



Northern States Power Company  
Prairie Island Nuclear Generating Plant  
1717 Wakonade Dr. East  
Welch, Minnesota 55089

November 4, 1996

NRC Bulletin 96-04

U S Nuclear Regulatory Commission  
Attn: Document Control Desk  
Washington, DC 20555

**PRAIRIE ISLAND INDEPENDENT SPENT FUEL STORAGE INSTALLATION**  
Docket No. 72-10  
Materials License No. SNM-2506

**Final Response to Bulletin 96-04**  
**Chemical, Galvanic, or Other Reactions in**  
**Spent Fuel Storage and Transportation Casks**

In response to the Ventilated Storage Cask (VSC-24) loading event that occurred at the Point Beach Nuclear Plant on May 28, 1996, the NRC Staff issued Bulletin 96-04, dated July 5, 1996. The bulletin requested a response by those addressees who are licensees with independent spent fuel storage installations (ISFSI), which includes the Prairie Island ISFSI. A preliminary response for the Transnuclear TN-40 casks utilized at the Prairie Island Independent Spent Fuel Storage Installation (ISFSI) was provided by our letter dated August 19, 1996. NSP has completed the review of the TN-40 cask materials, coatings and lubricants and our final response to Bulletin 96-04 is attached. This response is intended to stand alone and supersedes the August 19, 1996 preliminary response.

In this letter we have made no new Nuclear Regulatory Commission commitments. If you have any questions related to the attached information, please contact Gene Eckholt (612-388-1121).

*Michael D Wadley*

Michael D Wadley  
Plant Manager  
Prairie Island Nuclear Generating Plant

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NORTHERN STATES POWER COMPANY

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NMSS Project Manager, NRC  
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Senior Resident Inspector, NRC  
NRR Project Manager, NRC  
J E Silberg  
Prairie Island Independent Spent Fuel Storage Installation Service List

Attachments:

Affidavit

1. Final Response to NRC Bulletin 96-04
2. Aluminum Metal Interaction With Boric Acid Solution Test Report
3. Boron Metal Interaction With Boric Acid Solution Test Report
4. Zinc/Aluminum Metal Interaction With Boric Acid Solution Test Report

UNITED STATES NUCLEAR REGULATORY COMMISSION

NORTHERN STATES POWER COMPANY

PRAIRIE ISLAND INDEPENDENT SPENT FUEL STORAGE INSTALLATION  
DOCKET NO. 72-10

FINAL RESPONSE TO  
BULLETIN 96-04, CHEMICAL, GALVANIC, OR OTHER REACTIONS  
IN SPENT FUEL STORAGE AND TRANSPORTATION CASKS

Northern States Power Company, a Minnesota corporation, with this letter is submitting information requested by NRC Bulletin 96-04.

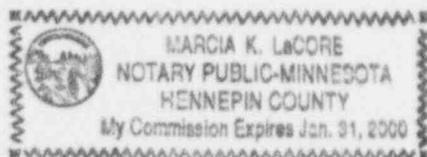
This letter contains no restricted or other defense information.

NORTHERN STATES POWER COMPANY

BY Michael D Wadley  
Michael D Wadley  
Plant Manager  
Prairie Island Nuclear Generating Plant

On this 4<sup>th</sup> day of November 1996 before me a notary public in and for said County, personally appeared Michael D Wadley, Manager, Prairie Island Nuclear Generating Plant; and being first duly sworn acknowledged that he is authorized to execute this document on behalf of Northern States Power Company, that he knows the contents thereof, and that to the best of his knowledge, information, and belief the statements made in it are true and that it is not interposed for delay.

Marcia K LaCore



**Attachment 1**

**Final Response to NRC Bulletin 96-04**

## Final Response to NRC Bulletin 96-04

### Chemical, Galvanic, or Other Reactions in Spent Fuel Storage and Transportation Casks

#### Requested Actions:

1. Address the following items relating to the susceptibility of the spent fuel storage or transportation cask design to chemical, galvanic, or other reactions:
  - (a) Review the cask materials, including coatings, lubricants, and cleaning agents, to determine whether chemical, galvanic, or other reactions among the materials, contents, and environment can occur during any phase of loading, unloading, handling, storage, and transportation. Consideration should be given to all environments that may be encountered under normal, off-normal, or accident conditions.

#### Response to Requested Action 1 (a):

The TN-40 storage cask(s) at the Prairie Island Nuclear Generating Plant (PINGP) consist of the following components:

- Basket assembly for support of the fuel assemblies
- Containment vessel enclosing basket assembly and fuel
- Gamma shield
- Neutron shield
- Outer shell
- Weather cover
- Pressure monitoring system
- Trunnions

The casks are self supporting cylindrical vessels. Table 1.3-2 of the Prairie Island ISFSI Safety Analysis Report (SAR) provides a list of component descriptions and **materials** for the TN-40 cask.

The basket structure consists of an assembly of stainless steel cells joined by a proprietary fusion welding process and separated by aluminum and poison plates (Boral) which form a sandwich panel. The panel consists of two 0.25 inch thick aluminum plates which sandwich a poison plate 0.075 inch thick. The aluminum provides the heat conduction paths from the fuel assemblies to the cask cavity wall. The poison material provides the necessary criticality control. This method of

## Final Response to NRC Bulletin 96-04

construction forms a very strong honeycomb-like structure of cell liners which provide compartments for 40 fuel assemblies. The interior panels are held in place by a series of stainless steel plugs which are welded to the stainless steel cell walls. Neither the aluminum plates nor Boral plates are welded or bolted to the stainless steel cells of the basket structure.

The containment vessel for the TN-40 cask consists of: an inner shell which is a welded, carbon steel cylinder with an integrally-welded, carbon steel bottom closure; a welded flange forging; a flanged and bolted carbon steel lid with bolts, and penetration assemblies (vent and drain) with bolts. There are six (6) aluminum rails bolted to the interior to provide basket alignment.

A gamma shield is provided around the walls of the containment vessel by an independent shell of carbon steel which is welded to a bottom shield plate and to the closure flange. The gamma shield completely encloses the containment vessel inner shell and bottom closure.

Neutron shielding is provided by a proprietary borated polyester resin compound around the cask body and a polypropylene disk that covers the top lid. The resin compound is enclosed in long, slender aluminum containers. The array of resin-filled containers is enclosed within a 1/2 inch thick outer steel shell. The polypropylene disk (top neutron shield drum) is enclosed in a steel shell which is attached to the lid for storage on the ISFSI pad.

Four trunnions are attached to the cask body for lifting and rotation of the cask. Two of the trunnions are located near the top of the body and two near the lower end of the body.

Reviewing SAR Table 1.3-2, the following components are made of low alloy steels:

- Shell & Bottom (Shell)
- Lid
- Inner Containment & Bottom (Containment)
- Trunnions
- Outer Shell Protective Cover
- Lid bolts
- Protective Cover bolts
- Top Neutron Shield bolts
- Over Pressure (OP) Tank
- Drain & Vent Port Cover bolts
- OP Port Cover bolts
- Lid Alignment Pins (Cask #01 only)
- Shield Plate
- Basket Guide Rail bolts

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From the above list, only the Shell, Lid, Containment, Trunnions, Basket Guide Rail bolts and Lid Alignment Pins are immersed in the Spent Fuel Pool (SFP). The shell, lid and containment have the exposed carbon steel surfaces coated with either/both a plasma sprayed Aluminum/Zinc coating or/and a high gloss polyamide epoxy enamel paint. Under section 1.3 of the SAR, General Description, it states: "the cask cavity surfaces and the outer shell have a sprayed metallic coating of Zn/Al for corrosion protection. The external surfaces of the cask are painted for ease of decontamination." Both the paint and metallic spray provide a protective barrier for the base material. This is supported in SAR section 4.2.3.1.4, Design Basis, where it states "The outer shell is carbon steel protected by a metallic coating and paint."

The trunnions' steel are exposed to the SFP water along the outer surface where there is contact with the lift beam. Using an estimated corrosion rate of 0.05 inches per year for low alloy steel, at the concentration of boric acid in the SFP, the maximum corrosion would be estimated at 0.0004 inches over a 72 hour period. Using this maximum corrosion rate, the effect on the trunnions is not significant to warrant further review. Similarly, the basket guide rail bolts would not be significantly impacted by the short duration that the cask is exposed to the SFP water.

The lid alignment pins have been replaced with an austenetic steel under a previous 72.48 safety evaluation. The corrosion rate for austenetic steel is a fraction of the low alloy steel as shown below. The original pins would conservatively be estimated to corrode at the same rate as above.

The following components are made from stainless steel:

- Type 304
  - Drain Port Cover
  - Vent Port Cover
  - Fuel Cell Compartments
  - OP Port Cover
  
- Type 316
  - Lid Alignment Pins

Only the Fuel Cell Compartments and lid alignment pins are exposed to the SFP water during normal cask loading operations. Reviewing several sources (NACE Corrosion Data Survey, Schweitzers Corrosion Resistance Tables, Perry's Chemical Engineering Handbook), the corrosion rate of 304 SS in boric acid solutions is less than 0.002 inches per year. Using 72 hours as an exposure period to the SFP water will yield an infinitesimally small corrosion value. Similarly small corrosion rates would apply to the alignment pin material.

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Aluminum or aluminum alloy are contained within the following:

- Radial Neutron Shield Boxes
- Lid Seal
- Drain and Vent Port Cover Seals
- Basket Structure (alloy 6061)
- Basket Poison Plate (Boral, an Aluminum-Boron Carbide composite)
- Plasma Spray (Zn/Al)
- Basket Rails (alloy 6061)

The radial neutron shield boxes are encased in the cask's outer shell and are not exposed to any of the cask's external environment. The lid seal contributes a minor volume of aluminum when compared to the last four items on the above list. The drain and vent port cover seals are not immersed in the SFP during normal operations with the TN-40 cask.

The last four items above comprise the majority of the aluminum that is exposed to the SFP water. Using information from NUREG-24532 "Hydrogen Release Rates From Corrosion of Zinc and Aluminum", it is possible to generate hydrogen given the correct set of conditions when in boric acid. The aluminum corrosion rate is approximately 1.5% of the zinc rate.

NSP contacted AAR Advanced Structures, the manufacturer of Boral used in the TN-40 cask. No tests were available regarding hydrogen generation when Boral is exposed to SFP environments. In the absence of data, NSP performed a test on a sample of Boral to quantify the generation of any gas. The test results are summarized later in this response.

The **coatings** used on the TN-40 cask consist of:

- Stainless steel overlay
- Zinc/aluminum (Zn/Al) metallic coating
- Epoxy based paint
- RTV sealant
- Chrome (bolt coating)
- Carboline 305 paint

A stainless steel overlay is applied to the O-ring seating surfaces on the containment closure flange and lid. The overlay is approximately 1/4 inch thick and provides a uniform, corrosion resistant surface for the cask seal.

