## 2010 CLEAR ROADS SNOW AND ICE CONTROL CHEMICAL PRODUCTS SPECIFICATIONS AND TEST PROTOCOLS

#### (Disclaimer: the Qualified Product List has been transitioned from the Pacific Northwest Snowfighters to Clear Roads. In the near future, this document will be updated to reflect this transition. However, the technical content will remain unchanged.)

#### I. GENERAL SPECIFICATIONS

To bid a product, that product shall be on the most current Qualified Products List (QPL), or the product is currently being evaluated for qualification as part of this bid process if the offer to submit samples is made by the agency. To submit a product for the qualification process, contact any of the PNS members for information. In the case of a request for bid, please contact the agency requesting the bid for information on how to become a qualified bidder.

The PNS Association of British Columbia, Idaho, Montana, Oregon, and Washington have developed the Qualified Products List. The list is composed of products that have been tested and found to be in conformance with these specifications. Any material changes to a product that is listed on the QPL by either the manufacturer or the bidder, which in any way makes the product different from the original qualified material, shall be grounds for disqualifying the product from the list. The new product will have to be re-qualified before it will be allowed to be placed back on the QPL.

The bidder of any product that is delivered and/or applied, which is found to be contaminated and is cause for environmental concerns, shall be responsible for all clean up expenses. This includes but is not limited to clean up measures as needed for the following: storage facility, yard, equipment, and roadside.

The bidder shall be liable, as determined by the purchaser for causing any unanticipated extraordinary damages to equipment used in the storage or distribution of the chemical products.

The PNS has the right to qualify or disqualify, accept or reject products based on the materials used to produce the product. The products will be assessed for the potential of causing a decrease in the public safety. The right to qualify or disqualify, accept or reject a product based on manufactured composition rest solely with the PNS. The PNS assessment shall be final and in the best interest of the PNS.

Each bidder submitting a sample will be notified whether the sample passes or fails to meet the specifications. Copies of the complete lab reports will be available upon request.

All submitted products shall be tested to the specified limits contained within these specifications and as per the products' specific category classifications. A product that passes the required specification testing limits and has passed the PNS review shall be placed onto the PNS Qualified Products List. A product that fails to meet the standard limits as specified will not be placed onto the Qualified Products List and the bid will be disqualified.

A. A submitted product that contains any constituent in excess of the following established total concentration limits as tested in accordance with the listed test methodology from Section VI shall be not be acceptable. Results are stated as **parts per million (ppm)**.

Arsenic	5.0
Barium	100.0
Cadmium	0.20
Chromium	1.0
Copper	1.0
Lead	1.0
Mercury	0.05
Selenium	5.0
Zinc	10.00
Phosphorus	2500.
Cyanide	0.20

Note: Liquid products shall be tested as received. Solid Salts are to be diluted to a 25% (W/V) concentration and then tested as if the material was a liquid sample. Report only the values determined from the 25% solution for all of the parameters as compared to the specification limits. Do not back calculate the concentration of the parameters to the dry weight of the material.

- B. No bid will be accepted on any corrosion inhibited product that has not successfully completed the National Association of Corrosion Engineers (NACE) Standard TM0169-95, as modified by the PNS, and found to have a Corrosion Value of at least 70% less than that of Sodium Chloride (salt).
- C. The manufacturer shall also supply the following analyses for information purposes for liquid products or solid products that will be converted into a liquid product for application purposes. Testing of the following parameters will be done by the listed testing methodology from Section VI.

Ammonia - Nitrogen Total Kjeldahl Nitrogen Nitrate and Nitrite - Nitrogen Biological Oxygen Demand Chemical Oxygen Demand Frictional Analysis Toxicity Testing Rainbow Trout or Fathead Minnow Toxicity Test Ceriodaphnia Dubia Reproductive and Survival Bioassay Selenastrum Capricornutum Algal Growth

#### **II. SAMPLE SUBMITTALS**

- A. If a product that is currently listed on the Qualified Products List is to be bid no sample submission or information packet is required. If a new product is being submitted for evaluation during a bidding opportunity the bid shall be accompanied by **two** one gallon (4 liter) containers of the product along with the chemical, biological, and physical analyses of the product by a qualified laboratory. See "Product Sample Checklist" for complete instructions "as to how to provide required samples and information. All samples must be marked with an easily distinguishable name and the associated paper work must be clearly marked as such so that the samples and the submitted product information can be easily identified and matched up. Failure to supply the required samples and product information will be cause for disqualification. These samples will be used to establish a database for future fingerprinting of all approved products when delivered to any of the PNS locations and for future bid comparisons. Any products purchased in the future will be expected to meet specifications as established in the bid process. All test data that is submitted with each product sample is subject to verification by one or more of the PNS laboratories. Results of the testing from the PNS's laboratories shall be verifiable and final. Information and laboratory results shall be submitted according to the general and specific product specification contained within this document. The following results and information are mandatory at the time of submission and shall be verified from the Product Sample Check List.
  - 1. Corrosion test data obtained according to NACE Standard TM0169-95 as modified by PNS.
  - 2. pH (liquid products only) The pH of submitted liquid chemical products shall be within the specified limits as designated in the appropriate categories. The pH of liquid chemical products that contains organic matter as one of it constituents may be waived by the PNS for each of the liquid categories that require adherence to a specified pH range. The right to waive the pH will be at the discretion of the PNS. The PNS decision to waive the pH requirement shall be in the best interest of the PNS and shall be final.

NOTE: Recent testing has concluded that brines inhibited with organic matter exhibit lower pH values than do brines with non-organic matter inhibitors. Organic matter, such as peat, routinely exhibits low pH values because they generate weak organic acids. These weak organic acids are prevalent in the ecology system and are necessary to maintain a healthy environment. Our main concern, in addressing pH, was to limit the amount of excess inorganic acidity or alkalinity that brine could carry. Corrosion testing has shown that these weak organic acids do not have a detrimental effect but seem to enhance the corrosion inhibiting power of the products. Due to this, the pH parameter on brines that contain organic matter may be waived by the PNS. The organic matter information shall be included in the Product Information section of the Bid Schedule and bidder may apply for the pH waiver. The bidder must also provide documentation as to what the organic material consists off, and the minimum concentration that it will be added to their product. The PNS reserves the right to use any and all testing procedures necessary to verify bidder data.

- 3. Analytical results of all constituents for which limits have been set by the General Specifications in Section I, Part A. <u>The analytical results shall reflect testing to the specified limit or below. For example the specified limit for Cadmium is 0.20 ppm, therefore the supplied analytical results need to reflect testing to that limit or below. A submitted value of less than 1.00 ppm is not acceptable.</u>
- 4. All biological, chemical, toxicology and friction test results as listed in Section I Part C. Friction testing shall be conducted on all liquid samples and may be required on solid products per the discretion of the PNS.
- 5. Specific gravity chart (liquid products only) with correlating weight percentage and freeze point information presented in 1% increments beginning with a five percent solution. The chart must contain information up to, including, and exceeding, *by* 5% (*or the solubility limits of your product*) the concentration being submitted for evaluations.
- 6. Detailed information on the corrosion inhibitor, the minimum concentration of the corrosion inhibitor contained in the product, complete and precise laboratory procedures for verifying inhibitor concentrations <u>SHALL</u> be included with the bid document. Failure to provide sufficient detail to address all specification requirements may result in bid disqualification. <u>Proprietary information must be included and will be held confidential by the PNS</u>. Mark and submit in a separate sealed envelope all the proprietary information to maintain confidentiality.
- B. Bids shall be accompanied with the most recent detailed product specification sheet and Material Safety Data Sheet (MSDS) including the MSDS of the inhibitor. <u>All documents</u> <u>must be clearly legible.</u>
- C. Most chemical products after successfully completing the PNS's initial screening process and corrosion tests may then be required to successfully complete field application/effectiveness tests. The decision as to whether or not to require a supplier to furnish an ample supply of their product (at no charge including shipping) for fieldtesting lies solely with the PNS. If the product requested for field-testing is not furnished, or if an inadequate amount is supplied, or if product performance is not satisfactory, the product will not be placed on the approved product list.

Field application/effectiveness testing of some products may be waived based on the chemical constituents of the product. The PNS has laboratory and field-tested many variations of these products. The results of the field tests should be predictable based on the percentage of the active chemical constituent. The option to waive field application/effectiveness tests lies solely with the PNS.

