following will be performed: visual inspection, high-resolution digital photography, determination of coupon dimensions and dry weight, neutron attenuation testing, microscopic examination and evaluation of corrosion rate. This work will be performed in NETCO's laboratory at Penn State, University Park, PA by NETCO personnel.

2.0 Applicability

This procedure applies to test measurements performed on Fast Start rectangular prepared Alcan coupons. The coupons have been supplied and inscribed with unique sequential identification numbers by NETCO. The procedures herein are to be used in conjunction with the NETCO-SNAP-IN® Materials Qualification Program.

3.0 Related Documents

- NETCO Quality Assurance Manual, Rev. 1, 8/10/07.
- NET-259-03, Rev 5, Material Qualification of ALCAN Composite For Spent Fuel Storage.
- SEP-259-04 Procedure for Accelerated Corrosion Testing of Aluminum Matrix Neutron Absorber Material Coupons for NETCO-SNAP-IN® Insert Absorber Qualification.
- SEP-259-11 Procedure for Precharacterizing the Fast Start Surveillance Coupons.
- SEP-259-12 Procedure for Post Test Characterization of the Accelerated Corrosion Coupons.
- ASTM G31-72 (Reapproved 2004) Standard Practice for Laboratory Immersion Corrosion Testing of Metals.
- ASTM G46-94 (2005) Standard Guide for Examination and Evaluation of Pitting Corrosion.
- ASTM G1-03 Standard Practice for Preparing, Cleaning and Evaluating Corrosion Test Specimens.
- ASTM G15-07 Standard Terminology Relating to Corrosion and Corrosion Testing.

4.0 Required Test Equipment and Materials

Test Equipment

1. Precision Micrometer (Starrett Model No. 222BR-1 or equivalent).
2. Gage Blocks, Steel, Weber Grade 2, .050" to 6.00" with Certificate of Calibration (NIST Traceable).
3. Balance (Ohaus TS400D or equivalent).
4. Standard masses, calibrated and certified (NIST Traceable).
5. High Resolution Digital Camera (Canon EOS D 60 or equivalent).
6. Thermometer, range 0 to 110° (1°C graduation) (Scientific Products Model T2050-1 or equivalent).
7. Desiccator.
8. Thickness measurement jig.
9. Metallurgical microscope with 50x to 500x magnification range and fine focus calibration knob.

Materials

1. Nitric Acid (1.42 sp. gr.)
2. Latex Gloves

To assure that tools, gages, instruments, and other measuring and test equipment used for the project are properly controlled, calibrated and adjusted at specified intervals in order to maintain the specified accuracy the following shall apply:

Calibration standards shall have certification and/or a sticker indicating the last date of calibration and the date when further calibration is required. At the time of tests, the calibration will be completed and appropriately recorded on the data form.

5.0 Coupon Description

The Fast Start surveillance coupons are rectangular and measure 2"x4" by 0.065" thick. All coupons are inscribed with a unique identification number. All test measurements and determinations will be keyed to the coupon ID number. The content of the coupons is 16 vol-% B₄C.

Precharacterization testing sequences for particular coupons is described in Section 6.0 of SEP-259-11, Procedure for Precharacterizing the Fast Start Surveillance Coupons. Table 1 of that document lists the tests to be applied to rectangular coupons. As applicable, Fast Start post-test characterization procedures are described in Section 7.0 below.

6.0 Schedule of Tests for Fast Start Surveillance Coupons and Test Sequence

Schedule of Tests

Table 1 shows the schedule of tests for each coupon.