The cask cavity surfaces and the outer shell have a plasma sprayed metallic coating of Zn/Al for corrosion protection. A review of the material certification for the raw wire indicates that the Zinc composition is 99.99% minimum and the Aluminum is 99.5% minimum. The two materials are applied simultaneously with a final composition of

## Final Response to NRC Bulletin 96-04

approximately 70% Zn and 30% Al. The metallic spray coating thickness is typically in the range of 0.004" to 0.015". On the exterior surfaces of the cask and lid, the Zn/Al coating is covered with a primer and finish coat of epoxy enamel paint for ease of decontamination. The Zn/Al coating is not sealed on the inner containment shell of the cask.

The RTV sealant is applied to portions of the cask exterior after final receipt inspection at the site. The primary location is around the lower trunnion and small irregularities in the outer surface finish to seal crevices from potential contamination during the loading sequence. The RTV is an approved product for use in the SFP environment and no further reviews are warranted, based on its limited use on only the cask exterior. Per the PINGP Chemical Material Control Plan, the definition of an approved chemical is one approved for use in the primary and secondary fluid systems or for contact with QA 1 stainless steel or nickel alloys.

The chrome is plated to the lid bolts and weather cover bolts at 0.0002" thick. The chrome plating is applied per ASTM B-177. No bolts are immersed in the SFP water during normal cask operations.

The top of the drain tube has approximately 3.5 sq. inches of Carboline 305 paint applied to it. The product manufacturer was contacted, and use of the 305 paint on the drain tube is consistent with their product recommendations in this case. Additionally, NSP reviewed video footage of two previous TN-40 fuel load verifications. The paint on the drain tube was not a source of gas bubbles as verified by the clean surface on the paint when seen in the footage during fuel load verification. Due to the small surface area, the manufacturer's response, and the passivity exhibited in the SFP, no further evaluation was deemed necessary.

Last, there are a series of polymeric materials used in the cask, they are:

Borated Polyester Resin	(radial neutron shield)
Polypropylene	(top neutron shield)
Viton	(protective cover O-ring)

All of these materials are either encapsulated when in the SFP water or are used outside the pool environment.

**Lubricants** used during assembly of the TN-40 cask are:

- Bolt lubricant
- WD-40

The bolt lubricant is applied to the threads and shoulders of the bolts used for the

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closure parts. The bolts' descriptions are below:

<u>Description</u>	<u>Qty</u>	<u>Size</u>
Lid closure	48	1-1/2"
Vent cover	8	3/4"
Drain cover	8	3/4"
Neutron shield drum	4	1-1/4"
Over pressure (OP) port cover	4	1/2"
Helium OP tank	4	1/2"
Weather cover	12	1-1/4"
Basket rails	48	1"

None of the closure bolts are immersed in the spent fuel pool water. The bolt lubricant applied to the closure bolts during shop assembly is removed from the mating holes as part of receipt inspection. However, some residue remains due to the nature of the lubricant and geometry of the bolt holes. PINGP currently uses both Never-Seez and FelPro N-5000 for lubricant on the TN-40 cask. Both lubricants are approved for use in the PINGP SFP environment. Only the basket rail bolts are immersed in the SFP water. They are installed during fabrication and are not removed during any of the cask operation.

Bolt lubricant is also used on the trunnions during some handling operations outside the SFP. This lubricant is removed completely before immersion in the spent fuel pool water.

When touching up the sealing surface during receipt inspection an emery block and aluminum oxide cloth is used in small, local areas. A light coating of demin water or WD-40 is occasionally applied to provide a lubricant on the seal surface. All the seal surfaces are cleaned following any touch up and prior to immersion in the SFP for loading.

**Cleaning agents** used onsite include:

- Demin water
- Radiac wash
- Syntech "touch-it-up"
- Acetone

The demin water is used to rinse the basket and cask's exposed surfaces during receipt inspection. It is also used during spent fuel pool operations on the cask exterior. Cleanliness of the cask and each basket cell is checked during receipt. A foreign material exclusion cover is kept in place over the cask when the lid is removed.

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Radiac wash and Syntech are both used for cask exterior decontamination. Syntech is typically used to spot clean any areas during receipt activities. Both are approved products for decontamination processes at Prairie Island.

Acetone has been used to clean the stainless steel sealing surfaces for the lid, vent and drain covers. Less than one pint is estimated in use on each cask. The acetone is applied with a clean rag and readily evaporates. Acetone is an approved chemical for use in PINGP's SFP environment.

**Normal cask operations** are controlled by procedures developed for the TN-40. A brief narrative of the procedures is below. The procedures are broken down into receipt inspection, cask loading, cask decontamination and storage.

The fuel storage operation commences with the cask being brought into the Auxiliary Building rail bay through the west roll-up door. The weather cover and lid of the storage cask are removed and the cask inspected. A lifting yoke is attached to the cask and connected to the Auxiliary Building crane hook. The cask is lifted by the crane from the base mat, laterally transferred and aligned with the access door to the fuel pool area, moved above the loading and unloading area of Pool Number 121 and lowered to the surface of the pool.

The cask is then filled with SFP pool water and the cask then lowered into the spent fuel pool. Spent fuel assemblies are loaded into the cask.

After the cask is loaded with spent fuel, the lid is placed on the cask, the cask is lifted to the pool surface and several lid bolts installed. The cask's internal fuel cavity is drained by displacing the water with air or with a suitable drain pump.

The cask is then returned to the Auxiliary Building rail bay by retracing the load path above. The remaining lid bolts are installed and torqued. The cask exterior is decontaminated in the cask decontamination area and dried internally by using a vacuum system. The cavity is filled with helium to the operating pressure and the cask lid seal is leak tested. The cask is positioned in the transport vehicle and the top neutron shield is installed on the lid. The overpressure monitoring system is installed, and the interspaces between the double metallic seals pressurized to the operating pressure. Prior to transfer from the Auxiliary Building to the ISFSI, the cask externals are surveyed for contamination, temperature, radiation dose rates and the proper functioning of the seal tightness monitoring system, and the protective cover is installed.

The transport vehicle is pulled to the ISFSI site by a tow vehicle. The cask is set on its storage position. The cask is finally connected to the cask seal monitoring system and a functional check of the monitoring system performed.

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## Environment:

In order to review cask environments, the following section is separated into the cask interior and exterior.

## Cask Interior:

The cask interior is exposed to three separate environments, during cask operations, which are reviewed below. This includes immersed in SFP water, vacuum drying and helium gas environment.

During loading and unloading, the interior is flooded with SFP water and the cask is immersed in the SFP. The ISFSI Technical Specifications require a dissolved boron concentration of 1800 ppm minimum (T.S. 3.2.1), in the SFP. Typical SFP boron concentration at Prairie Island is greater than 3000 ppm at the present time. Materials identified on the interior of the cask include the basket assembly, Zn/Al metallic coating and basket rails.

Potential sources of chemical or galvanic reactions are the interaction between the aluminum, borated aluminum and stainless steel within the basket itself, and the interaction of the zinc/aluminum spray on the cask cavity wall and the borated water. Stainless steel is used extensively in the spent fuel pool, and aluminum has become more prevalent in fuel pool environments as the Boral is used as a poison material in many current rack designs.

## Behavior of Aluminum in Borated Water

Transnuclear provides a detailed discussion on the corrosion properties of aluminum within their September 10, 1996 response to Bulletin 96-04 for the TN-40 cask. Their discussion is based on Brooks and Perkins Report #624, "Product Performance Report". The reader is referred to the Brooks and Perkins Report or Transnuclear's September 10, 1996 submittal for further detail.

In addition to the above references literature, NSP performed a lab analysis of aluminum alloy 6061 in a solution of 3500 ppm boron. (This is considered to be a maximum boron concentration in the SFP at Prairie Island.) (See Attachment 2). The report summarizes no evidence of effervescence over a 192 hour period, and only trace amounts of hydrogen gas was detected in the observed gas volume. This lab analysis is additional confirmation of the ability of the aluminum to resist corrosion from the boron solution found in SFP environments.

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### Behavior of Austenitic Stainless Steel in Borated Water

The basket cell plates and plugs are made from Type 304 stainless steel. In addition, the gasket sealing surfaces are stainless steel clad.

Stainless steel has been used in spent fuel pools for storage racks for over two decades. In addition, stainless steel is used as the liner in the spent fuel storage pools almost universally in this country. The boric acid does not affect the 304 stainless steel.

### Behavior of Boral in Borated Water

AAR Advanced Structures has published literature defining the corrosion resistance of their product. The AAR summary follows.

The general corrosion behavior of Boral does not differ appreciably from that of Type 1100 aluminum, which shows excellent corrosion resistance in the environments encountered in fuel storage applications. When exposed to water, aluminum reacts to form an impervious, tightly adhering layer of hydrated aluminum oxide, which protects the surface from further attack. Galvanic corrosion (with the couplet formed by the aluminum of the Boral and the stainless steel structural material) is not a problem, for stainless steel is compatible with aluminum in all but severe marine or high chloride environmental conditions. Pitting of aluminum can occur in areas where the material is in contact with stainless steel, particularly if pockets or crevices out of the main fluid stream permit the build up of dissolved salts, metal ions, oxygen or other gases. Such pitting or crevice corrosion is avoided by proper application of the Boral, in this case, by utilizing the vented concept and its provisions for pool water flow past the Boral panels.

NSP performed a lab analysis of the Boral product in a solution of 3500 ppm boron. (See Attachment 3). The testing showed no evidence of effervescence over a 192 hour period. The hydrogen detected in the gas sample translated to a generation rate that could be as much as 1.04 ml/hr/ft<sup>2</sup>. This value is attributed to the exposed edge of the Boral. Due to the small surface area of the Boral edge (< 20 ft<sup>2</sup>), along with the small generation rate, the possible generation of hydrogen gas from Boral in the cask is not capable of accumulating at hazardous amounts during the time that the lid is on and the interior is in the process of being drained. Given that the rate of hydrogen generation is less than 1/5 of that from the Zn/Al and the surface area is 1/15 of the Zn/Al, as shown below, the potential for hydrogen generation is a fraction of that which may be generated from the Zn/Al and is therefore not considered further.

### Behavior of Zn/Al Coating in Borated Water

NSP performed a lab analysis of a Zn/Al chip in a solution of 3500 ppm boron. (See Attachment 4). The testing showed no evidence of effervescence over a 73 hour

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period. The hydrogen generation rate could be as much as 5.7 ml/hr/ft<sup>2</sup>. The Zn/Al surface area on the inside of the cask is nominally 330 ft<sup>2</sup>. This correlates to about 1.7 l/hr of hydrogen generated in the cask environment when filled with SFP water.

### Operational Procedures:

Operational procedures for the TN-40 prevent any significant concentration of hydrogen from accumulating. The cask interior is water solid with the lid in place, until the cask is raised to the pool surface and drain operations begin. Conservatively assuming 8 hours for drain operations, and further assuming no open vent path in the lid, a peak hydrogen concentration of 0.5% is reached at the initiation of draining. As draining continues, the air volume increases in the cask and the hydrogen concentration is shown to approach 0.25% after 30 minutes. Two additional conservatisms in the above model assume the peak hydrogen generation rate throughout the draining process and, all the soluble hydrogen is liberated from solution as a gas.