## **III. ORDERS, DELIVERIES, AND INVOICING OF PRODUCTS**

# ATTENTION: PLEASE REFER TO EACH INDIVIDUAL AGENCIES SPECIFICATIONS REGARDING ORDERING AND DELIVERING PRODUCTS.

- A. Bidder will be responsible for all necessary equipment to transfer liquid chemical products to purchasers' storage tanks. Purchaser's storage tanks will be fitted with a three-inch male pipe fitting to allow for unloading of product.
- B. Each shipment shall be accompanied by a current and clearly legible MSDS.
- C. An anti-foaming agent will be available from the Bidder for use as needed, at no additional charge to the Purchaser, to control foaming during loading, unloading, and agitation of liquid chemical products.
- D. The bill of lading for each shipment must contain the following information.
  - 1. Name of product.
  - 2. Supplier and manufacturer of product.
  - 3. Delivery Destination.
  - 4. Total number of units being delivered.
  - 5. Total weight of delivery using a certified scale ticket or certified flow meter. As an option on liquid deliveries only, the bidder can use a legibly printed certified ticket from a flow meter that has been tested and certified by an approved PNS member's agency of Weight and Measures. The certification of the meter shall not be older than one year. Any PNS member can request that the meter be retested and certified again during the delivery year if the data from the meter is in question. This retesting and certification shall be done at no extra charge to the PNS member. Reciprocity among the PNS members for meter calibration may be employed. The bidder shall provide a copy of the certification and product information about the flow meter at the time of bid. The PNS member may at any time choose to spot check a delivery of liquid product by having the load weighed on certified scales before and after delivery to insure the accuracy of the flow meter. No additional cost will be charged to the PNS member for spot-checking deliveries of liquid products.
  - 6. Lot Number for the product being delivered. The Lot Number is a specific number assigned to that particular product as delivered. This number must be denoted as the "LOT NUMBER" on the bill of lading and shall be clearly legible. The lot number must enable purchaser to track a delivered product back to its manufacture point, date of manufacture and specific batch. Failure to have a defined LOT NUMBER that appears on the Bill of Lading is grounds for rejection of the load.
  - 7. Transport information--Name of transporting company, tank, trailer or rail car number, point and date of origin.

- 8. For liquid products include the Bidder Quoted Concentration and Specific Gravity.
- E. <u>The Agency will not process invoices for payment until the bidder has met all</u> requirements under this section. The invoice shall include the following:
  - 1. A copy of the original bill of lading.
  - 2. Contract unit of measure.
  - 3. Total number of units delivered.
  - 4. Contract unit price for product delivered.
  - 5. Total price for units delivered.

## IV. FIELD INSPECTION, UNLOADING, SAMPLING AND TESTING

All material is subject to field inspection, sampling, and testing on an as delivered base. Sampling and field-testing is the prerogative of the Purchaser. The bidder shall not off load any material without affording the Purchaser an opportunity to conduct the field inspection, sampling or the testing. Off loading of material without affording the Purchaser an opportunity to conduct said work shall deem the delivered material non-compliant and is subject to total rejection. The bidder shall only off load material without field inspection, testing and sampling by the Purchaser when the agency representative grants prior written approval.

## A. FIELD INSPECTION

## BEFORE ALLOWING ANY PRODUCT TO BE UNLOADED AGENCY PERSONNEL WILL ADHERE TO THE FOLLOWING PROCEDURES:

- 1. Document and maintain records on all deliveries, including those that are rejected.
- 2. Check to assure that the product is being delivered according to the terms of the contract. This may include but is not limited to the following:
  - a. Date of the order.
  - b. Date and time of delivery.
  - c. Verification of advance delivery notification.
  - d. Delivered within allowable times.
  - e. Name of Delivery Company and license plate numbers.
  - f. Is any price adjustment assessments required?
  - g. Is the product being delivered what you ordered?
  - h. Document all procedures prior to unloading of product.
  - i. Verify that all papers required of a delivery are present, complete, and legible.
    - 1. Accurate, complete, and legible bill of lading and/or invoice.
    - 2. Legible and current MSDS sheet.
    - 3. Certified weight slip.
- 3. Verify separation or non-separation of product.

4. Visually inspect the load to determine if there are any obvious reasons why the load should be rejected.

5. No precipitate or flocculation in liquid products shall be allowed in excess of the specification limits. Material portraying these or other uncharacteristic traits when delivered may be immediately rejected at the option of the agency or their representative at the delivery location.

6. Any problems must be noted at the point of delivery by agency personnel, documented, and relayed to their agency representative for action.

#### B. UNLOADING

- 1. Provided that all the required information is in place and the material appears to be the correct material as ordered, document the amount of product currently in storage prior to unloading and begin the unloading process.
- 2. The delivery truck shall unload solid materials in a windrow.
- 3. For liquid products, visually inspect the discharge valve prior to unloading for the presence of any foreign material.
- 4. Visually inspect the delivered product again while unloading. If problems are noted that are a cause for rejection of the load, immediately halt the unloading process. Take photos if applicable and record any pertinent information. Conduct the following procedures if the material is to be rejected.
  - a. If material fails the field inspection or testing, reload the product and reject the load.
  - b. If reloading can't be done, (mixed with previous material) note the amount of product (liquid only) pumped into the tank and total product now present in the tank.
  - c. Circulate the tank and then pull two one-gallon (4 Liter) samples of the contaminated chemical material now in the tank
  - d. Check and record the specific gravity of the samples.
  - e. Take appropriate action as needed to assure the integrity of product on hand if possible. Will all products on hand have to be removed?
  - f. Send samples directly to the Agency's designated testing laboratory.
  - g. Immediately advise the Agency's Representative of any ordering, delivery, storage, or product quality issues.

## C. SAMPLING AND TESTING

One sample, of the liquid or dry product being delivered, may be taken from the delivered shipment for laboratory testing after the shipment has passed the initial inspection and is approved for unloading. This sample will be used for testing and/or fingerprinting at the agency's expense to insure product quality. Clearly, label samples for identification. Send the sample directly to the appropriate agency testing laboratory. Be sure the Transmittal form is placed in the box and contains at least the following information; Manufacture or bidders name, name of product, lot number of product, shipping date, date received, name of

delivery point, quantity of material delivered, and name and phone number of person who received the load and took the samples. Test results from the appropriate Laboratory will be final and in the best interest of the Purchaser.

- 1. If the load is liquid, a one-gallon sample will be taken from the transfer hose in three equal parts. Each part will be compositely mixed together with the other parts to make up the one-gallon sample that will be submitted to the laboratory for testing. The samples will be collected during unloading as the first third, the second third and the last third of the product that is being delivered. If the trailer or pup has compartments the three equal samples shall be taken from only one of the compartments to complete the sample. Check and record the specific gravity of the samples.
- 2. If the load is solid, the delivery truck shall unload the solid material in a windrow. Samples of the windrow materials should be obtained from the complete cross section of the windrow. Portions of the sample shall be taken from the top, center, and bottom in proportion to the cross section area at that point and well within the stack each time. It is best practice to cut completely through the stack if practical. Fine material sifts to the bottom. Care should be taken to obtain a complete and representative sample. The sample shall be placed into a wide mouth 1-gallon container with a screw top lid as soon as the sample has been taken to avoid exposing the sample unduly to atmospheric moisture.
- 3. Samples sent to the Laboratory will be tested for conformance to specification during the year. Each type of product may be tested for those parameters listed in the General Specifications and in the appropriate Category requirements.

## V. CHEMICAL PRODUCT AND INHIBITOR PRODUCT CATEGORIES

#### **Chemical Product Category 1**

#### Corrosion Inhibited Liquid Magnesium Chloride Specifications

In addition to the General Specifications the following requirements shall also apply:

1. Product must contain no less than 25% magnesium chloride.

Test Method: Number 1

2. Weight per gallon will be established according to the specific gravity and percentage of magnesium chloride contained in the product bid as indicated by the bidder.

Test Method: Number 2

3. Product will contain the corrosion control inhibitor in quantities not less than those indicated by the bidder. The finished deicing product, including corrosion inhibitors, must be completely accomplished at the original manufacturing plant location. Post adding of corrosion inhibitors or any other ingredients and splash mixing is unacceptable after the product has left the original manufacturing plant.

Test Method: Number 3

4. The pH must be 6.0 - 9.0

Test Method: Number 4

5. This chemical product shall not contain greater than 1.0% (V/V) Total Settleable Solids and shall have Ninety-nine percent (99.0%) of the Solids Passing through a Number 10 sieve after being stored at  $-17.8^{\circ}C + -1^{\circ}C (0^{\circ}F + -2^{\circ}F)$  for 168 hours (Seven days).