Test	Pre-Characterization	Post-Characterization
Visual (High Resolution Digital Photography)	✓	✓
Dimensions	✓	✓
Dry Weight	✓	✓
Density	✓ +	✓
Areal Density	✓	✓
Acid Cleaning		✓
Weight Loss		✓
Corrosion Rate		✓
Microscopy		✓ *

TABLE 1: Schedule of Tests for Fast Start Surveillance Coupons

+ Measurement on archive coupons are acceptable

* as-required

Data developed during the course of the test schedule is to be recorded on the data sheet provided for rectangular fast start surveillance coupons. Use the following data sheet for recording measurement data:

Coupon Type

Rectangular

Data Sheet

SEP-259-13-01, Rev 0 (See Page 7)

After filling out data sheet, make a copy. File the copy as "file copy" and file the original as "working copy". The working copy will be used to transfer data to tables in the NETCO ACCESS database MasterCouponData.mdb. Alternatively, the data may be entered directly into the NETCO ACCESS database.

Test Sequence

- a. Perform visual inspection and high resolution photography per paragraphs 7.1 and 7.2.
- b. Measure coupon weight per paragraph 7.3.
- c. Measure coupon thickness as per paragraph 7.4.
- d. For those coupons scheduled for neutron attenuation testing, conduct testing per paragraph 7.5.
- e. For those coupons scheduled for density measurement, conduct testing per paragraph 7.6.

CAUTION

The following steps are intended to remove only the aluminum oxide film and not the base metal. Inspect surface of coupon under a microscope after each acid wash to determine whether the oxide has been removed.

- f. Prepare a bath of nitric acid (HNO_3) (1.42sp.gr.) and immerse coupons for the number of minutes specified by the Project Manager. Remove coupons and rinse in deionized water.
- g. Dry coupons at 220° F for 1 hour to remove surface moisture.
- h. Cool coupons to room temperature in a desiccator and determine the dry weight per paragraph 7.3.
- i. Repeat steps f. through h. until constant weight is achieved.
- j. Measure coupon thickness per paragraph 7.4.
- k. Perform optical microscopy of any anomalies of the coupon and take photomicrographs as directed by the Project Manager.

7.0 Test Procedures

7.1 Procedure for Visual Inspection

- a. Inspect each coupon and note any anomalies such as scratches or surface or edge imperfections. Use data entry to describe observed appearance and to record the location of anomalies.

7.2 Procedure for High Resolution Photography

- a. Take a minimum of one high-resolution color macro photograph of each side of each coupon. Several coupons can be photographed at once at the discretion of the Project Manager.
- b. Adjust magnification of the lens to show any anomalies and take additional photographs as required. Note the location of any anomalies on the data entry form.

7.3 Procedure for Coupon Drying and Dry Weight Determination

- a. Check the calibration of the balance by weighing the appropriate calibration masses.
- b. Make sure coupon is clean, dry and free of foreign matter
- c. Weigh coupon and record weight on data form.

7.4 Procedure for Coupon Thickness Measurements

- a. Check calibration of measuring instruments with certified gage block and record on the data sheet. Record number of NIST certification on the data sheet. Retain NIST certification as QA record.
- b. The measurement of coupon thickness shall be made with the coupons placed in a special fixture which assures that the thickness is measured at the same location for pre-test and post-test characterizations.
- c. Place measurement jig on flat surface with the markings aligned as shown on the sketch in Data Sheet SEP-259-13-01, Rev 0.
- d. Place the coupon into measurement jig such that ID number is located in the upper right hand corner of the jig.
- e. Measure coupon thickness as shown on the data input form. Record measurements on data input form in the locations corresponding to locations of measurement. The instrument for measuring coupon thickness should provide a resolution of ± 0.0001 inches.

7.5 Procedure for Neutron Attenuation Testing

(This procedure is to be performed in the Beam Hole Laboratory of The Pennsylvania State University TRIGA Reactor Facility).

- a. Place the coupon, the archive coupon and the Cd plate in the positioning track.
- b. Confirm the counter plateau and operating high voltage of the neutron detection system.