Using the above model as a conservative basis, it is evident that the generation of hydrogen will not approach the 4% flammable limit. Three reasons for this include:

- 1) The reaction of the cask interior material with the SFP water does not generate significant amounts of hydrogen.
- 2) The cask is drained at the SFP surface, prior to removal from the SFP.
- 3) The cask's lid vent is maintained open during the lid placement and drain operations.

During unloading, item (2) above is not applicable. The cask would be backfilled, until solid with SFP water. Continuous venting of any gases along with steam would be permitted through the vent opening. This is controlled by plant procedures and as shown in item (1) above, the generation of any hydrogen gas is not significant enough to accumulate in flammable concentrations.

During loading and handling, the interior is vacuum dried. The cask interior is reduced in pressure to 8 mbars. During testing of the Transnuclear TN-24P storage cask, it was shown there is no change in the temperature of the cavity wall between the vacuum and helium runs. EPRI Report NP-5128 (1987) shows axial temperature profiles for vertical casks (TN-24P) under vacuum and helium conditions. This confirms there is no change in  $\Delta T$  across the cask body, only a change in  $\Delta T$  across the cavity due to a change in cavity gas medium. Therefore, the Zn/Al coating is stable, with respect to temperature, during the drying process and is not susceptible to reaching it's vapor pressure.

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During storage, the cask interior is pressurized with 20 psia helium and sealed. The helium cover gas is assumed present for the thermal analysis performed for the TN-40. Also, the use of an inert gas (helium) ensures long term maintenance of fuel clad integrity. The helium environment does not support the occurrence of chemical or galvanic reactions, because moisture must be present for a galvanic reaction to occur. The cask is thoroughly dried before storage by a vacuum drying process and backfilled with helium, thus preventing corrosion. Therefore, no degradation of the cask interior is postulated during the storage phase.

### Cask Exterior:

The cask exterior is exposed to two environments during cask operation. It is immersed in the SFP during loading and unloading and exposed to the local climate during storage.

The outer shell, which surrounds the radial neutron shielding is SA516. The trunnions are SA-105 carbon steel. The exterior of the casks, with the exception of the trunnion bearing surfaces, are coated with the Zn/Al metallic spray. This in turn is painted using an epoxy primer and top coat. The paint which is used to date on the TN-40 casks is a polyamide epoxy enamel. It is recommended for use as a topcoat for steel surfaces subject to radiation and decontamination, and is used extensively in nuclear power plants. The paint has been qualified for use in the spent fuel pool, exhibiting no leaching and is easily decontaminated.

The paint is visually inspected prior to immersion of the cask in the spent fuel pool and periodically during storage. Touch-up painting is performed, if the paint surface is damaged.

Since the cask is not immersed in the SFP for any significant amount of time, there will be no deterioration of the cask metal components during normal operations. During storage, the cask surface is subject to the local climate. All exterior surfaces are painted, including the trunnion bearing surfaces, while stored on the ISFSI pad. The casks are visually inspected to determine if any damage or deterioration of the exterior of the cask has occurred. Any gross deterioration of the paint would be touched-up, as required. The Zn/Al metallic coating provides a uniform protective layer for the cask's metal components during normal operations and therefore no deterioration of the cask's exterior is anticipated during the short amount of time the cask is immersed in the SFP.

**Off-normal operations** are defined in Section 8.1 of the SAR. Of the various types of off-normal events defined in ANSI/ANS 57.9, only the loss of external power supply for a limited duration is considered applicable and credible to PINGP's ISFSI operations.

Loss of power at the ISFSI location has no affect on material compatibility and introduces no new set of conditions relating to this bulletin.

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**Accident conditions** are outlined in Section 8.2 of the SAR. The events are listed below.

<u>Accident</u>	<u>SAR Section</u>
Earthquake	8.2.1
Extreme Wind	8.2.2
Flood	8.2.3
Explosion	8.2.4
Fire	8.2.5
Inadvertent Loading of a Newly Discharged Fuel Assembly	8.2.6
Cask Seal Leakage	8.2.7
Hypothetical Cask Drop & Tipping	8.2.8
Loss of Confinement Barrier	8.2.9

The first five accident scenarios (earthquake, extreme wind, flood, explosion, & fire) are all external to the cask. There are no new activities or reactions with the cask material, contents or environment that impact the SAR analysis. The results of these events are not affected because there are no postulated reductions in the strength of a cask's structural components or increased loading stress from any of the described materials, cask content or other reactions.

### Inadvertent Loading of a Newly Discharged Fuel Assembly

The possibility of a spent fuel assembly, with a heat generation rate greater than 0.675 kW, being erroneously selected for storage in a cask has been considered. This event is not considered credible, as evaluated in the SAR, due to the multiple administrative controls. No reactions with the cask material, contents or environment impact this SAR analysis. There is no postulated reduction in the strength of any cask structural component or increased loading stresses from any of the described materials, chemicals, or other reactions.

### Cask Seal Leakage

The double metallic cask seal is monitored for leakage using a helium overpressure system. No credible mechanisms that could result in a leakage of radioactive products were identified in the SAR analysis. No reactions with the cask material, contents or environment impact this SAR analysis. There is no postulated reduction in the strength of any cask structural component or increased loading stresses from any of the described materials, chemicals, or other reactions.

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## Hypothetical Cask Drop and Tipping

These events involve the handling of the cask using the upper trunnions (cask drop) and storage while on the concrete pad (tipping). No reactions with the cask material, contents or environment impact this SAR analysis. No postulated reductions in the strength of any cask structural component or increased loading stresses occur from any of the described materials, chemicals, or other reactions.

## Loss of Confinement Barrier

This event was evaluated in the SAR and viewed as not credible. However as a hypothetical scenario the accident evaluated assumed failure of all fuel cladding, release of captured gas within each pellet and a breach of the confinement barrier of the TN-40 on the concrete pad. None of the activities that result from the previously described material, cask content, or environment compatibility concerns impact this SAR analysis. No precipitates have been discovered in tests performed by NSP using credible cask operating conditions. Any release of possible precipitates formed as a result of cask loading is expected to be minor and this SAR analysis is bounding and unaffected. Therefore, no reactions with the cask material, contents or environment impact this SAR analysis.

## Conclusion:

In summary, the materials of the TN-40 cask have been reviewed to determine whether chemical, galvanic or other reactions among the materials, contents and environment might occur during any phase of loading, unloading, handling and storage.

During loading and unloading, the casks are submerged in borated water. This affects the interior and exterior surfaces of the cask body, lid and the basket. The protective cover, the top neutron shield, and the overpressure system are not submerged in the spent fuel pool.

During handling and storage, the exterior of the casks are exposed to normal environmental conditions of temperature, rain, snow, etc. All of the exterior surfaces, with the exception of stainless steel components and trunnion bearing surfaces, are protected from environmental exposure by the Zn/Al metallic spray. The paint is touched up periodically, if there are any areas which peel or otherwise deteriorate. Therefore, the cask exterior is protected from chemical, galvanic or other reactions during storage.

During storage, the interior environment of the cask is exposed to an inert helium environment. The helium environment does not support the occurrence of chemical or galvanic reactions because moisture must be present for a reaction to occur. The cask is thoroughly dried before storage by a vacuum drying process. It is then sealed and backfilled with helium, thus preventing corrosion. Since the cask is vacuum dried,

## Final Response to NRC Bulletin 96-04

galvanic corrosion is also precluded since there is no water present at the point of contact between dissimilar metals.

The neutron shielding materials and the aluminum resin boxes are sealed during all normal operations. The cured resin material is inert and does not affect the aluminum boxes or the carbon steel housing.

### Requested Actions:

1. (b) Evaluate the effects of any identified reactions to determine if any adverse conditions could result during cask operations, including loading and unloading. Consideration should be given, but not limited, to:
  - (i) generation of flammable or explosive quantities of hydrogen or other combustible gases; and
  - (ii) increased neutron multiplication in the fuel in a cask because of boron precipitation from a chemical reaction among the borated water and cask materials.

### Response to Requested Action 1 (b) (i) and (ii):

In response to 1(b)(i), the potential for the generation of hydrogen exists when the basket and interior surfaces of the cask are in contact with the SFP water. Any generation of hydrogen would cease when there is no water in contact with the cask internals. It is expected that the exposed aluminum surfaces of the cask internals will become inactive due to the development of a protective oxide layer, in which case the generation of hydrogen would be greatly diminished or halted.

During unloading of a TN-40 cask, hydrogen could be generated when the cask is backfilled with SFP water and while set in the SFP cask load/unload area of pool #121.

During cask unloading, SFP water is pumped through the lid's drain opening. The lid's vent penetration is kept open to allow for removal of steam, helium and any hydrogen gas that may be generated. The cask interior surfaces are at a higher temperature initially, when compared to the loading sequence. Other parameters remain unchanged. The small surface area initially exposed to the borated water during re-filling, along with the small generation rates and previous passivation due to the development of a protective oxide layer is not expected to yield a hazardous concentration of hydrogen. Any concentration of gases generated would be continuously vented by displacement from the borated water. Filling will stop when a solid stream of water is present at the vent opening.

## Final Response to NRC Bulletin 96-04

Hydrogen would be generated from radiolysis when fuel is in the cask with the SFP water. The characteristics of the fuel to be loaded in the TN-40 cask are expected to produce a negligible amount of hydrogen by radiolysis. The spent fuel permitted for storage is a minimum of 10 years discharged from the reactor, with a maximum initial enrichment of 3.85 weight percent U-235, and an average burnup not exceeding 45,000 MW-days.

The only time the TN-40 cask is configured to accumulate any gas is when the lid is in place. Combustible gases such as hydrogen require a sufficient concentration of flammable gas to accumulate and an ignition source to be present, in order to ignite. The minimum volume of hydrogen in air for ignition is 4%. Once the cask is raised to the pool's surface, the cask is drained. At all times when SFP water is present in the cask, the lid's vent penetration is open. The lid is a bolted closure and no active ignition source is used on the cask during any phase of loading and unloading.

The small amount of hydrogen which may be generated does not result in a safety hazard during normal operations for the TN-40 cask.

In response to 1(b)(ii), the design of the TN-40 cask is intended to maintain subcriticality as described in SAR paragraph 3.3.4.1 (Control Methods for Prevention of Criticality). There are four design/licensing features to limit the effective neutron multiplication factor  $k_{eff}$  to less than 0.95. They are:

1. Incorporation of neutron absorbing material (boron) in the basket material.
2. The Spent Fuel Pool boron concentration is at least 1800 ppm boron whenever a TN-40 cask is being loaded/unloaded.
3. The weather cover, containment seals and seal pressurization system prevent water from entering the loaded cask.
4. The maximum fuel enrichment is 3.86 w/o U-235.