#### Corrosion Inhibited Liquid Calcium Chloride Specifications

In addition to the General Specifications the following requirements shall also apply:

1. Product must contain no less than 25% calcium chloride.

Test Method: Number 1

2. Weight per gallon will be established according to the specific gravity and percentage of calcium chloride contained in the product bid as indicated by the bidder.

Test Method: Number 2

3. Product will contain corrosion control inhibitor in quantities not less than those indicated by the bidder. The finished deicing product, including corrosion inhibitors, must be completely accomplished at the original manufacturing plant location. Post adding of corrosion inhibitors or any other ingredients and splash mixing is unacceptable after the product has left the original manufacturing plant.

Test Method: Number 3

4. The pH must be 6.0 - 10.0

Test Method: Number 4

5. This chemical product shall not contain greater than 1.0% (V/V) Total Settleable Solids and shall have ninety nine percent (99.0%) of the Solids Passing through a Number 10 sieve after being stored at  $-29^{\circ}C$  +/- 1°C (-20°F +/- 2°F) for 168 hours (Seven days).

#### Liquid Calcium Magnesium Acetate CMA Specifications (Bidder Manufactured)

In addition to the General Specifications the following requirements shall also apply:

1. Product must contain no less than 25% calcium magnesium acetate (CMA).

Test Method: Number 1

2. Weight per gallon will be established according to the specific gravity and percentage of CMA contained in the product bid as indicated by the bidder.

Test Method: Number 2

3. The pH must be 8.0 - 10.0

Test Method: Number 4

4. This chemical product shall not contain greater than 4.0 % (V/V) Total Settleable Solids and shall have ninety nine percent (99.0%) of the Solids Passing through a Number 10 sieve after being stored at <u>-12°C +/- 1°C (+10°F +/- 2°F)</u> for 168 hours (Seven days).

Test Method: Number 6

5. Calcium to magnesium mole ratio shall be 3 to 7.

Test Method: Number 1

6. Residual base shall be a maximum of 0.30 meq (milliequivalents) base per gram of sample.

#### Corrosion Inhibited Solid Sodium Chloride Specifications

## CATEGORIES 4A AND 4B

The Categories shall be defined as follows:

1.	Category 4A	Corrosion Percent Effectiveness of 30% or less
		Gradation – ASTM D 632 Type I, Grade 2
2.	Category 4B	Corrosion Percent Effectiveness of 31% to 85%
		Gradation ASTM D 632 Type I, Grade - Modified

In addition to the General Specifications the following requirements shall also apply:

1. Gradation - Test Method: Number 13

CATEGORY 4A		CATEGORY 4B	
Sieve	Wt. %	Sieve	Wt. %
Size	Passing <b>Passing</b>	Size	<u>Passing</u>
3/4"	100	3/4"	100
#4	20 - 100	1/4"	75 - 85
#8	10 - 60	#8	50 - 70
#30	0 - 15	#30	10 - 20

2. Anti-Caking agent will be included to insure that the material remains free from hard caking and suitable for its intended purpose.

Test Method: Number 14

**NOTE:** Salt for highway use is usually treated with either Ferric Ferrocyanide, also known as Prussian Blue, or Sodium Ferrocyanide, also known as Yellow Prussiate of Soda (YPS), to prevent the salt from caking. The amount of Prussian Blue added is 70 to 165 parts per million (ppm), equivalent to 0.33 to 1.14 pounds per ton of salt. YPS is added in the amount of 50 to 250 ppm, equivalent to 0.1 to 0.5 pounds per ton of salt. YPS is also used as an anti-caking agent in table salt, and has approval of the U.S. Food and Drug Administration. Based on exhaustive testing no evidence of toxicity was demonstrated. If used, the presence of these products will not be assessed towards the total cyanide concentration when testing this product. However, the total cyanide concentration of the original material must meet specifications. Information may be obtained from the Salt Institutes Highway Digest Publication.

Bidder may bid this product with or without the anti-caking agent. Bidders must note on the Sample Checklist if the sample does contain anti-caking agent or not. If the Bidder

#### **Chemical Product Category 4---Continued**

chooses not to add the anti-caking agent it does not prevent the bidder from assuring that the delivered product is in a free-flowing state.

3. Material must be clean and free from extraneous matter. The material must be homogenous or manufactured in such a manner to assure that the corrosion inhibitor, anti-caking agent and the chemical product does not segregate.

Test Method: Number 14

4. Moisture Content

#### **Category 4A**

The salt shall be dried to a maximum moisture content of 0.5 % (percent by weight). Water in excess of 0.5% of dry salt weight will not be paid for. The amount of salt to be paid for, when moisture exceeds 0.5% shall be computed as follows:

Pay Weight = (100.5 x Wet Wt. of Salt) divided by (100 + Percent of Moisture)

NOTE: The moisture content is judged as available free water. Organic Bases Corrosion Inhibitors that are used in the processes of making this product that impart a loss in weight (Organic Matter Weight Loss) when ran according to the prescribe test method but do not reflect the loss of available free water shall be limited to a maximum of 3% by weight. Products that exceed the 3% by weight limit shall be subject to the same equation as above with the limit being adjusted to 3%. Additionally, the use of said inhibitors may be used provided that the material remain free flowing, will not clump, cause hard caking and remains suitable for use. The use of these types of inhibitors may require additional testing to be provided by the bidder at the request of the PNS before approval to the qualified products list is granted. The amount of available water in the inhibitor and the base salt will be required along with a mass balance analysis of the two products to show the theoretical amount of free water that is available in the finished product.

Test Method: Number 12

## Category 4B

The salt shall not exceed a maximum moisture content of 5.0 % (percent by weight). Water in excess of 5.0% of dry salt weight will not be paid for. The amount of salt to be paid for, when moisture exceeds 5.0% shall be computed as follows:

Pay Weight = (105.0 x Wet Wt. of Salt) divided by (100 + Percent of Moisture)

## **Chemical Product Category 4---Continued**

5. Pay Weight Schedule for Insoluble Residue

## **Category 4B**

The salt shall have a maximum insoluble residue of 10.0 % (percent by dry weight). Insoluble residue in excess of 10.0% of dry salt weight will not be paid for. The amount of salt to be paid for, when the insoluble residue exceeds 10.0% shall be computed as follows:

Pay Weight = (110.0 x Dry Wt. of Salt) divided by (100 + Percent Insoluble Residue)

6. Corrosion Control Inhibitor and Concentration

#### Corrosion Inhibited Sodium Chloride Plus 10% Magnesium Chloride Specifications

In addition to the General Specifications the following requirements shall also apply:

The bidder must state the use of solid or liquid magnesium chloride. For liquid applications the manufacturer shall use at a minimum a 28% concentration of magnesium chloride. The manufacturer shall supply information as to what concentration of the magnesium chloride was used in the process.

1. Gradation of product shall be Type 1, Grade 2, for Sodium Chloride.

Test Method: Number 13

#### PHYSICAL REQUIREMENTS AND TOLERANCES

Sieve	Wt. %
Size	Passing Passing
3/4"	100
#4	20 - 100
#8	10 - 60
#30	0 - 15

2. Anti-Caking agent will be included to insure that the material remains free from hard caking and suitable for its intended purpose.

Test Method: Number 14

**NOTE:** Salt for highway use is usually treated with either Ferric Ferrocyanide, also known as Prussian Blue, or Sodium Ferrocyanide, also known as Yellow Prussiate of Soda (YPS), to prevent the salt from caking. The amount of Prussian Blue added is 70 to 165 parts per million (PPM), equivalent to 0.33 to 1.14 pounds per ton of salt. YPS is added in the amount of 50 to 250 PPM, equivalent to 0.1 to 0.5 pounds per ton of salt. YPS is also used as an anti-caking agent in table salt, and has approval of the U.S. Food and Drug Administration. Based on exhaustive testing no evidence of toxicity was demonstrated. If used, the presence of these products will not be assessed towards the total cyanide concentration when testing this product. However, the total cyanide concentration of the original material must meet specifications. Information may be obtained from the Salt Institutes Highway Digest Publication.

Bidder may bid this product with or without the anti-caking agent. Bidders must note on the Sample Checklist if the sample does contain anti-caking agent or not. If the Bidder chooses not to add the anti-caking agent it does not prevent the bidder from assuring that the delivered product is in a free-flowing state.

#### **Chemical Product Category 5---Continued**

3. Material must be clean and free from extraneous matter. The material must be homogenous or manufactured in such a manner to assure that the corrosion inhibitor, anti-caking agent and the chemical product does not segregate.