- c. With the reactor set and locked at a steady state power level of 800 kilowatts, obtain the following:
 1. Five minute count of the direct beam with no absorber in place.
 2. Thirty second or longer counting rate with the METAMIC coupon in place between the neutron beam and detector such that the total counts are $> 10,000$.
 3. Three-minute background count with a cadmium plate inserted in the path of the neutron beam.
 4. Steps 1 to 3 above may be performed in any convenient sequence.
- d. Record count rates for steps 1-3 above on data input form.
- e. Repeat step 2 for each coupon in the measurement series, any archive samples specified by the Project Manager and the following hot pressed boron composite calibration standards:
 - 0.020
 - 0.025
 - 0.030
 - $0.020 + 0.025$
 - $0.020 + 0.030$
 - $0.025 + 0.030$

The identifying number above refers to the thickness of the boron carbide standard in inches.

7.6 Procedure for Measuring Coupon Density

- a. Set up balance over a vessel containing deionized water. Check balance by weighing appropriate calibration masses and record measured values on data input form. Record the number of NIST certification of calibration mass on the data sheet.
- b. Record coupon final dry weight on data input form.
- c. Place an appropriate suspension yoke and wire harness on the pan of the balance. Re-zero the balance to account for the tare weight of the yoke and harness.
- d. Place coupon in weighing harness and immerse in water.
- e. Inspect immersed coupon and verify that there are no adherent bubbles of air on either the coupon or harness. Air bubbles may be removed by lightly rubbing with wetted finger.
- f. Record immersion weight on the appropriate data form.
- g. Remove coupon and dry the surface with a soft absorbent cloth.
- h. Measure and record temperature of the immersion water.
- i. Compute and record coupon density on the appropriate data form.

Data Sheet, SEP-259-13-01

Coupon ID _____

1. Visual Inspection:

Pre-test; _____

Date: _____ Time: _____ By: _____

Post-test; _____

Date: _____ Time: _____ By: _____

2. Photo ID's: Pre-test _____ Post-test: _____

3. Coupon Dimensions (in.) See coupon on upper left*

	<u>Pre-test, in.</u>	<u>Post-test, in.</u>	<u>After Acid Wash</u>
Date:	_____	_____	_____
Time:	_____	_____	_____
By:	_____	_____	_____
T1	_____	_____	_____
T2	_____	_____	_____
T3	_____	_____	_____
T4	_____	_____	_____
T5	_____	_____	_____
T6	_____	_____	_____
T7	_____	_____	_____
T8	_____	_____	_____
T9	_____	_____	_____

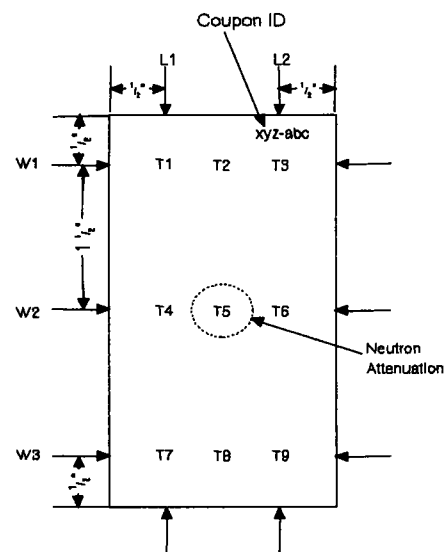
4. Dry Weight (gms)

Standard Masses

Date: _____ Time: _____ By: _____

	<u>Pre-test</u>	<u>Post-test</u>
As received, gms	_____	_____
As dried, gms	_____	_____

5. Comments: _____



* For bend samples T1 through T6 only.

Appendix C

Certification of Calibration for Gage Blocks, Masses and Rockwell Hardness Tester



Shore® Instruments - Instron®
825 University Avenue ■ Norwood, MA 02062-2643
Tel: +1-781-575-6000 ■ Fax: +1-781-575-5770

Certificate of Calibration



Customer Name: NETCO

Customer Address: 108 NORTH FRONT STREET KINGSTON, NY 12401

Durometer Model: Analog Durometer Type: A Certificate Number: 12506110754

Serial Number: 110754 Date of Calibration: 12/5/2006 Date of Last Calibration: December 19, 2002

The procedure used to calibrate this unit was: ICA-5-11

Calibration performed in compliance with: ASTM D2240(05), ISO/IEC 17025(05), ANSI/NCSL Z540-1(02) & ISO 10012(03)

Does this Instrument contain a maximum indicator: ☒ Yes or ☐ No and/or a timing device: ☐ Yes or ☒ No

The calibration measurement values are traceable to NIST or other recognized National Standards.