The SAR (Table 3.3-4) also addresses the variation of the effective multiplication factor as the water level in the cask cavity is lowered during draining. This assumes fresh fuel is loaded in the cask. The SAR (Table 3.3-5) presents the variation of the effective multiplication factor for various water densities (without boron) in the entire cask cavity. The calculation was performed using 1.9 weight percent U-235 to approximate irradiated fuel placed in the cask. When the water density reaches unity, the cask cavity is filled with pure water and the value of  $k_{eff}$  is  $0.9306 \pm .0036$ . This calculation is not considered as a limiting analysis since the SAR does not seek relief from the requirement for 1800 ppm boron in the SFP water. However, it is reflective of the large margin of safety incorporated in the design of the TN-40 for maintaining subcriticality. In both cases the maximum effective multiplication factor is less than 0.95.

## Final Response to NRC Bulletin 96-04

To maintain margin above the ISFSI Technical Specification limit, the procedures used for cask loading at PINGP require the minimum boron concentration in the SFP to be 2280 ppm. Actual boron concentration in the PINGP SFP has been greater than 3000 ppm during cask loading operations.

It is clear that there is a large margin in the design features and procedural controls to compensate for possible boron loss in the SFP due to boron precipitation. While no precipitate has been discovered during the loading of the TN-40, we committed in NSP's 8/19/96 response to Bulletin 96-04, to sample the drain discharge of the next cask loaded at PINGP to determine if there is a loss of boron. In response to that commitment, NSP sampled the drain discharge of the fifth (#05) cask, recently loaded at PINGP, to determine if there is a loss of boron. Three (3) samples were taken from the cask during draining operations, one sample each, at the start, middle and end of draining. No changes in the cask procedures were necessary to gather the drain sample(s). The results of the sampling are presented below.

The cask was first flooded with SFP water during cask placement in the SFP. Initial boron concentration was 3340 ppm. After the cask was loaded with 40 spent fuel assemblies, the lid was placed on the cask and the cask was raised to the surface of the SFP to begin drain operations. Approximately 48 hours had elapsed from the time SFP water was first introduced in the cask, until draining was initiated.

The boron analysis results of the three drain samples indicated:

<u>Drain Time Frame</u>	<u>PPM Boron</u>
Start	3345
Middle	3347
End	3343

Variation in the boron analysis is within the accuracy of the analysis method (boron by NaOH titration). Therefore, no definitive change in boron concentration was observed and any variation (less than 1%) is attributed to instrument accuracy.

### Conclusion:

No detectable boron loss was detected after the recent cask loading and, since no chemical, galvanic or other reactions have been identified in the TN-40 cask, no significant precipitate involving a reaction with the dissolved boron in the SFP is expected during normal cask operations. Therefore, no increased neutron multiplication will occur in the cask.

In response to other considerations in 1(b), no "other" adverse conditions are known to result due to the reactions identified and described in part (a) above.

# Final Response to NRC Bulletin 96-04

## Requested Actions:

1. (c) **Review current cask operating procedures to determine if adequate controls and procedures are in place to minimize hazardous conditions that may be created by any identified reactions.**

## Response to Requested Action 1 (c):

All cask receipt, handling, loading and transfer operations are administered by plant procedures. These actions were developed for the handling and loading of the first cask and remain in place as part of the cask operating procedures. The actions described may not have been developed primarily for minimizing the hazardous conditions outlined in the subject bulletin, but they do provide the benefit of minimizing the identified reactions. NSP performs the following actions during all TN-40 cask loadings:

1. During shipment of the cask from the fabricator, the cask is sealed with a cover gas of helium on the inside. The exterior of the cask is wrapped in a protective tarpaulin while in transit.
2. Upon receipt, the cask weather cover, neutron shield drum and lid are disassembled. The cask and basket are rinsed using demin water, and the water is pumped out following the rinse. The exterior of the components that will be immersed in the SFP water are inspected for imperfections in the paint surface. Areas that need repair are repainted or sealed with RTV. Imperfections as small as pin holes in the paint surface are treated in this manner to aid in the final decontamination of the cask after removal from the pool.
3. All bolt holes are brushed clean and vacuumed to remove any foreign material. The lid bolt holes are chased with a lid bolt to verify the hole threads are clear of all obstructions.
4. All the basket cells (fuel and non-fuel) are inspected to assure that no debris is settled in the bottom of the cask. Following this inspection a foreign material exclusion (FME) cover is placed over the cask. This remains in place until the cask is raised to the SFP for loading.
5. A project engineer familiar with cask procedures and design, is assigned to work with the plant personnel during cask activities. Additionally, the Quality group routinely provides QA and QC oversight during cask activities.
6. Spent fuel pool temperature is monitored prior to placing a cask in the pool. A maximum temperature limit of 110<sup>0</sup> F is listed as a prerequisite in the procedures. Since any hydrogen generation would have an increased rate at higher temperatures, minimizing the temperature is beneficial.

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The vent penetration through the lid is left open when the cask is removed from the SFP water. The purpose has been to allow for a direct vent path when draining operations begin. A protective layer of griff tape has been placed over the vent seal area to prevent minor scratches or marring during handling operations. In NSP's 8/19/96 response to Bulletin 96-04, we committed to revise procedures to allow for an opening in the griff tape where it passes over the vent opening. This will allow a clear vent path from the cask internals once the lid is in place. It will also continue to function as a protective layer on the vent seal surface. NSP has implemented the necessary procedure revisions and this commitment is complete.

The sequence of operation for the TN-40 provides for draining the cask after it is raised to the surface of the pool. Draining the cask at the pool surface removes most of the water in contact with the cask internals, further reducing the possibility hydrogen gas generation. Any residual water remaining after the initial draining, is removed by the vacuum drying system.

No active ignition source is present which could cause burning of any hydrogen that may be generated and released as a gas. The lid closure on the TN-40 is bolted with forty-eight 1-1/2" bolts. These bolts are torqued in place following placement of the cask in the decon area.

A double metallic seal is maintained that separates the bolts, bolt holes and lubricant from the cask internals. Any possible reaction between the thread lubricant and the SFP water is not of concern with regard to collection of gas under the lid. While it is expected that the thin film of compound that remains on the bolt holes after cleaning would not be sufficient to provide for significant reaction, the bolt holes and bolts are not in enclosed volumes, and are separated from the confinement boundary.

Following the vacuum drying of the cask internals, the cask is backfilled with helium to provide an inert environment. The cask lid confinement and penetrations use a double barrier seal system. To preclude leakage of air into the cask, the cask cavity is pressurized above atmospheric pressure with helium. The interspace between the metallic O-rings in the cask lid confinement and lid penetration seals is monitored and connected to a helium filled tank that sits atop the steel encased polypropylene drum that is bolted to the cask lid confinement. The tank pressure is maintained above the cask cavity pressure, to ensure that any seal leakage would be into rather than out of the cavity.

All the above stated actions serve to limit or preclude the generation and collection of gases that may be present during the loading sequence of the cask.

During the unloading sequence, the internal atmosphere of the TN-40 is sampled prior to removing the lid, to determine if there is indication of failed fuel. In NSP's 8/19/96 response to Bulletin 96-04, we committed to revise the cask unloading procedure to

## Final Response to NRC Bulletin 96-04

include a check for flammable gases. Should flammable gases be found in the TN-40 in a concentration greater than 10% of the lower explosive limit by volume, unloading operations will not continue until a review is performed by plant management. NSP has implemented the procedure revision and this commitment is complete.

It should be noted that no active ignition source is utilized during the unloading process of the TN-40. During reintroduction of the SFP water, the vent penetration is open to allow for the removal of steam, helium and other gases that may be present as previously determined with the gas sample. The cask refilling process takes place while at the surface of the SFP and is enclosed within the pool's special vent zone. Once the cask is water solid the cask is lowered in the SFP and the lid assembly is removed.

Boron concentration margins for unloading are the same as described for loading. Any boron reduction in the cask during an unloading process will be insignificant as discussed above.

### Requested Actions:

1. (d) Evaluate the effects of any identified reactions to determine if their reaction products could reduce the overall integrity of the cask or its contents during storage or transportation. Determine if the reaction products could adversely affect the cask ability to maintain the structural integrity and retrieveability of the spent fuel throughout the term of the license or to transport fuel safely. Consideration should be given, but not limited, to:
  - (i) changes in cask and fuel cladding thermal properties, such as emissivity;
  - (ii) binding of mechanical surfaces, especially fuel-to-basket clearances; and
  - (iii) degradation of any safety components, either caused directly by the effects of the reactions, or by the effects of the reactions combined with the effects of long-term exposure of the materials to neutron and gamma radiation, high temperatures, or other possible conditions.

### Response to Requested Action 1 (d) (i), (ii) and (iii):

As discussed in the response to part (a), there are no significant reactions which could reduce the overall integrity of the cask or its contents during storage. The cask and

## Final Response to NRC Bulletin 96-04

Fuel cladding thermal properties are provided in the SAR. The emissivity of the fuel compartment is 0.3, which is typical for non-polished stainless steel surfaces. If the stainless steel is oxidized, this value would increase, improving heat transfer. The fuel rod emissivity value used is 0.8, which is a typical value for oxidized zircaloy. Therefore, the passivation reactions would not reduce the thermal properties of the component cask materials or the fuel cladding.

There are no reactions which would cause binding of mechanical surfaces of the fuel to basket clearances due to galvanic or chemical reactions. There is no significant degradation of any safety components caused directly by the effects of the reactions or by the effects of the reactions combined with the effects of long term exposure of the materials to neutron or gamma radiation, high temperatures, or other possible conditions.

NSP has performed tests on the aluminum, boral and zinc/aluminum metallic spray all of which are present in the cask's interior. This is in addition to the published data from AAR Advanced Structures the supplier of the boral. Results from the NSP tests of cask materials subjected to the borated water indicate no precipitate is generated that would contribute to changes in thermal properties or binding of mechanical surfaces when exposed to the SFP environment. The chemist report on the performance of the above listed cask materials in a borated water environment are included in attachments 2, 3, and 4.

### Requested Actions:

- 2. For storage casks currently loaded with spent fuel, determine the extent, if any, of the chemical, galvanic, or other reactions that have occurred, and the effect of these reactions on the cask ability to maintain the structural integrity and retrieveability of the spent fuel throughout the term of the license.**

### Response to Requested Action 2:

NSP currently has five casks loaded at the PINGP site. During cask loading, the fuel arrangement is videotaped with an underwater camera just above the top nozzles. A review of two casks' tapes has revealed good water clarity with no indication of gas generation and subsequent release of gas bubbles while the cask is loaded with fuel. The film footage is typically taken from 1 to 10 hours after fuel transfer to the TN-40. With the completion of NSP's lab analysis, test results show no gas effervescence, no formation of precipitate under credible cask operating conditions, and no significant generation rate of hydrogen gas. No effervescence has been present for cask loadings in the past based on video footage and discussions with project personnel. The recent loading of cask #05 revealed no gas bubbles originating from the cask's interior or exterior surfaces.

## Final Response to NRC Bulletin 96-04

Once loaded, the cask internals are kept in an inert environment by use of a helium cover gas. This provides a stable environment that removes the potential for later reactions. The cask external surfaces are protected from local climate effects and periodically monitored for damage or deterioration to assure continued design performance.