Test Method: Number 14

- 4. Moisture Content Of Sodium Chloride Only.
  - A. Sodium Chloride Only

The salt shall be dried to a maximum moisture content of 0.5 % (percent by weight). Water in excess of 0.5% of dry salt weight will not be paid for. The amount of salt to be paid for, when moisture exceeds 0.5% shall be computed as follows:

Pay Weight = (100.5 x Wet Wt. of Salt) divided by (100 + Percent of Moisture)

Test Method: Number 12

B. Magnesium Chloride Hexahydrate Only

The total moisture content of the magnesium chloride (both free and bound) shall not exceed 56%.

\*Unbound water is defined as that water that is not a normal part of the ingredients and becomes part of the product due to hygroscopic action.

Test Method: Number 12

- NOTE: The moisture content is judged as available free water. Organic Bases Corrosion Inhibitors that are used in the processes of making this product that impart a loss in weight (Organic Matter Weight Loss) when ran according to the prescribe test method but do not reflect the loss of available free water shall be limited to a maximum of 3% by weight. Products that exceed the 3% by weight limit shall be subject to the same equation as above with the limit being adjusted to 3%. Additionally, the use of said inhibitors may be used provided that the material remain free flowing, will not clump, cause hard caking and remains suitable for use. The use of these types of inhibitors may require additional testing to be provided by the bidder at the request of the PNS before approval to the qualified products list is granted. The amount of available water in the inhibitor and the base salt will be required along with a mass balance analysis of the two products to show the theoretical amount of free water that is available in the finished product.
- 5. Corrosion Control Inhibitor and Concentration

## **Chemical Product Category 5---Continued**

6. Product Must Contain No Less Than 10% Magnesium Chloride Hexahydrate by Weight.

This product will consist of 10% magnesium chloride hexahydrate (MgCl<sub>2</sub> +6H2O) as specified by weight. Weight of the magnesium chloride shall be calculated as a percent of the total mixture with zero percent <u>unbound water</u> \*. The manufacture shall establish unit densities and correlating weight for the product based on the zero percent of unbound water content at time of manufacturing. The required percentage of magnesium chloride (MgCl<sub>2</sub>) in the total mixture shall be based on the weight of magnesium chloride hexahydrate (MgCl<sub>2</sub>+6H2O).

#### Corrosion Inhibited Sodium Chloride Plus 20% Magnesium Chloride Specifications

In addition to the General Specifications the following requirements shall also apply:

The bidder must state the use of solid or liquid magnesium chloride. For liquid applications the manufacturer shall use at a minimum a 28% concentration of magnesium chloride. The manufacturer shall supply information as to what concentration of the magnesium chloride was used in the process.

1. Gradation of product shall be Type 1, Grade 2, for Sodium Chloride.

Test Method: Number 13

#### PHYSICAL REQUIREMENTS AND TOLERANCES

Sieve	Wt. %
Size	<u>Passing</u>
3/4"	100
#4	20 - 100
#8	10 - 60
#30	0 - 15

2. Anti-Caking agent will be included to insure that the material remains free from hard caking and suitable for its intended purpose.

Test Method: Number 14

**NOTE:** Salt for highway use is usually treated with either Ferric Ferrocyanide, also known as Prussian Blue, or Sodium Ferrocyanide, also known as Yellow Prussiate of Soda (YPS), to prevent the salt from caking. The amount of Prussian Blue added is 70 to 165 parts per million (PPM), equivalent to 0.33 to 1.14 pounds per ton of salt. YPS is added in the amount of 50 to 250 PPM, equivalent to 0.1 to 0.5 pounds per ton of salt. YPS is also used as an anti-caking agent in table salt, and has approval of the U.S. Food and Drug Administration. Based on exhaustive testing no evidence of toxicity was demonstrated. If used, the presence of these products will not be assessed towards the total cyanide concentration when testing this product. However, the total cyanide concentration of the original material must meet specifications. Information may be obtained from the Salt Institutes Highway Digest Publication.

Bidder may bid this product with or without the anti-caking agent. Bidders must note on the Sample Checklist if the sample does contain anti-caking agent or not. If the Bidder chooses not to add the anti-caking agent it does not prevent the bidder from assuring that the delivered product is in a free-flowing state.

## **Chemical Product Category 6---Continued**

3. Material must be clean and free from extraneous matter. The material must be homogenous or manufactured in such a manner to assure that the corrosion inhibitor, anti-caking agent and the chemical product does not segregate.

Test Method: Number 14

- 4. Moisture Content Of Sodium Chloride Only.
  - A. Sodium Chloride Only

The salt shall be dried to a maximum moisture content of 0.5 % (percent by weight). Water in excess of 0.5% of dry salt weight will not be paid for. The amount of salt to be paid for, when moisture exceeds 0.5% shall be computed as follows:

Pay Weight = (100.5 x Wet Wt. of Salt) divided by (100 + Percent of Moisture)

Test Method: Number 12

B. Magnesium Chloride Hexahydrate Only

The total moisture content of the magnesium chloride (both free and bound) shall not exceed 56%.

\*Unbound water is defined as that water that is not a normal part of the ingredients and becomes part of the product due to hygroscopic action.

Test Method: Number 12

- NOTE: The moisture content is judged as available free water. Organic Bases Corrosion Inhibitors that are used in the processes of making this product that impart a loss in weight (Organic Matter Weight Loss) when ran according to the prescribe test method but do not reflect the loss of available free water shall be limited to a maximum of 3% by weight. Products that exceed the 3% by weight limit shall be subject to the same equation as above with the limit being adjusted to 3%. Additionally, the use of said inhibitors may be used provided that the material remain free flowing, will not clump, cause hard caking and remains suitable for use. The use of these types of inhibitors may require additional testing to be provided by the bidder at the request of the PNS before approval to the qualified products list is granted. The amount of available water in the inhibitor and the base salt will be required along with a mass balance analysis of the two products to show the theoretical amount of free water that is available in the finished product.
- 5. Corrosion Control Inhibitor and Concentration

Test Method: Number 3

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## **Chemical Product Category 6---Continued**

6. Product Must Contain No Less Than 20% Magnesium Chloride Hexahydrate by Weight.

This product will consist of 20% magnesium chloride hexahydrate (MgCl<sub>2</sub> +6H2O) as specified by weight. Weight of the magnesium chloride shall be calculated as a percent of the total mixture with zero percent <u>unbound water \*</u>. The manufacture shall establish unit densities and correlating weight for the product based on the zero percent of unbound water content at time of manufacturing. The required percentage of magnesium chloride (MgCl<sub>2</sub>) in the total mixture shall be based on the weight of magnesium chloride hexahydrate (MgCl<sub>2</sub> +6H2O).

#### Solid Calcium Magnesium Acetate Specifications

In addition to the General Specifications the following requirements shall also apply:

1. Product will consist of Calcium Magnesium Acetate (CMA)

Only those ingredients that are normally found in high quality CMA will be acceptable. Any products that do not meet this requirement during the bid process will be immediately rejected unless scientific data shows the additional ingredients/ingredients result in an improvement to the product.

Test Method: Number 14

2. Calcium to magnesium mole ratio shall be 3 to 7

Test Method: Number 1

3. This product when liquefied at or near a 25% concentration shall not contain greater than 4.0 % (V/V) settleable solids and shall have ninety nine percent (99.0%) of the Solids Passing through a Number 10 sieve after being stored at <u>-12°C +/- 1°C (-10°F +/- 2°F)</u> for 168 hours (Seven days).

Test Method: Number 6

4. Moisture (free and hydration) shall not exceed 10%.

Test Method: Number 12

5. Product attrition shall be less than 2.5% with minimum dust generated on handling.

Test Method: Number 14 and any other tests deemed necessary.

6. Residual base shall be 0.30 milliequivalents base per gram of sample.

Test Method: Number 11

7. The pH of product in a 10% solution shall be 8 to 10.

Test Method: Number 4 except in this case a 10% solution will be used.

#### Non Corrosion Inhibited Solid Sodium Chloride Specifications

#### **CATEGORIES 8A, 8B, and 8C**

The Categories shall be defined as follows:

1. Category 8A	Dry Salt, Standard Gradation
A. Category 8A-B	Brining Salt
B. Category 8A-R	Road Salt
2. Category 8B	Wet Salt, Standard Gradation
3. Category 8C	Dry Salt, Fine Gradation
A. Category 8C-B	Brining Salt
B. Category 8C-R	Road Salt

In addition to the General Specifications, the following requirements shall apply.