The calibration standards used for this unit were: SHO21, SAS179

Calibration standard date(s): 4-14-05, 7-7-06

Calibration standard due date(s): 4-14-07, 7-7-07

Condition of unit as received: IN TOLERANCE

Load limits of permissible error: +/-0.075 Newtons = (+/-7.65 Grams Force) = (+/-1.00 Durometer Points)

Indenter extension limits of permissible error: 2.5mm (0.098 Inches) +/- 0.04mm (0.002 Inches)

Environmental conditions: Temperature: 23 C Humidity: 15%

Durometer Test Point	Nominal Load		As Found		As Left		As Left Data Deviation	
	Newtons	(Grams)	Newtons	Grams	Newtons	Grams	Newtons	Duro Points
10	1.300	132.6	1.324	135.0	1.344	137.0	0.044	0.59
20	2.050	209.0	2.050	209.0	2.059	210.0	0.009	0.12
30	2.800	285.5	2.785	284.0	2.785	284.0	-0.015	-0.20
40	3.550	362.0	3.521	359.0	3.521	359.0	-0.029	-0.39
50	4.300	438.5	4.276	436.0	4.276	436.0	-0.024	-0.32
60	5.050	515.0	5.050	515.0	5.041	514.0	-0.009	-0.12
70	5.800	591.4	5.786	590.0	5.796	591.0	-0.004	-0.05
80	6.550	667.9	6.551	668.0	6.541	667.0	-0.009	-0.12
90	7.300	744.4	7.296	744.0	7.286	743.0	-0.014	-0.19
Indenter Extension:			0.0990 Inches		0.0990 Inches			

* Indicates Out of Tolerance Condition

Maintenance, adjustments, repairs or modifications made prior to final calibration: REPLACE INDENTOR & RECALIBRATE.

The Durometer's maximum indicator is operating correctly and within Shore's internal calibration tolerance: ☒ Yes ☐ No ☐ N/A

The test force measurement uncertainty is: +/- .013 N = +/- .17 D.P. (Durometer Points)

The indenter extension measurement uncertainty is: +/- 1.4 Micrometers = +/- .06 D.P.

The estimated overall measurement uncertainty utilizing the indenter extension and test force components is: +/- 1.16 Durometer Points

Note: The reported expanded measurement uncertainty is based on a standard uncertainty multiplied by a coverage factor of K = 2 providing a level of confidence of approximately 95%

Note: the uncertainties in DP units are given for information only and were calculated from the defined relationships between the applied force or the indenter travel

This certificate shall not be reproduced, except in full, without the written approval of Instron Corporation

This test report must not be used by the client to claim product certification, approval, or endorsement by NVLAP, NIST or any agency of the federal government.

Calibration Notes: _____

Calibrated By: HIEU NGUYEN Title: E/M ASSEMBLER

Calibration Approved By: [Signature] Title: E/M Technician



CERTIFICATE OF CALIBRATION



Cert#: 1198.01

Calibration Performed By:
EDMUNDS GAGES
45 SPRING LANE
FARMINGTON INDUSTRIAL PARK
FARMINGTON, CT 06032
USA

Calibration Performed For:
NORTHEAST TECHNOLOGY CORP
KINGSTON, NY 12402
USA

Customer Set Information:

Set Serial No.: IHR15
Make: Webber
Material: Steel
Grade: 2
Type: Rectangle
No. of Pieces: 9

Master Set Information:

Set Serial No. Used: EG-25-7
Last Calibrated: Sep 05, 2006
Calibration Due Date: Sep 05, 2007

Comparator Information:

Comparator Number: EG-040-002
Calibrated: Daily

Calibration Date:
Dec 05, 2006

Certificate Number: 2899-GB

Cust. P.O. Number: 061103

Deviation Expressed In Micro Inches

Size	Serial No.	Deviation	Annot.	Size	Serial No.	Deviation	Annot.	Size	Serial No.	Deviation	Annot.	Size	Serial No.	Deviation	Annot.
.030	IHR15	3.0													
.050	IHR15	2.0													
.070	IHR15	3.0													
.090	IHR15	0.0													
.120	IHR15	4.0													
1.000	IHR15	1.0													
2.000	IHR15	6.0													
3.000	IHR15	10.0													
4.000	IHR15	3.0													

This Certificate of Calibration conforms to ISO/IEC 17025. Temperature is held to 68 +/- .5 degrees & humidity is held to 35-50 percent. The reference standards for this calibration is block set 134, NIST #821/263676-00. Measurements made conform to the tolerance specifications listed in ANSI B89.1.9M unless listed as otherwise. Measurement uncertainty is stated in Table 1 below and is NOT taken into account with respect to the tolerance. The report expanded uncertainty of measurement is stated as the standard uncertainty of measurement multiplied by the coverage factor K=2, which for a normal distribution corresponds to a coverage probability of approx. 95%. This report is neither a contract nor a contractual representation. Edmunds responsibility shall in no event, nor in any case whatsoever exceed the purchase price of this calibration. This report shall not be copied or reproduced except in full without the written approval from Edmunds Gages.

Inspected By:

Pass ☒ Fail ☐

Table 1:	Range	Uncert.	Range	Uncert.
	0-1 in	3.6 μ in	8-12 in	20 μ in
	1-4 in	6.3 μ in	12-20 in	30 μ in
	4-8 in	15 μ in		

Comments:

AS FOUND COND. FAIR.



CERTIFICATE OF CALIBRATION



Cert#: 1198.01

Calibration Performed By:
EDMUNDS GAGES
45 SPRING LANE
FARMINGTON INDUSTRIAL PARK
FARMINGTON, CT 06032
USA

Calibration Performed For:
NORTHEAST TECHNOLOGY CORP
KINGSTON, NY 12402
USA

Customer Set Information:

Set Serial No.: LES 2
Make: Webber
Material: Steel
Grade: 2
Type: Rectangle
No. of Pieces: 1

Master Set Information:

Set Serial No. Used: EG-25-20
Last Calibrated: Aug 30, 2006
Calibration Due Date: Aug 30, 2007

Comparator Information:

Comparator Number: EG-040-002
Calibrated: Daily

Calibration Date:
Dec 05, 2006

Certificate Number: 2898-GB

Cust. P.O. Number: 061103

Deviation Expressed in Micro Inches

Size	Serial No.	Deviation	Annot.
6.000	LES 2	11.0	

Size	Serial No.	Deviation	Annot.
------	------------	-----------	--------

Size	Serial No.	Deviation	Annot.
------	------------	-----------	--------

Size	Serial No.	Deviation	Annot.
------	------------	-----------	--------

Size	Serial No.	Deviation	Annot.
------	------------	-----------	--------

This Certificate of Calibration conforms to ISO/IEC 17025. Temperature is held to 68 +/- .5 degrees & humidity is held to 35-50 percent. The reference standards for this calibration is block set 134, NIST #821/263676-00. Measurements made conform to the tolerance specifications listed in ANSI B89.1.9M unless listed as otherwise. Measurement uncertainty is stated in Table 1 below and is NOT taken into account with respect to the tolerance. The report expanded uncertainty of measurement is stated as the standard uncertainty of measurement multiplied by the coverage factor K=2, which for a normal distribution corresponds to a coverage probability of approx. 95%. This report is neither a contract nor a contractual representation. Edmunds responsibility shall in no event, nor in any case whatsoever exceed the purchase price of this calibration. This report shall not be copied or reproduced except in full without the written approval from Edmunds Gages.