The response to item 1 contains detailed information that demonstrates none of the identified reactions will have an adverse effect on the structural integrity or retrievability of the spent fuel in the TN-40 casks.

**Attachment 2**

**Aluminum Metal Interaction With Boric Acid Solution Test Report**

*Testing Laboratory* Internal Correspondence

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**Date** September 12, 1996

**To** Mark Mc Keown

**Location** Prairie Island

**From** Tim Fair

**Location** CSC-2

**Subject** Aluminum metal interaction with Boric Acid Solution.

**Purpose**

A sample of Aluminum metal, reported to be alloy 6061 from P.O. PH-6932, was evaluated in the presence of Boric Acid to determine the rate of generation and identity of any gas evolved.

**Sample**

The sample was received on August 23, 1996 and was given lab number D8758. The sample was a flat rectangular piece of metal. A rectangular piece 1.8098 inches long by .5535 inches wide and .0626 inches thick was cut from the sample using a shears. Results of the evaluation are presented below.

**Test Methods**

The sample was weighed using a Mettler AE163 Balance (B63490).

Next, the surface area was determined by measuring the length, width, and thickness of the sample using a digital micrometer. The surface area of the Aluminum (including edges) was thus determined to be 2.30 square inches.

**Preparation of the Boric Acid Solution:**

A 3500 PPM solution of Boron from Boric Acid ( $H_3BO_3$ ) crystals (Guaranteed Reagent EM BX0865-3) was prepared in water (Ultra Resi-Analyzed J.T. Baker lot J32265) as follows. Using a Mettler AE200 (J91086) Balance, 5.0012 grams of  $H_3BO_3$  (Mol wt. = 61.83) was placed into a 250 ml class A volumetric flask. Approximately 200 ml of water was added. Solution of the acid crystals was accomplished in about five minutes with mild hand agitation. The flask was filled to the mark to yield a solution of 3500 PPM Boron. N.B. This solution would provide a slightly higher acid concentration than reported in use (3300 PPM). Therefore, this represents a more conservative situation for this evaluation.

### Test Performance:

The flat metal sample was placed in a centrifuge tube (Kimax 45241) which is graduated to 100 mls. The total volume of the solution in the tube, used for this evaluation was 111 mls. The Boric Acid solution was added to the centrifuge tube until it slightly over filled the tube. A one hole, black rubber stopper with approximately 24 inches of small diameter plastic tubing protruding from the hole, was used to plug the opening in the centrifuge tube. Liquid from the centrifuge tube filled the plastic tubing and no visible gas volume was present in the centrifuge tube prior to immersion in the constant temperature bath.

The centrifuge tube was inverted with the pointed end up, and secured in a constant temperature bath at 110 degrees Fahrenheit. The bath temperature was monitored using an ASTM 3F thermometer (08604). Readings of time, temperature, barometric pressure and volume were recorded manually from 7:30 August 26, 1996, until 6:30 September 3, 1996 (see Table One). The sample was removed from the tube, dried, and weighed.

Within the first few minutes, as the Boric Acid liquid expanded due to the increased temperature, some liquid leakage from the plastic pressure equalization tube was noted. This decreased over time and finally stopped when the liquid reached the temperature of the bath. At about this same time, a small gas volume was noted to have formed at the apex of the centrifuge tube. Release of dissolved gas (Air) from within the Boric acid is the most likely explanation. During the test, no bubbles were noticed on the surface of the sample.

At the conclusion of the evaluation, additional tests on the gas evolved and the ion concentration in the acid solution were also run. The identity of gasses present in the volume above the acid solution were determined using Gas Chromatography (GC) method (SP G01). This same method allows for the determination of the amount of these gasses present. An aliquot of the acid solution was tested using Inductively Coupled Plasma (ICP) methods to determine the amount of Aluminum dissolved in the solution.

### Test Results

Table one presents the raw data as transcribed from the lab worksheet. The table also includes calculations for correcting the observed gas volumes. The corrections take into account changes in barometric pressure, the partial pressure of water vapor and temperature. A graph of this corrected data, over time, is also included.

About .4 mls of gas was generated in 192 hours from a surface area of 2.30 square inches of Aluminum. This equates to about .0008 mls of gas per hour per square inch of Aluminum.

Gas Chromatography results (see included worksheets) show that about .18% of the gas that was collected (recovery of 96%) is Hydrogen.

Weight loss from the sample was .0002 grams from a 2.9414 gram starting weight or a .006% loss in 192 hours.

Dissolved material from the sample in the Boric Acid solution (.0002 grams in 111 mls) was calculated to be about 1.8 PPM.

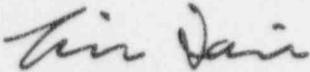
ICP analysis of the Boric Acid solution showed trace evidence of Aluminum ion in solution (.45 PPM in solution with a 101% recovery. After subtracting blank values, only Zinc (2.70 PPM) was detected at a level greater than 1 PPM (Cr, Cu, Fe, Pb, Mg, Mo, Ni, Mn all less than 1 PPM).

## Testing Laboratory Internal Correspondence

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### Conclusions/Recommendations

No evidence of effervescence from the sample was noticed. Metal from sample D8697 in contact with 3500 PPM Boric Acid at 110 degrees generates some small amount of Hydrogen. It could be as much as .13 mls per hour per square foot of Aluminum (total volume as Hydrogen). However, since only .18% of the gas formed was determined to be Hydrogen, only .00023 mls per hour per square foot may be generated. Aluminum was detected at less than 1 PPM in the Boric Acid solution after 192 hours at 110 degrees Fahrenheit.



Tim Fair

Testing Laboratory Team Leader

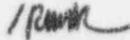
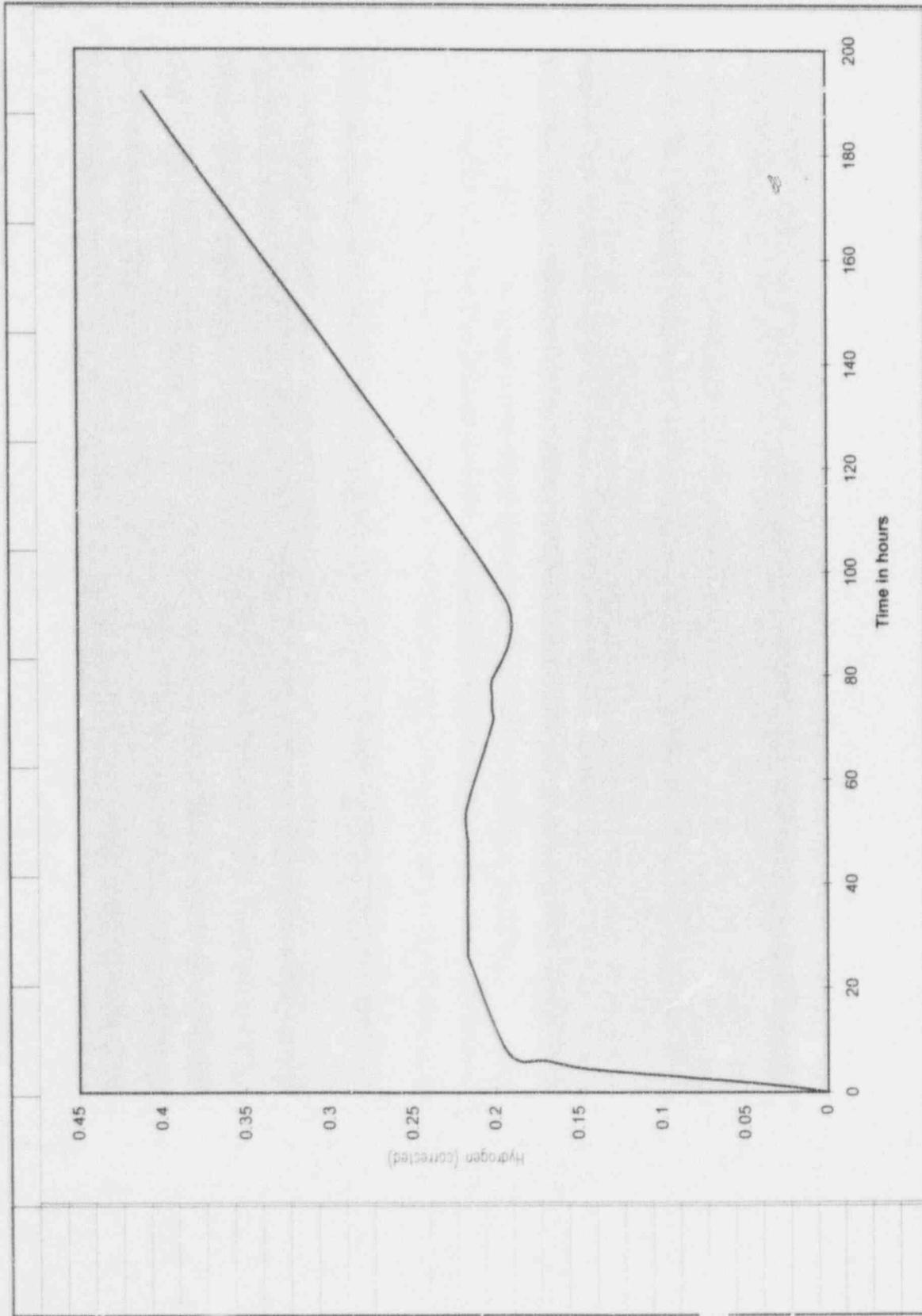


TABLE ONE

Volume	Pressure	Water Vapor Corrected Pressure	Standard Pressure Correction	Standard Temperature Correction	Time (hours)	Corrected Volume
					0	0
0.03	741.6	678	0.89210526	0.863924051	1	0.02312134
0.07	741.2	677.6	0.89157895	0.863924051	2	0.05391795
0.13	741.3	677.7	0.89171053	0.863924051	3.25	0.10014812
0.19	742.1	678.5	0.89276316	0.863924051	4.5	0.14654312
0.22	742.1	678.5	0.89276316	0.863924051	6	0.1696815
0.25	742.4	678.8	0.89315785	0.863924051	7.5	0.19290515
0.28	742.8	679.2	0.89368421	0.863924051	25.25	0.21618108
0.28	745.2	681.6	0.89684211	0.863924051	26	0.21694497
0.28	745.1	681.5	0.89671053	0.863924051	28	0.21691314
0.28	745.3	681.7	0.89697368	0.863924051	32	0.2169768
0.28	744.4	680.8	0.89578947	0.863924051	47.5	0.21669034
0.28	745.7	682.1	0.8975	0.863924051	49	0.21710411
0.28	746.4	682.8	0.89842105	0.863924051	54.5	0.21732692
0.26	745.4	681.8	0.89710526	0.863924051	71	0.20150801
0.26	746.4	682.8	0.89842105	0.863924051	73	0.20180356
0.26	746.7	683.1	0.89881579	0.863924051	74	0.20189223
0.26	746.9	683.3	0.89907895	0.863924051	76	0.20195134
0.26	747.1	683.5	0.89934211	0.863924051	79	0.20201045
0.25	746.7	683.1	0.89881579	0.863924051	95	0.19412714
0.5	740	676.4	0.89	0.922297297	192	0.4104223