- <u>Moisture Content Test Method No. 12</u> Category 8A – 0.5% Maximum Category 8B – 5.0% Maximum Category 8C – 0.5% Maximum
- <u>Insoluble Material- Test Method No. 22</u> Category 8A-R – 10.0 % Maximum Category 8B - 10.0% Maximum Category 8C-R - 10.0 % Maximum Category 8A-B – 1.0% Maximum Category 8C-B – 1.0% Maximum
- 3. Gradation Test Method No. 13

Type 1, Grade 2, with the following Gradation for each Sodium Chloride Category.

Category 8A and 8B		Category 8C	
Sieve	Wt. %	Sieve	Wt. %
Size	Passing	Size	Passing
3/4"	100	#4	100
#4	20 - 100	#100	0 - 3
#8	10 - 60		
#30	0 - 15		

4. Anti-Caking agent will be included to insure that the material remains free from hard caking and suitable for its intended purpose.

## **Chemical Product Category 8---Continued**

Test Method: Number 14

**NOTE:** Salt for highway use is usually treated with either Ferric Ferrocyanide, also known as Prussian Blue, or Sodium Ferrocyanide, also known as Yellow Prussiate of Soda (YPS), to prevent the salt from caking. The amount of Prussian Blue added is 70 to 165 parts per million (PPM), equivalent to 0.33 to 1.14 pounds per ton of salt. YPS is added in the amount of 50 to 250 PPM, equivalent to 0.1 to 0.5 pounds per ton of salt. YPS is also used as an anti-caking agent in table salt, and has approval of the U.S. Food and Drug Administration. Based on exhaustive testing no evidence of toxicity was demonstrated. If used, the presence of these products will not be assessed towards the total cyanide concentration when testing this product. However, the total cyanide concentration of the original material must meet specifications. Information may be obtained from the Salt Institutes Highway Digest Publication.

Bidder may bid this product with or without the anti-caking agent. Bidders must note on the Sample Checklist if the sample does contain anti-caking agent or not. If the Bidder chooses not to add the anti-caking agent it does not prevent the bidder from assuring that the delivered product is in a free-flowing state.

5. Material must be clean and free from extraneous matter. The material must be homogenous or manufactured in such a manner to assure that the corrosion inhibitor, anti-caking agent and the chemical product does not segregate.

Test Method: Number 14

6. Pay Weight Schedule for Excessive Moisture

#### Category 8A and 8C

The salt shall be dried to a maximum moisture content of 0.5 % (percent by weight). Water in excess of 0.5% of dry salt weight will not be paid for. The amount of salt to be paid for, when moisture exceeds 0.5% shall be computed as follows:

Pay Weight = (100.5 x Wet Wt. of Salt) divided by (100 + Percent of Moisture)

#### Category 8B

The salt shall be dried to a maximum moisture content of 5.0 % (percent by weight). Water in excess of 5.0% of dry salt weight will not be paid for. The amount of salt to be paid for, when moisture exceeds 5.0% shall be computed as follows:

Pay Weight = (105.0 x Wet Wt. of Salt) divided by (100 + Percent of Moisture)

#### **Chemical Product Category 8---Continued**

7. Pay Weight Schedule for Insoluble Residue

#### Category 8A-R, 8B, and 8C-R

The salt shall have a maximum insoluble residue of 10.0 % (percent by dry weight). Insoluble residue in excess of 10.0% of dry salt weight will not be paid for. The amount of salt to be paid for, when the insoluble residue exceeds 10.0% shall be computed as follows:

Pay Weight = (110.0 x Dry Wt. of Salt) divided by (100 + Percent Insoluble Residue)

#### Category 8A-B and 8C-B

The salt shall have a maximum insoluble residue of 1.0 % (percent by dry weight). Insoluble residue in excess of 1.0% of dry salt weight will not be paid for. The amount of salt to be paid for, when the insoluble residue exceeds 1.0% shall be computed as follows:

Pay Weight = (101.0 x Dry Wt. of Salt) divided by (100 + Percent Insoluble Residue)

## Corrosion Inhibited Liquid Sodium Chloride Specifications

In addition to the General Specifications the following requirements shall also apply:

1. Product must contain no less than 21% sodium chloride.

Test Method: Number 1 (Adapted to measure Sodium by emission spectroscopy.) Number 23

2. Weight per gallon will be established according to the specific gravity and percentage of sodium chloride contained in the product bid as indicated by the bidder.

Test Method: Number 2

3. Product will contain the corrosion control inhibitor in quantities not less than those indicated by the bidder. The finished deicing product, including corrosion inhibitors, must be completely accomplished at the original manufacturing plant location. Post adding of corrosion inhibitors or any other ingredients and splash mixing is unacceptable after the product has left the original manufacturing plant.

Test Method: Number 3

4. The pH must be 6.0 - 9.0

Test Method: Number 4

5. This chemical product shall not contain greater than 1.0% (V/V) Total Settleable Solids and shall have Ninety-nine percent (99.0%) of the Solids Passing through a Number 10 sieve after being stored at <u>-17.8°C +/- 1°C (0°F +/- 2°F)</u> for 168 hours (Seven days).

## Corrosion Inhibited Liquid Sodium Chloride Plus Calcium Chloride Specifications

In addition to the General Specifications the following requirements shall also apply:

1. Product must contain no less than 15% sodium chloride plus no less than 2% calcium chloride.

Test Method: Number 1 (Adapted to measure Sodium by emission spectroscopy) Number 23

2. Weight per gallon will be established according to the specific gravity and percentage of sodium chloride and calcium chloride contained in the product bid as indicated by the bidder.

Test Method: Number 2

3. Product will contain the corrosion control inhibitor in quantities not less than those indicated by the bidder. The finished deicing product, including corrosion inhibitors, must be completely accomplished at the original manufacturing plant location. Post adding of corrosion inhibitors or any other ingredients and splash mixing is unacceptable after the product has left the original manufacturing plant.

Test Method: Number 3

4. The pH must be 6.0 - 9.0

Test Method: Number 4

5. This chemical product shall not contain greater than 1.0% (V/V) Total Settleable Solids and shall have Ninety-nine percent (99.0%) of the Solids Passing through a Number 10 sieve after being stored at <u>-17.8°C +/- 1°C (0°F +/- 2°F)</u> for 168 hours (Seven days).

#### Corrosion Inhibited Liquid Chloride Blended Brines Specifications

In addition to the General Specifications the following requirements shall also apply:

1. Product must contain no less than 25% concentration of the total accumulation of chloride based salts in percent including Magnesium Chloride, Calcium Chloride, Sodium Chloride and Potassium Chloride. Any one individual chloride based salt shall exist in a concentration above 2% to be added to the total accumulated concentration.

Test Method: Number 1 (Adapted to measure Sodium and Potassium by emission spectroscopy.) Number 23

2. Weight per gallon will be established according to the specific gravity and total percentage of chloride blended brines contained in the product bid as indicated by the bidder.

Test Method: Number 2

3. Product will contain the corrosion control inhibitor in quantities not less than those indicated by the bidder. The finished deicing product, including corrosion inhibitors, must be completely accomplished at the original manufacturing plant location. Post adding of corrosion inhibitors or any other ingredients and splash mixing is unacceptable after the product has left the original manufacturing plant.

Test Method: Number 3

4. The pH must be 6.0 - 9.0

Test Method: Number 4

5. This chemical product shall not contain greater than 1.0% (V/V) Total Settleable Solids and shall have Ninety-nine percent (99.0%) of the Solids Passing through a Number 10 sieve after being stored at <u>-17.8°C +/- 1°C (0°F +/- 2°F)</u> for 168 hours (Seven days).

## PNS EXPERIMENTAL CATEGORY

The PNS Experimental Category is designed for potential products that do not fit the current chemical profiles of the already existing defined PNS categories.

The submitted experimental products shall meet the specified limits of the General Specifications including corrosion inhibition. The experimental products shall be analyzed for the informational requirements also listed in the General Specifications.

Products submitted for acceptance testing within the Experimental Category shall remain in this category until other similar products warrant a new category to be developed at the discretion of the PNS. The manufacturer shall submit all test results as required along with the following information:

Define the active ingredient that can be analytically measured.

Define the concentration of the active constituent at which the product will be manufactured.

Test protocols for analyzing the primary constituent.

For liquid products the manufacturer shall designate the appropriate temperature at which the Percent Total Settleable Solids and Percent Passing the No. 10 Sieve test shall be accomplished.