Inspected By:

Pass Fail

Table 1:

Range	Uncert.	Range	Uncert.
0-1 in	3.6 µin	8-12 in	20 µin
1-4 in	6.3 µin	12-20 in	30 µin
4-8 in	15 µin		

Comments:

AS FOUND COND. FAIR.



CERTIFICATE OF CALIBRATION



Cert#: 1198.01

Calibration Performed By:
EDMUNDS GAGES
45 SPRING LANE
FARMINGTON INDUSTRIAL PARK
FARMINGTON, CT 06032
USA

Calibration Performed For:
NORTHEAST TECHNOLOGY CORP
KINGSTON, NY 12402
USA

Customer Set Information:

Set Serial No.: LES 2
Make: Webber
Material: Steel
Grade: 2
Type: Rectangle
No. of Pieces: 1

Master Set Information:

Set Serial No. Used: EG-25-20
Last Calibrated: Aug 30, 2006
Calibration Due Date: Aug 30, 2007

Comparator Information:

Comparator Number: EG-040-002
Calibrated: Daily

Calibration Date:
Dec 05, 2006

Certificate Number: 2897-GB

Cust. P.O. Number: 061103

Deviation Expressed In Micro Inches

Size	Serial No.	Deviation	Annot.
5.000	LES 2	7.0	

Size	Serial No.	Deviation	Annot.
------	------------	-----------	--------

Size	Serial No.	Deviation	Annot.
------	------------	-----------	--------

Size	Serial No.	Deviation	Annot.
------	------------	-----------	--------

Size	Serial No.	Deviation	Annot.
------	------------	-----------	--------

This Certificate of Calibration conforms to ISO/IEC 17025. Temperature is held to 68 +/- .5 degrees & humidity is held to 35-50 percent. The reference standards for this calibration is block set 134, NIST #821/263676-00. Measurements made conform to the tolerance specifications listed in ANSI B89.1.9M unless listed as otherwise. Measurement uncertainty is stated in Table 1 below and is NOT taken into account with respect to the tolerance. The report expanded uncertainty of measurement is stated as the standard uncertainty of measurement multiplied by the coverage factor K=2, which for a normal distribution corresponds to a coverage probability of approx. 95%. This report is neither a contract nor a contractual representation. Edmunds responsibility shall in no event, nor in any case whatsoever exceed the purchase price of this calibration. This report shall not be copied or reproduced except in full without the written approval from Edmunds Gages.

Inspected By:

4 P

Pass Fail

Table 1:

Range	Uncert.	Range	Uncert.
0-1 in	3.6 µin	8-12 in	20 µin
1-4 in	6.3 µin	12-20 in	30 µin
4-8 in	15 µin		

Comments:

AS FOUND COND. FAIR.



STATE OF CONNECTICUT

DEPARTMENT OF CONSUMER PROTECTION

Report of Test for: 1 - SET OF METRIC WEIGHTS, 1 KILOGRAM TO 1 GRAM (13 WEIGHTS)

Submitted by:

ATTN: LEO MARIANI

NORTHEAST TECHNOLOGY CORP.

108 NORTH FRONT STREET

UPO BOX 4178

KINGSTON ,NY 12402

TESTED BY: DEPT. OF CONSUMER PROTECTION, DIVISION OF FOOD AND STANDARDS - METROLOGY LABORATORY

Manufacturer: TROEMNER

S/N: 7977

CT. TEST NO. 15240

The Weights described above have been compared with the standards of the State of Connecticut (Working Standards), National Institute of Standards and Technology Test No.822/266626-02 and were found (adjusted) to be accurate to within ASTM CLASS 6 TOLERANCE as indicated on the attached tolerance table.

The following mass values reflect the as found condition of weights.