Graph 3



# Gas in Oil Analysis Sheet Special Projects Lab



Run #: 2

Date Tested: 9-3-96

## Equipment Data

Location: "D 8758"

Ambient Temp: \_\_\_\_\_

Serial Number: \_\_\_\_\_

Wind Temp: \_\_\_\_\_

Trans Number: \_\_\_\_\_

Oil Temp: \_\_\_\_\_

Date Sampled: 9-3-96

Sample Vol. Inj. 0.3 ml.

ml of gas: \_\_\_\_\_ ml of oil: \_\_\_\_\_

Std. Vol. Inj. 1.1 ml.

$$CF = \frac{1.1}{0.3} = 3.67$$

## Analysis Data

### Inorganic:

Oxygen, O<sub>2</sub> 4.53% → 16.6%

Hydrogen, H<sub>2</sub> <0.05% → <0.18%

Carbon Monoxide, CO <0.05% → <0.18%

Carbon Dioxide, CO<sub>2</sub> 0.17% → 0.62%

Nitrogen, N<sub>2</sub> 21.4% → 78.5%

### Organic:

Methane, CH<sub>4</sub> 0.003% → 0.011%

Ethane, C<sub>2</sub>H<sub>6</sub> <0.001% → <0.004%

Ethylene, C<sub>2</sub>H<sub>4</sub> <0.001% → <0.004%

Acetylene, C<sub>2</sub>H<sub>2</sub> <0.001% → <0.004%

total → 95.73%



*MM King*

**Attachment 3**

**Boral Metal Interaction With Boric Acid Solution Test Report**

*Testing Laboratory* Internal Correspondence

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**Date** September 12, 1996

**To** Mark Mc Keown

**Location** Prairie Island

**From** Tim Fair

**Location** CSC-2

**Subject** Boral metal interaction with Boric Acid Solution.

**Purpose**

A sample of Boral metal, reported to contain two surface layers of Aluminum enclosing a core of Aluminum powder and powdered Boron Carbide, was evaluated in the presence of Boric Acid to determine the rate of generation and identity of any gas evolved.

**Sample**

The sample was received on August 20, 1996 and was given lab number D8697. The sample was a flat rectangular piece of metal. A rectangular piece 1.994 inches long by .6016 inches wide and .0925 inches thick was cut from the sample using a band saw. Results of the evaluation are presented below.

**Test Methods**

The sample was weighed using a Mettler AE163 Balance (B63490).

Next, the surface area was determined by measuring the length, width, and thickness of the sample using a digital micrometer. The surface area of the Aluminum layers was thus determined to be 2.40 square inches. In a similar manner, the area of the exposed edge was determined to be .48 square inches.

**Preparation of the Boric Acid Solution:**

A 3500 PPM solution of Boron from Boric Acid ( $H_3BO_3$ ) crystals (Guaranteed Reagent EM BX0865-3) was prepared in water (Ultra Resi-Analyzed J.T. Baker lot J32265) as follows. Using a Mettler AE200 (J91086) Balance, 5.0012 grams of  $H_3BO_3$  (Moi wt. = 61.83) was placed into a 250 ml class A volumetric flask. Approximately 200 ml of water was added. Solution of the acid crystals was accomplished in about five minutes with mild hand agitation. The flask was filled to the mark to yield a solution of 3500 PPM Boron. N.B. This solution would provide a slightly higher acid concentration than reported in use (3300 PPM). Therefore, this represents a more conservative situation for this evaluation.

### Test Performance:

The flat metal sample was placed in a centrifuge tube (Kimax 45241) which is graduated to 100 mls. The total volume of the solution in the tube, used for this evaluation was 111 mls. The Boric Acid solution was added to the centrifuge tube until it slightly over filled the tube. A one hole, black rubber stopper with approximately 24 inches of small diameter plastic tubing protruding from the hole, was used to plug the opening in the centrifuge tube. Liquid from the centrifuge tube filled the plastic tubing and no visible gas volume was present in the centrifuge tube prior to immersion in the constant temperature bath.

The centrifuge tube was inverted with the pointed end up, and secured in a constant temperature bath at 110 degrees Fahrenheit. The bath temperature was monitored using an ASTM 3F thermometer (08604). Readings of time, temperature, barometric pressure and volume were recorded manually from 7:30 August 26, 1996, until 6:30 September 3, 1996 (see Table One). The sample was removed from the tube, dried, and weighed.

Within the first few minutes, as the Boric Acid liquid expanded due to the increased temperature, some liquid leakage from the plastic pressure equalization tube was noted. This decreased over time and finally stopped when the liquid reached the temperature of the bath. At about this same time, a small gas volume was noted to have formed at the apex of the centrifuge tube. Release of dissolved gas (Air) from within the Boric acid is the most likely explanation. During the test, no bubbles were noticed on the surface of the sample.

On the morning of August 30, 1996, a slightly dark pale green cloudy layer appeared in the tube containing the sample. It surrounded the portion of the rubber stopper that protrudes into the neck of the tube. There was no definitive break line between the cloudy layer and the clear liquid layer. The change in color diffused upward. Removing the tube from the temperature bath agitated the cloudy layer and resulted in the appearance of a light colored flocculate which filled the total volume of the tube. The tube was put back into the bath at 13:30 and removed again at 6:30 September 3, 1996. No increase in the flocculate was noted. There did appear to be a collection of the flocculate on the edges of the sample. After settling (due to gravity), the total volume of the low density flocculate was about .5 milliliters. It was removed and retained.

At the conclusion of the evaluation, additional tests on the gas evolved and the ion concentration in the acid solution were also run. The identity of gasses present in the volume above the acid solution were determined using Gas Chromatography (GC) method (SP G01). This same method allows for the determination of the amount of these gasses present. An aliquot of the acid solution was tested using Inductively Coupled Plasma (ICP) methods to determine the amount of Aluminum dissolved in the solution.

### Test Results

Table one presents the raw data as transcribed from the lab worksheet. The table also includes calculations for correcting the observed gas volumes. The corrections take into account changes in barometric pressure, the partial pressure of water vapor and temperature. A graph of this corrected data, over time, is also included.

About .66 mls of gas was generated in 192 hours from a surface area of <sup>2.40</sup>~~2.34~~ square inches of Aluminum and .48 square inches of powered edge. Allowing that Aluminum has been shown to produce little if any Hydrogen under identical conditions (see report on sample D8758), this equates to about .007 mls per hour per square inch of edge area.

MVA  
10/30/96

Gas Chromatography results (see included worksheets) show that about 25% of the gas that was collected (recovery of 91%) is Hydrogen. In addition, small amounts of Methane and Ethane were detected at .15% and .002% respectively. Oxygen was present at only 1.25%.

Weight loss from the sample was .0136 grams from a 4.7680 gram starting weight or a .28% loss in 192 hours.

## Testing Laboratory Internal Correspondence

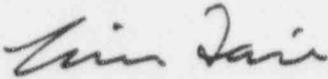
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Material lost from the sample in the Boric Acid solution (.0136 grams in 111 mls) was calculated to be about 125 PPM.

ICP analysis of the Boric Acid solution showed trace evidence of Aluminum ion in solution (.42 PPM) with a 101% recovery. After subtracting blank values, only Zinc (2.56 PPM) was detected at a level greater than 1 PPM (Cr, Cu, Fe, Pb, Mg, Mo, Ni, Mn all less than 1 PPM).

### **Conclusions/Recommendations**

No evidence of effervescence from the sample was noticed. The sample D8697 in contact with 3500 PPM Boric Acid at 110 degrees generates some Hydrogen. It could be as much as 1.04 mls per hour per square foot of edge (total volume as Hydrogen). Aluminum was detected at less than 1 PPM in the Boric Acid solution after 192 hours at 110 degrees Fahrenheit. Evidence of Methane and Ethane in the gas formed plus the reduced amount of Oxygen detected in the gas formed and the absence of dissolved metals, suggest the possibility of bacterial contamination. Since the band saw cutting operation was the only deviation between this sample and a similar test, the band saw might be the source of contamination.