Once the testing information is completed the manufacturer shall then follow the protocols for submitting samples and testing information to the PNS for Quality Assurance Testing. Upon request of the PNS the manufacturer shall supply all additional testing information that may be deemed necessary to complete the review of the product before acceptance to a provisional standing is provided. Provisional standing will be imposed on products that have satisfactorily completed the standards of the PNS. Provisional standing will be issued for the products for a period not to exceed 12 months so that field testing and evaluations can be completed. Provided that the field testing and evaluations are determined to be successful the product will then be classified as a Qualified Product in the Experimental Category.

Field testing of the products for this category shall be conducted by the PNS members or by agencies within the Associations' domain. If other than a PNS member is conducting the testing the manufacture shall be responsible for collecting the field data and submitting it to the PNS for review. Field Data from Taper logs will be reviewed for the products ability to perform. Additionally, the names and telephone numbers of the individuals conducting the field testing and providing the taper logs shall be submitted so that the PNS can not only review questions of performance but also handling, storage, application information and any other information that the PNS feels is relevant regarding a product and its use.

## **INHIBITOR PRODUCT CATEGORY A-1**

## CORROSION INHIBITOR FOR SODIUM CHLORIDE (SALT) BRINE

This specification is for a liquid corrosion inhibitor for field addition to concentrated sodium chloride (salt) brine.

The finished corrosion inhibited sodium chloride shall have a minimum sodium chloride concentration of no less than 21% and shall have a Corrosion Percent Effectiveness Rating of 30% or less as tested by PNS specifications.

This liquid corrosion inhibitor when added to concentrated sodium chloride brine will provide a finished product that is compliant to all the General Provisions of the PNS Specifications.

The finished product shall provide eutectic temperature points equal to or lower than that of a standard uninhibited liquid sodium chloride brine of 23.3% concentration. The manufacture shall provide a eutectic temperature graph and table showing both eutectic curves of the finished product and the standard uninhibited liquid sodium chloride solution of 23.3% concentration for direct comparison. The graph shall be constructed according the specifications in Section II Sample Submittals.

For testing purposes, the inhibitor product shall be added to reagent grade sodium chloride brine prepared from distilled water meeting ASTM D 1193 Type II. The salt brine concentration will be prepared in a weight to weight ratio with water. The inhibitor concentration will be added as a volume to volume measurement to the brine solution. The sodium chloride brine and inhibitor concentrations will be prepared according to the inhibitor manufacturer's specifications and guidelines.

The inhibitor shall be capable of being homogenously mixed with the 23% to 24% concentration of sodium chloride brine and resulting in a finished product that does not separate or settle out.

The corrosion inhibitor product bid shall be flowable and have the capability to be mixed fully into the concentrated sodium chloride brine solution at a minimum temperature of  $15^{\circ}$  F.

Temperature Storage Class of Inhibitor: The corrosion inhibitor must be capable of being stored at a minimum temperature Class as delivered until time of use with no separation or settling.

Class 1: 10° F Class 2: 0° F

This chemical product shall not contain greater than 1.0% (V/V) Total Settleable Solids and shall have Ninety-nine percent (99.0%) of the Solids Passing through a Number 10 sieve after being stored at the designated Temperature Storage Class (+/-  $2^{\circ}F$ ) for 168 hours (Seven days).

## **INHIBITOR PRODUCT CATEGORY A-2**

## CORROSION INHIBITOR FOR SODIUM CHLORIDE PLUS CALCIUM CHLORIDE BRINE

This specification is for a field added liquid corrosion inhibitor to produce corrosion inhibited sodium chloride/calcium chloride brine.

The finished corrosion inhibited product shall have a minimum concentration of 15% sodium chloride and a minimum concentration of 2% calcium chloride. The product shall have a minimum of 10% inhibitor added to the product. The finished product shall have a Corrosion Percent Effectiveness Rating of 30% or less as tested by PNS specifications.

The finished product shall provide eutectic temperature points equal to or lower than that of a standard uninhibited liquid sodium chloride brine of 23.3% concentration. The manufacture shall provide a eutectic temperature graph and table showing both eutectic curves of the finished product and the standard uninhibited liquid sodium chloride solution of 23.3% concentration for direct comparison. The graph shall be constructed according the specifications in Section II Sample Submittals.

The process by which this is achieved is classified into the following Types:

Type I – The corrosion inhibitor contains sufficient calcium chloride that additional calcium chloride is not required to be added to the salt brine.

Type II – The corrosion inhibitor, salt brine, and calcium chloride are added separately.

For testing purposes of Type I inhibitors, the inhibitor product shall be added to the concentrated liquid salt brine prepared from reagent grade sodium chloride and distilled water meeting ASTM D 1193 Type II. The salt brine concentration will be prepared in a weight to weight ratio with distilled water. The inhibitor concentration will be added as a volume to volume measurement to the brine solution. The sodium chloride brine and inhibitor concentrations will be prepared according to the inhibitor manufacturer's specifications and guidelines.

For Testing purposes of Type II inhibitors, the inhibitor product shall be added to a mixture of concentrated salt brines prepared from reagent grade sodium chloride and calcium chloride, and distilled water meeting ASTM D 1193 Type II. The salt brine concentrations will be prepared in a weight to weight ratio with distilled water. The inhibitor concentration will be added as a volume to volume measurement to the brine solution. The brine and inhibitor concentrations will be prepared according to the inhibitor manufacturer's specifications and guidelines.

The inhibitor shall be capable of being homogenously mixed with the 23% to 24% concentration of sodium chloride brine and resulting in a finished product that does not separate or settle out.

The corrosion inhibitor product bid shall be flowable and have the capability to be mixed fully into the concentrated brine solution at a minimum temperature of 15° F.

Storage Class of Inhibitor: The corrosion inhibitor must be capable of being stored at a minimum temperature Class as delivered until time of use with no separation or settling.

Class 1: 10° F Class 2: 0° F

This chemical product shall not contain greater than 1.0% (V/V) Total Settleable Solids and shall have Ninety-nine percent (99.0%) of the Solids Passing through a Number 10 sieve after being stored at the designated Temperature Storage Class (+/- 2°F) for 168 hours (Seven days).

## **INHIBITOR PRODUCT CATEGORY A-3**

## CORROSION INHIBITOR FOR SODIUM CHLORIDE (SALT) BRINE

This specification is for a liquid corrosion inhibitor for field addition to concentrated sodium chloride (salt) brine.

The finished corrosion inhibited sodium chloride shall have a minimum sodium chloride concentration of no less than 15% and have a minimum corrosion inhibitor concentration of no less than 15%. The finished product shall have a Corrosion Percent Effectiveness Rating of 30% or less as tested by PNS specifications.

The finished product shall provide eutectic temperature points equal to or lower than that of a standard uninhibited liquid sodium chloride brine of 23.3% concentration. The manufacture shall provide a eutectic temperature graph and table showing both eutectic curves of the finished product and the standard uninhibited liquid sodium chloride solution of 23.3% concentration for direct comparison. The graph shall be constructed according the specifications in Section II Sample Submittals.

This liquid corrosion inhibitor when added to concentrated sodium chloride brine will provide a finished product that is compliant to all the General Provisions of the PNS Specifications.

For testing purposes, the inhibitor product shall be added to a salt brine prepared from reagent grade sodium chloride and distilled water meeting ASTM D 1193 Type II. The salt brine concentration will be prepared in a weight to weight ratio with water. The inhibitor concentration will be added as a volume to volume measurement to the brine solution. The sodium chloride brine and inhibitor concentrations will be prepared according to the inhibitor manufacturer's specifications and guidelines.

The inhibitor shall be capable of being homogenously mixed with the 23% to 24% concentration of sodium chloride brine and resulting in a finished product that does not separate or settle out.

The corrosion inhibitor product bid shall be flowable and have the capability to be mixed fully into the concentrated sodium chloride brine solution at a minimum temperature of 15° F.

Storage Class of Inhibitor: The corrosion inhibitor must be capable of being stored at a minimum temperature Class as delivered until time of use with no separation or settling.

Class 1: 10° F Class 2: 0° F

. This chemical product shall not contain greater than 1.0% (V/V) Total Settleable Solids and shall have Ninety-nine percent (99.0%) of the Solids Passing through a Number 10 sieve after being stored at the designated Temperature Storage Class (+/-  $2^{\circ}$ F) for 168 hours (Seven days).

Test Method: Number 6

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#### VI. TEST METHODS

#### **1.** Percent Concentration of Active Ingredient In The Liquid

Test Method: Atomic Absorption or Inductively Coupled Plasma Spectrophotometry as described in "Standard Methods for the Examination of Water and Waste Water", APHA-AWWA-WPCF is acceptable. Test Method "A" in Appendix "A" is used to determine percent concentration of Calcium Chloride or Magnesium Chloride by Atomic Absorption. The operator should be aware that the high solids content of the samples can present special considerations when conducting the analysis.