<u>Denomination</u>	<u>Class F TOLERANCE MILLIGRAMS</u>	<u>VALUE FOUND</u>		<u>ADJUSTED MILLIGRAMS</u>	<u>Uncertainty (+/- MILLIGRAMS)</u>
		<u>Correction</u>	<u>vs. 8.0g/cu.cm</u>		
1Kg	100	1.20		Same	0.37
500g	70	0.70		Same	0.36
200g	40	19.56		Same	0.36
200g *	40	23.66		Same	0.36
100g	20	11.08		Same	0.10
50g	10	4.51		Same	0.10
20g	4	2.50		Same	0.10
20g *	4	0.39		Same	0.10
10g	2	1.54		Same	0.10
5g	1.5	0.597		Same	0.037
2g	1.12	1.034		Same	0.037
2g *	1.12	1.016		Same	0.037
1g	0.9	0.392		Same	0.037

Test Procedure: SOP #8, MODIFIED SUBSTITUTION

temp - 25.3C, humidity - 23.0%, pressure - 764.7 mmHg

DATE OF TEST: 12/05/06

NEXT CALIBRATION DUE PER REQUEST: 12/05/10

Signing Authority

METROLOGIST: MICHAEL DYMIA
FOOD AND STANDARDS DIVISION

NOTE:

This report relates only to the items listed in this report at the time of test.

The uncertainties are calculated according to NIST Technical Note 1297, where the expanded uncertainty is the root sum square of the of the Type A and Type B uncertainties.

The effect of air buoyancy has been considered negligible at this tolerance level.

This report shall not be reproduced except in full , without written approval of the laboratory.

165 Capitol Avenue, Hartford, Connecticut 06106-1630
TDD (Telecommunications Device for the Deaf): (860) 713-7240

Internet Web Site: <http://www.ct.gov/dcp>

An Affirmative Action / Equal Opportunity Employer



STATE OF CONNECTICUT

DEPARTMENT OF CONSUMER PROTECTION

Report of Test for: 1 - SET OF METRIC WEIGHTS, 400 GRAMS TO 10 GRAM (7 WEIGHTS)

Submitted by:
Attn: Leo Mariani
Northeast Technology Corp.
108 North Front Street
UPO Box 4178
Kingston, NY 12402

ASSUMED DENSITY OF STANDARDS
7.84g/cu.cm.

TESTED BY: DEPT. OF CONSUMER PROTECTION, DIVISION OF FOOD AND STANDARDS METROLOGY LABORATORY

Manufacturer: Troemner
S/N: AS BELOW

CT. TEST NO. 15239

The weights described above have been compared with the Standards of the State of Connecticut (Primary Metric Set), National Institute of Standards and Technology Test No. 822/266626-02 and were found to have the following conventional mass values.


Conventional Mass Correction Versus Reference Density of 8.0g/ cubic cm					Uncertainty
Denomination	S/N		(Milligrams)		(+/- Milligrams)
400g	41454		0.27		0.11
100g	31259	MINUS	-0.136		0.042
50g	31260	MINUS	-0.051		0.028
30g	24609		0.075		0.024
20g	31261	MINUS	-0.038		0.023
10g *	25311	MINUS	-0.001		0.011
10g **	25312		0.016		0.011

Test Procedure: SOP #4, DOUBLE SUBSTITUTION

temp - 25.1C, humidity - 22%, pressure - 764.8 mmHg

DATE OF TEST: 12/5/06
NEXT CALIBRATION DUE PER REQUEST: 12/5/10

Signing Authority


METROLOGIST: MICHAEL DYNIA
FOOD AND STANDARDS DIVISION

NOTES:

- (1) Apparent Mass has been changed to Conventional Mass to conform to international terminology and ISO guidelines.
- (2) The uncertainties are calculated according to NIST Technical Note 1297, where the expanded uncertainty is 2 times the root sum square of the of the Type A and Type B uncertainties.
- (3) This report shall not be reproduced except in full , without written approval of the laboratory.
- (4) THE REPORT OF TEST IS INVALID WITHOUT THE OFFICIAL STATE OF CONNECTICUT WEIGHTS AND MEASURES SEAL AFFIXED.
- (5) This report relates only to the items listed in this report at the time of test.