Tim Fair

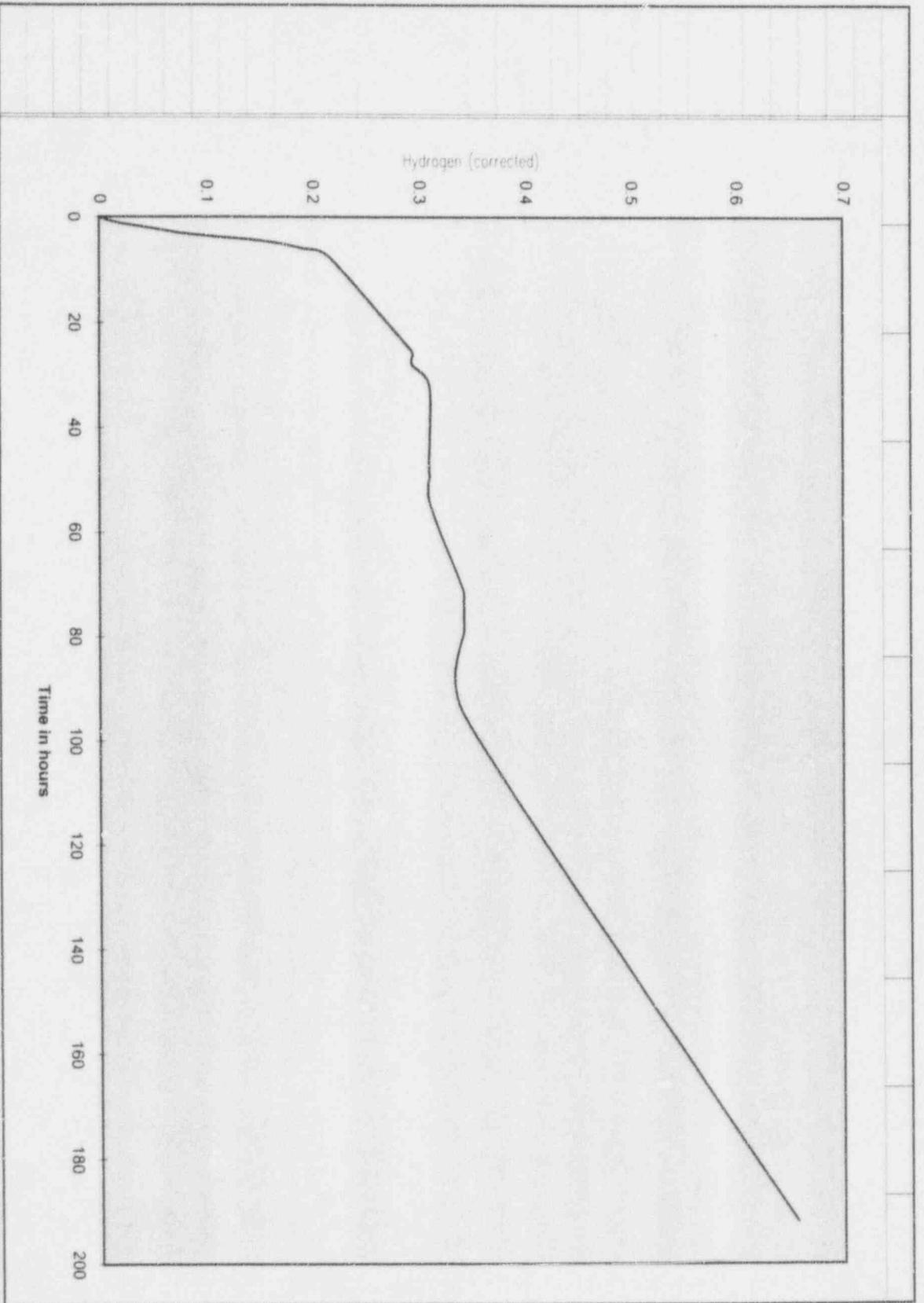
Testing Laboratory Team Leader



TABLE ONE

Volume	Pressure	Water Vapor Corrected Pressure	Standard Pressure Correction	Standard Temperature Correction	Time (hours)	Corrected Volume
					0	0
0.02	741.6	678	0.89210526	0.86392405	1	0.0154142
0.06	741.2	677.6	0.89157895	0.86392405	2	0.0462154
0.11	741.3	677.7	0.89171053	0.86392405	3.25	0.0847407
0.2	742.1	678.5	0.89276316	0.86392405	4.5	0.1542559
0.25	742.1	678.5	0.89276316	0.86392405	6	0.1928199
0.28	742.4	678.8	0.89315789	0.86392405	7.5	0.2160538
0.38	742.8	679.2	0.89368421	0.86392405	25.25	0.2933886
0.38	745.2	681.6	0.89684211	0.86392405	26	0.2944253
0.38	745.1	681.5	0.89671053	0.86392405	28	0.2943821
0.4	745.3	681.7	0.89697368	0.86392405	32	0.3099669
0.4	744.4	680.8	0.89578947	0.86392405	47.5	0.3095576
0.4	745.7	682.1	0.8975	0.86392405	49	0.3101487
0.4	746.4	682.8	0.89842105	0.86392405	54.5	0.310467
0.44	745.4	681.8	0.89710526	0.86392405	71	0.3410136
0.44	746.4	682.8	0.89842105	0.86392405	73	0.3415137
0.44	746.7	683.1	0.89881579	0.86392405	74	0.3416638
0.44	746.9	683.3	0.89907895	0.86392405	76	0.3417638
0.44	747.1	683.5	0.89934211	0.86392405	79	0.3418638
0.44	746.7	683.1	0.89881579	0.86392405	95	0.3416638
0.8	740	676.4	0.89	0.9222973	192	0.6566757

Graph 2



# Gas in Oil Analysis Sheet

## Special Projects Lab



Run #: 1

Date Tested: 9-3-96

### Equipment Data

Location: "D8697"

Ambient Temp: \_\_\_\_\_

Serial Number: \_\_\_\_\_

Wind Temp: \_\_\_\_\_

Trans Number: \_\_\_\_\_

Oil Temp: \_\_\_\_\_

Date Sampled: 9-3-96

Sample Vol. Inj. 0.5 ml.

ml of gas: \_\_\_\_\_ ml of oil: \_\_\_\_\_

Std. Vol. Inj. 1.1 ml.

$$"CF" = \frac{1.1}{.5} = \boxed{2.2}$$

### Analysis Data

#### Inorganic:

Oxygen, O<sub>2</sub> 0.57% → 1.25%

Hydrogen, H<sub>2</sub> 11.28% → 24.8%

Carbon Monoxide, CO <0.05% → <0.1%

Carbon Dioxide, CO<sub>2</sub> 0.15% → 0.32%

Nitrogen, N<sub>2</sub> 29.3% → 64.5%

#### Organic:

Methane, CH<sub>4</sub> 0.066% → 0.15%

Ethane, C<sub>2</sub>H<sub>6</sub> 0.001% → 0.002%

Ethylene, C<sub>2</sub>H<sub>4</sub> <0.001% → <0.002%

Acetylene, C<sub>2</sub>H<sub>2</sub> <0.001% → <0.002%

Total → 91.02%



*RM Rumpf*

**Attachment 4**

**Zinc/Aluminum Metal Interaction With Boric Acid Solution Test Report**

## Testing Laboratory Internal Correspondence

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**Date** September 12, 1996

**To** Mark Mc Keown

**Location** Prairie Island

**From** Tim Fair

**Location** CSC-2

**Subject** Zinc/Aluminum metal interaction with Boric Acid Solution.

### **Purpose**

A sample of sputtered metal coating, reported to contain equal volumes of Zinc and Aluminum, was evaluated in the presence of Boric Acid to determine the rate of generation and identity of any gas evolved.

### **Sample**

The sample was received on August 8, 1996 and was given lab number D8688. The sample was flat, with one relatively smooth side and one less smooth side. It was roughly rectangular in shape with rough or jagged edges. Results of the evaluation are presented below.

### **Test Methods**

The sample was weighed using a Mettler AE200 Balance (J91086).

Next, the surface area was determined by placing the flat sample on a copier and generating a copy with the silhouette of the sample. A rectangle, one and one half inches by three quarters of an inch, was drawn around the silhouette of the sample. The rectangle was cut from the paper and weighed using a Mettler AE163 Balance (B63490). The white area around the sample silhouette was cut away and again, the remaining paper, described by the silhouette alone, was weighed. The weight of the paper silhouette (.0438 Gms.) divided by the weight of the paper rectangle (.0569 Gms.), yields a ratio (.77). This ratio (.77), when multiplied by the surface area (1.125 square inches) of the rectangle, yields an approximation of the area of the sample (.87 times two sides equals 1.73 square inches). A correction was made for the two sides of the sample but no correction was made for edges. The surface area of the sample was thus determined to be 1.73 square inches.

### **Preparation of the Boric Acid Solution:**

A 3500 PPM solution of Boron from Boric Acid ( $H_3BO_3$ ) crystals (Guaranteed Reagent EM BX0865-3) was prepared in water (Ultra Resi-Analyzed J.T. Baker lot J32265) as follows. Using a Mettler AE200 (J91086) Balance, 5.0054 grams of  $H_3BO_3$  (Mol wt. = 61.83) was placed into a 250 ml class A volumetric flask. Approximately 200 ml of water was added. Solution of the acid crystals was accomplished in about five minutes with mild hand agitation. The flask was filled to the mark to yield a solution of 3500 PPM Boron. N.B. This solution would provide a slightly higher acid concentration than reported in use (3300 PPM). Therefore, this represents a more conservative situation for this evaluation.

### Test Performance:

The flat metal sample was placed in a centrifuge tube (Kimax 45241) which is graduated to 100 mls. The total volume of the solution in the tube, used for this evaluation was 111 mls. The Boric Acid solution was added to the centrifuge tube until it slightly over filled the tube. A one hole, black rubber stopper with approximately 24 inches of small diameter plastic tubing protruding from the hole, was used to plug the opening in the centrifuge tube. Liquid from the centrifuge tube filled the plastic tubing and no visible gas volume was present in the centrifuge tube prior to immersion in the constant temperature bath.

The centrifuge tube was inverted with the pointed end up, and secured in a constant temperature bath at 110 degrees Fahrenheit. The bath temperature was monitored using an ASTM 3F thermometer (08604). A problem with the temperature controller within the first half hour was detected and corrected. The temperature of the bath reached about 120 degrees for approximately 30 minutes (a previous evaluation using pure Zinc (20 Mesh) showed no indications of a controller problem). This temperature excursion of plus ten degrees Fahrenheit for one half hour over the 73 hours of the evaluation appears to be of no consequence.

Readings of time, temperature, barometric pressure and volume were recorded manually from 7:00 August 13, 1996, until 8:00 August 16, 1996 (see Table One). The sample was removed from the tube, dried, and weighed.

Within the first few minutes, as the Boric Acid liquid expanded due to the increased temperature, some liquid leakage from the plastic pressure equalization tube was noted. This decreased over time and finally stopped when the liquid reached the temperature of the bath. At about this same time, a small gas volume was noted to have formed at the apex of the centrifuge tube. Release of dissolved gas (Air) from within the Boric acid is the most likely explanation.

Shortly thereafter, and for the remainder of the evaluation, small bubbles appeared on the surface of the sample. The distribution on each side appeared uniform but one surface (the less smooth side) appeared to exhibit more bubbles. During the entire time of periodic observation, not once was a bubble seen to leave the surface of the sample of its own accord. Later in the evaluation, to get better gas volume measurements, the centrifuge tube was removed from the constant temperature bath momentarily, and inverted several times to dislodge those bubbles on the surface of the sample. The resulting accumulation of gas at the top of the tube, allowed a more accurate measurement of the volume to be made. This agitation also dislodged sporadic gas bubbles that formed/adhered to the interior surface of the glass centrifuge tube.

At the conclusion of the evaluation, additional tests on the gas evolved, the gas in the acid solution, and the ion concentration in the acid solution were also run. The identity of gasses present in the volume above the acid solution were determined using Gas Chromatography (GC) method (SP G01). This same method allows for the determination of the amount of these gases present. A portion of the liquid was placed in a reduced pressure apparatus (Dobel Gas Extractor type CA-1) and the amount of gas soluble in the liquid at room temperature was determined. Again, the dissolved gas was identified and quantified using GC method (SP G01). An aliquot of the acid solution was tested using Inductively Coupled Plasma (ICP) methods to determine the amount of Zinc and Aluminum dissolved in the solution. Finally, a piece of the metal sample used in the evaluation was dissolved in HCl and analyzed using ICP methods.

### Test Results

Table one presents the raw data as transcribed from the lab worksheet. The table also includes calculations for correcting the observed gas volumes. The corrections take into account changes in barometric pressure, the partial pressure of water vapor and temperature. A graph of this corrected data, over time, is also included.

About 5 mls of gas was generated in 73 hours from a surface area of 1.73 square inches. This equates to about .04 mls per hour per square inch or 5.7 mls per hour per square foot. (1.7 liters per hour for 300 square feet)

## Testing Laboratory Internal Correspondence

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Gas Chromatography results (see included worksheets) show that about 50% of the gas that was collected (recovery of 82%) is Hydrogen.

Gas Chromatography results (see included worksheets) show that about 40% of the gas that was released from the Boric Acid solution (recovery of 88%) is Hydrogen.

From the degassing procedure, it was determined that 1.83 mls of gas was dissolved in 110 mls Boric Acid solution at room temperature.

Weight loss from the sample was .0118 grams from a .7028 gram starting weight or a 1.7% loss in 73 hours.

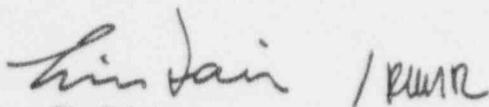
Dissolved metal ions from the sample in the Boric Acid solution (.0118 grams in 111 mls) were calculated to be about 110 PPM.

ICP analysis of the Boric Acid solution showed trace evidence of Aluminum ion in solution (.44 PPM). ICP analysis of the Boric Acid solution found 121 PPM Zinc in solution with a 107% recovery.