#### 2. Weight Per Gallon

Test Method: Specific Gravity by ASTM D 1429 Test Method A - Pycnometer at 20° C +/- 1° C.

#### 3. Corrosion Control Inhibitor Presence and Concentration

Test Method: The Materials Laboratory may use the test procedures provided by the bidder or manufacture for testing quantitative concentrations of additives. These same tests can then be used to verify that materials being delivered are the same as those previously tested and approved in the bid process.

#### <u>4. pH</u>

Test Method: ASTM D 1293 except a dilution shall be made of 1 part chemical product to 4 parts distilled water before attempting a reading.

#### 5. Corrosion Rate

Test Method: NACE Standard TM0169-95 (1995 Revision) as modified by PNS. This procedure is listed as Test Method "B" in Appendix A.

#### 6. Percent Total Settleable Solids and Percent Solids Passing a 10 Sieve

Test Method: This procedure is listed as Test Method "C" in Appendix A.

#### 7. Total Phosphorus

Test Method: Total Phosphorous as described in "Standard Methods for the examination of Water and Waste Water", APHA-AWWA-WPCF.

#### 8. Total Cyanide

Test Method: Total Cyanide as described in "Standard Methods for the examination of Water and Waste Water", APHA-AWWA-WPCF.

#### 9. Total Arsenic, Barium, Cadmium, Chromium, Copper, Lead, Selenium and Zinc.

Test Method: Atomic Absorption Spectrophotometry or Plasma Emission Spectroscopy as described in "Standard Methods for the examination of Water and Waste Water", APHA-AWWA-WPCF.

#### **10. Total Mercury**

Test Method: Cold Vapor Atomic Absorption Spectrophotometry as described in "Standard Methods for the examination of Water and Waste Water", APHA-AWWA-WPCF.

#### **<u>11. Milliequivalents OR "meq"</u>**

Test Method: This is a measure of the amount of unreacted base in the product. "meq" means milliequivalents or the milligrams of acetic acid to neutralize 1 gram of unreacted base.

Method for measuring unreacted base is a standard acid/base titration procedure. A fixed volume of acid (30 ml of 0.1 N HCl) is added to 1 gram sample of CMA. The excess acid is titrated with a standard base (0.1 N NaOH) to phenolphthalein endpoint, pH of 8.6.

## 12. Moisture Content Of Solid Chemical Products.

Test Method: According to ASTM E 534

#### 13. Gradation

Test Method: Gradation shall be ran according to ASTM D 632. The sample size shall be a minimum of 300 grams and be hand shaken through each sieve until the sample has been adequately processed. Caution: Care should be used when running the gradation test, as the salt is very soft and can be resized by over shaking. Salts that contain sticky organic matter inhibitors may require additional attention with a rubber policeman to insure that the sample passes the screens correctly as the sticky inhibitors will tend to clump up smaller particles of salt and prohibit them from being analyzed correctly.

#### **14.** Visual Inspection and Field Observations.

Test Method: Visual inspection and field observations to assure that the material remains clean and free of extraneous matter, free from hard caking, does not segregate, and remains suitable for the intended purpose and as otherwise outlined in Section IV. <u>NOTE</u>: Purchaser may use any laboratory test method necessary to verify conclusions from visual inspections.

#### 15. Toxicity Test

Test Method: According to "Short-Term Methods for Estimating the Chronic Toxicity of Effluent and Receiving Waters to Freshwater Organisms", Third Edition, EPA-600/4-91/002.

#### 16. Ammonia - Nitrogen

Test Method: Ammonia as described in "Standard Methods for the examination of Water and Waste Water", APHA-AWWA-WPCF.

#### 17. Total Kjeldalh Nitrogen

Test Method: Total Kjeldalh Nitrogen as described in "Standard Methods for the examination of Water and Waste Water", APHA-AWWA-WPCF.

#### 18. Nitrate and Nitrite as Nitrogen

Test Method: Nitrate and Nitrite as Nitrogen as described in "Standard Methods for the examination of Water and Waste Water", APHA-AWWA-WPCF.

#### 19. Biological Oxygen Demand

Test Method: Biological Oxygen Demand as described in "Standard Methods for the examination of Water and Waste Water", APHA-AWWA-WPCF.

## 20. Chemical Oxygen Demand

Test Method: Chemical Oxygen Demand as described in "Standard Methods for the examination of Water and Waste Water", APHA-AWWA-WPCF.

## 21. Frictional Analysis

Test Method: Frictional Analysis shall be conducted on products that have been applied at the prescribe application rate to a pavement surface within a sealed and controlled humidity chamber. The frictional coefficient shall be measured on pavement surface as the humidity in the chamber is lowered and raised over the course of time. The data shall show a plot of the humidity curve and a plot of the coefficient of friction curve over time. The device that measures the friction coefficient shall be calibrated and certified prior to use on the sample analysis.

## 22. Insoluble Material

Test Method: ASTM E534 "Standard Test Methods for Chemical Analysis of Sodium Chloride". The method shall be modified by dissolving 100 grams of the sodium chloride sample into the prescribed volume and filtering the entire solution through a Whatman No. 541(or equal), 125 mm diameter filter paper seated in a Buchner Funnel.

#### 23. Chloride

Test Method: Chloride as described in "Standard Methods for the examination of Water and Waste Water", APHA-AWWA-WPCF.

#### VII. PRODUCT REJECTION AND PRICE ADJUSTMENTS

## ATTENTION: PLEASE REFER TO EACH INDIVIDUAL AGENCIES SPECIFICATIONS FOR PRODUCT REJECTIONS AND PRICE ADJUSTMENTS.

#### VIII. BID EVALUATION PROCESS

#### A. <u>BID PREFERENCES FOR HIGHER CONCENTRATIONS</u> (Approved Liquid Chemical Products)

STEP 1: Best buy (FOB delivery destination) based on percentage of active chemical in the product will be determined by the following formula. <u>Bidder Quoted Concentrations</u> (BQC) and price per ton will be used for calculations. Delivered Price/Concentration Percentage equals the best buy factor for this step of the process. (The bidders quoted concentration will be used in the calculation.)

Example:

a. 60.00/27% = 222.22 best buy factor

b. 65.00/30% = 216.67 best buy factor

Example "b" at the higher purchase price per ton, with the higher concentration, and with the lower best buy factor would be selected if this were the final step.

# **B.** <u>**BID PREFERENCES FOR SUPERIOR CORROSION INHIBITION</u> (Approved Liquid and Solid Chemical Products)**</u>

**STEP 2:** Bid preferences based on the corrosion inhibiting ability of a product as demonstrated by the PNS's laboratories and verified by field applications will be applied from the values as shown in the following table. The values shown in the table under "Value Added" are used to reduce the calculated best buy factor (see above) to arrive at the final calculation/determination of best buy.

PERCENT CORROSION EFFECTIVENESS RANGES	VALUE ADDED
25.0 to 30.0	0.00
20.0 to 24.9	40.00
15.0 to 19.9	60.00
10.0 to 14.9	80.00
5.0 to 09.9	100.00
4.9 and less	150.00

Example:

As noted above in step 1, based on concentration calculations, product "b" resulted in the lowest best buy factor. When corrosion inhibiting values are considered, the calculations will be as follows. Product "a" has a corrosion value of 15.5%, which equates to 60.00 added value points while product "b" displayed a corrosion value of 27.0%, which results in no added value points. See the following:

a. \$60.00/27% = 222.22 - 60.00 = 162.00 our final best buy factor.

b. 65.00/30% = 216.67 - 00.00 = 216.67 our final best buy factor.

Example "a" with the lower concentration but with higher corrosion inhibiting value would be determined to be the best buy in the final step.

Acceptance of bids will be based on approved PNS laboratory results. Final determination of the liquid chemicals products will be based on the "final best buy factor" calculated from the combination of the lowest cost per percent concentration of liquid chemical and credit for corrosion inhibiting ability as specified in Steps 1 & 2. On solid chemical products, only the value added for corrosion inhibiting performance will be used in the "final best buy factor" determination process as specified in Step 2. Bids will be awarded for the lowest "final best buy factor" for each category and to each designated location or zone.

## IX. QUALIFIED PRODUCTS LIST

Purchased products that appear on the Qualified Products List may be tested for compliance to the material that was originally submitted for qualification. The agency has the right to conduct this testing at its own will. The most current Qualified Products List can be viewed at the PNS web site location of <u>http://www.wsdot.wa.gov/partners/pns/</u> or by contacting one of the PNS members.