ICP analysis of a piece of metal used in the evaluation shows 61% Zinc and 26% Aluminum (remainder most likely oxides and minor trace elements).

### **Conclusions/Recommendations**

No evidence of precipitation was detected at either the elevated temperature of 110 degrees Fahrenheit or after the solution was cooled to room temperature. No evidence of effervescence from the sample was noticed. Metal from sample D8688 in contact with 3500 PPM Boric Acid at 110 degrees generates Hydrogen. It could be as much as 5.7 mls per hour per square foot (total volume as Hydrogen). Some Hydrogen is soluble in the 3500 PPM Boric Acid solution. It could be as much as 1.83 mls Hydrogen per 110 mls Boric Acid solution or 16.7 mls Hydrogen per liter of Boric Acid solution (total volume as Hydrogen). Zinc was detected (121 PPM) in the Boric Acid solution after 73 hours at 110 degrees Fahrenheit. Aluminum was detected at less than 1 PPM in the Boric Acid solution after 73 hours at 110 degrees Fahrenheit.

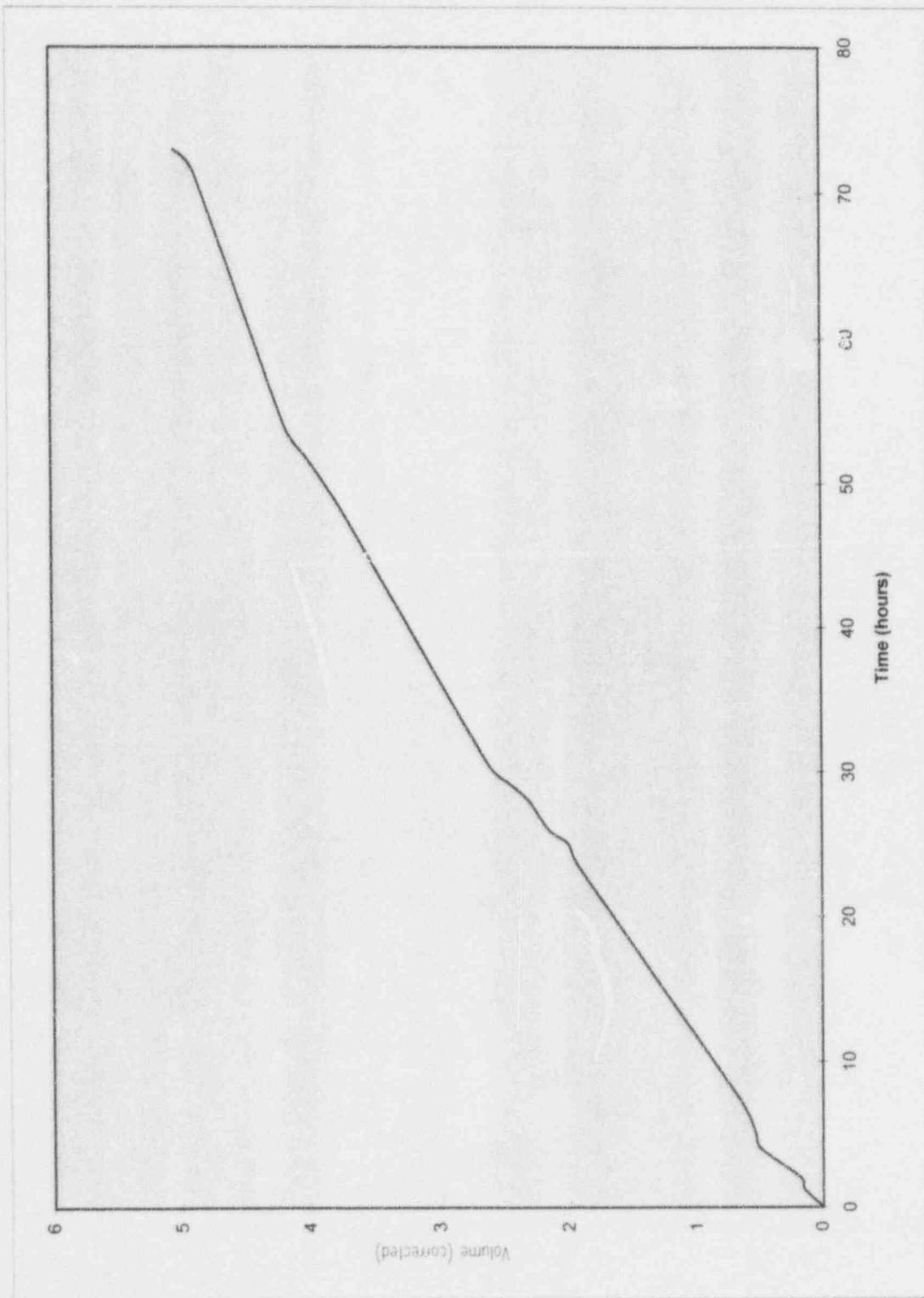


Tim Fair  
Testing Laboratory Team Leader

TABLE ONE

Volume	Pressure	Water Vapor Corrected Pressure	Standard Pressure Correction	Standard Temperature Correction	Time (hours)	Corrected Volume
					0	0
0.2	741.4	677.8	0.89184211	0.863924051	1.25	0.154097
0.23	741	677.4	0.89131579	0.863924051	2	0.177107
0.65	741	677.4	0.89131579	0.863924051	4	0.500519
0.7	740.8	677.2	0.89105263	0.863924051	5	0.538861
0.75	740.6	677	0.89078947	0.863924051	6	0.577181
0.85	740	676.4	0.89	0.863924051	7.3	0.653559
1	739.6	676	0.88947368	0.863924051	8.75	0.768438
2.5	740.7	677.1	0.89092105	0.863924051	23.5	1.92422
2.6	740.8	677.2	0.89105263	0.863924051	25	2.001485
2.8	740.9	677.3	0.89118421	0.863924051	26	2.155763
3	741.1	677.5	0.89144737	0.863924051	28	2.310428
3.2	741.3	677.7	0.89171053	0.863924051	29.25	2.465185
3.4	742	678.4	0.89263158	0.863924051	30.5	2.621964
4.8	743.5	679.9	0.89460526	0.863924051	47.75	3.709781
5	744.1	680.5	0.89539474	0.863924051	50	3.867765
5.2	744.3	680.7	0.89565789	0.863924051	52	4.023658
5.4	744.4	680.8	0.89578947	0.863924051	54	4.179028
6.3	744.8	681.2	0.89631579	0.863924051	71.5	4.878397
6.5	744.8	681.2	0.89631579	0.863924051	73	5.033267

Graph 1



Gas in Oil Analysis Sheet  
Special Projects Lab



Run #: 0

Date Tested: 8-16-96

Sample ID  
"D 8688"

Equipment Data

Location: \_\_\_\_\_ Ambient Temp: \_\_\_\_\_  
 Serial Number: \_\_\_\_\_ Wind Temp: \_\_\_\_\_  
 Trans Number: \_\_\_\_\_ Oil Temp: \_\_\_\_\_  
 Date Sampled: \_\_\_\_\_ Sample Vol. Inj. \_\_\_\_\_

ml of gas: X ml of oil: X (from gas Std. Vol. Inj. \_\_\_\_\_)

Injection of gas, bubble → (removed from centrifuge tube)  
 Injected 0.55 ml.

CF = 2.0

Analysis Data

(x 2.0)

Inorganic:

Oxygen, O<sub>2</sub> 1.94 → 3.88%  
 Hydrogen, H<sub>2</sub> 24.63 → 49.3%  
 Carbon Monoxide, CO <0.05 → <0.1%  
 Carbon Dioxide, CO<sub>2</sub> 0.047 → 0.094%  
 Nitrogen, N<sub>2</sub> → 14.3 → 28.6%

Organic:

Methane, CH<sub>4</sub> 0.005 → 0.01%  
 Ethane, C<sub>2</sub>H<sub>6</sub> <0.001 → <0.002%  
 Ethylene, C<sub>2</sub>H<sub>4</sub> <0.001 → <0.002%  
 Acetylene, C<sub>2</sub>H<sub>2</sub> <0.001 → <0.002%



total: 81.9%

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Gas in Oil Analysis Sheet  
Special Projects Lab



Run #: 1

Date Tested: 8-16-96

Sample ID "D 8688"

Equipment Data

Location: \_\_\_\_\_ Ambient Temp: \_\_\_\_\_  
 Serial Number: \_\_\_\_\_ Wind Temp: \_\_\_\_\_  
 Trans Number: \_\_\_\_\_ Oil Temp: \_\_\_\_\_  
 Date Sampled: \_\_\_\_\_ Sample Vol. Inj. \_\_\_\_\_  
 Std. Vol. Inj. \_\_\_\_\_

ml of gas: 0.5 ml of oil: 30ml.

gas extracted from fluid :

injected : 0.3ml

C.F. = 3.666

Analysis Data

Inorganic:

$$\left( \times \frac{1.1}{.3} \right)$$

Oxygen, O<sub>2</sub> 3.04 = 11.15%

Hydrogen, H<sub>2</sub> 10.82 = 39.7%

Carbon Monoxide, CO <0.05 = <0.18%

Carbon Dioxide, CO<sub>2</sub> 0.594 = 2.18%

Nitrogen N<sub>2</sub> 9.53 = 34.9%

Organic:

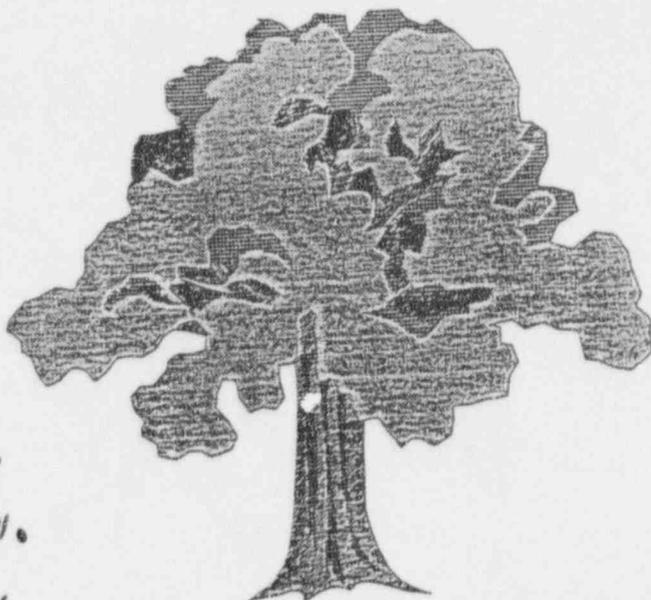
Methane, CH<sub>4</sub> 0.003 = 0.011%

Ethane, C<sub>2</sub>H<sub>6</sub> <0.001 = <0.004%

Ethylene, C<sub>2</sub>H<sub>4</sub> <0.001 = <0.004%

Acetylene, C<sub>2</sub>H<sub>2</sub> <0.001 = <0.004%

total : 87.9%



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