## X. CHEMICAL PRODUCT SUBMISSION FOR THE QUALIFIED PRODUCTS LIST

The PNS member who is conducting the qualification testing has the right to test for verification or to accept the product as approved. Bidders of samples to be tested for acceptance to the Qualified Product List shall complete all the information and submit all the required documentation as specified in these specifications. Two One-gallon samples of the bid product shall accompany the required information for qualification testing.

## XI. PRODUCT SAMPLE CHECKLIST

All samples that are submitted must be accompanied with the Product Sample Checklist if they are to be considered for evaluation to the Qualified Product List. Fill in blanks with yes, no, or what is appropriate. If something does not apply, use N/A. Do not leave blanks. Blanks will be considered missed information and may be cause for rejection. Type or print clearly in ink. All documents must be clear and legible. If unreadable, it may be rejected.

Bidder's response to the following items will be considered representative of their product. During qualification testing of the submitted sample the liquid products cannot deviate from the percent concentration by more than minus one full percentage of the bidder quoted concentration as indicated below. If the submitted sample exceeds this deviation tolerance, that product will be disqualified. **During a bid opportunity the submitted Percent Concentration and the Percent Effectiveness will be compared to the approved product test results for verification. If different, the qualification results that appear on the PNS Qualified Products List will be used to determine the "final best buy factor".)** At no time will any sample be allowed to be below the minimum concentration requirement for that product as stated in these specifications.

# PRODUCT SAMPLE CHECKLIST

Bi	idder Information	
1.	Name of bidding company?	
2.	Mailing Address:	
3.	Email Address:	
4.	Phone number with area code:	
5.	Fax Number with area code:	
6.	Name of company contact:	
Ch	hemical Product Information (Categories 1-11 or Experimental)	
1.	Which chemical product category is your chemical to be sold under?	
2.	What is the name of the product?	
3.	The product is manufactured by	
4.	If the product is a liquid what is the percent concentration of the product? %.** (**This is the Bidder Quoted Concentration <u>NO</u> ranges please. If a range is used, the low bidder specified concentration will be used for cost analysis.)	vest
5.	Corrosion inhibited products have a Percent Effectiveness determined to measure the products corrosion rate on steel. What is the Percent Effectiveness of the Product?%	)
In	hibitor Product Information (Inhibitor Categories A1-A3)	
1.	Which inhibitor product category is your chemical to be sold under?	
2.	What is the name of the product?	
3.	The product is manufactured by?	
4.	What is the finished concentration(s) of the brine(s)?	
5.	What is the percent volume of the inhibitor to be added to the brine(s)?	
6.	What is the corrosion Percent Effectiveness of the finished product?	
7.	What temperature class is the product (1 or 2)?	
8.	If the product is submitted for category A2, what Type is it (I or II)?	
In	formation to be included with all submittals.	
1.	Product Data Sheet:	
2.	Material Safety Data Sheets (MSDS) for the product and the corrosion inhibitor:	
3.	Specific gravity information for liquid products as required:	
4.	Eutectic Temperature chart and graphs:	
5.	pH data (liquid products only):	
6.	Does your product contain an organic matter based corrosion inhibitor?	
7.	If yes, complete and submit the required information on the inhibitor as specified within these specifications.	

- 8. Waiver of pH requirements being requested? Yes \_\_\_\_\_ No \_\_\_\_\_
- 9. Percentage of organic matter present in your material?
- 10. Analytical results of all specified and informational chemical constituents as specified in the General Specifications, and for the specific category for which application is being made.
- 11. Toxicity Report \_\_\_\_\_
- 12. Frictional Analysis Report
- 13. Corrosion test data for corrosion inhibited products\_\_\_\_\_
- 14. Proprietary information regarding the corrosion inhibitor shall be included in a separate sealed envelope and marked in large bold lettering "Confidential Information".
- 15. Analytical testing procedures for verifying corrosion inhibitor concentration.
- 16. Two each one gallon containers samples of the product included with submittal.

#### **Experimental Products**

- 1. In addition the information contained above the following information is required.
- 2. Identify the primary active ingredient that the product can be measured for \_\_\_\_\_
- 3. Test protocols for testing the main ingredient \_\_\_\_\_
- 4. For liquid products, what is the lowest temperature that the material can be stored to while meeting the requirements of the Settleable Solids and Percent Passing the No. 10 sieve?

Have you completely read the PNS specifications and included all the required information into the submittal package? Yes \_\_\_\_\_ No \_\_\_\_\_

Signature of the Individual making the submission \_\_\_\_\_

Date of Submission \_\_\_\_\_

Please send all information to the following address:

Attention: Ron Wright Idaho Transportation Department Materials Section P.O. Box 7129 Boise, Idaho 83707-1129

Please ship all samples to the following address:

Attention: Ron Wright Idaho Transportation Department Materials Section 3311 W. State Street Boise, Idaho 83703-5879

#### XII. BID AND SAMPLE DELIVERY

All bids and samples shall be delivered by the time and date of the bid opening. Bids and samples that are received late will be rejected and not tested. Mark all samples submitted to the Laboratory in large black lettering as "<u>BID SAMPLES-TIME CRITICAL</u>".

#### XIII. BID SCHEDULE

The following quantities of chemical products are projected from use for the terms of this contract. These quantities are estimates to be used for bidding purposes only. They are not guaranteed deliverable quantities as the winter weather can and does change and quantities may be less or more than what is being represented. Bidders can bid their approved products but are limited to two new chemical product submissions per category. **Bids will be awarded for the lowest "final best buy factor" for each category (if applicable) and to each designated Area.** All prices are to be bid per ton and based on BULK DELIVERY, FOB point of delivery. If your are not entering a bid for an Area of the selected category enter a "No Bid" for that line item.

## LIQUID CHEMICAL PRODUCTS

The liquid portion of this contract will be bid based on the following locations within an Area. These locations are the sites of delivery. The unit price bid for each Area will be the price of delivery to all location within the Area and will be used in the analysis for the "Final Best Buy Factor". <u>The bid will</u> be award based on the lowest "Final Best Buy Factor" of each category per Area.

Identify the Category for which you are bidding and provide the product name, the name of your company and the Vendor Quoted Concentration of the Product.

Category \_\_\_\_\_

Product Name\_\_\_\_\_

Bidders Name\_\_\_\_\_

Vendor Quoted Concentration of Product\_\_\_\_\_

# ATTACHED AGENCY BID LIST FOR AREAS, LOCATIONS AND QUANTITES

## SOLID CHEMICAL PRODUCTS

The solid portion of this contract will be bid based on the following locations within an Area. These locations are the sites of delivery. The unit price bid for each Area will be the price of delivery to all location within the Area and will be used in the analysis for the "Final Best Buy Factor". <u>The bid will</u> be award based on the lowest "Final Best Buy Factor" of each category per Area (if applicable).

Identify the Category for which you are bidding and provide the product name and the name of your company.

Category \_\_\_\_\_

Product Name\_\_\_\_\_

Bidders Name\_\_\_\_\_

#### DOES YOUR PRODUCT CONTAIN AND ANTICAKING AGENT? (Circle One) YES NO

# IF YOUR PRODUCT DOES CONTAIN AN ANTICAKING AGENT PLEASE PROVIDE THE FOLLOWING INFORMATION:

#### AMOUNT OF ANTICAKING AGENT ADDED PER TON OF PRODUCT:

# WHAT IS THE NAME OF THE ANTICAKING AGENT ARE YOU ADDING:

# ATTACHED AGENCY BID LIST FOR AREAS, LOCATIONS AND QUANTITES

# **INDEX**

TEST METHOD "A" –	<b>Concentration Percentage of Active Ingredient In</b> Liquid Chemical Products
TEST METHOD "B" –	Corrosion Rate As Conducted From The NACE Standard TM0169-95 (1995 Revision) As Modified By The Pacific Northwest States
TEST METHOD "C" –	Percent Total Settleable Solids And Percent Solids Passing A No. 10 Sieve

# **TEST METHOD A**

# **Concentration Percentage of Active Ingredient In Liquid Chemical Product**

#### I. Test Method

Atomic Absorption Spectrophotometry as described in "Standard Methods for the Examination of Water and Waste Water", APHA-AWWA-WPCF

## **II.** Apparatus

Atomic Absorption Spectrophotometer 250, 500 ml Graduated Cylinders 2000 ml Beaker 100, 500, 1000 ml Volumetric Flasks 5, 10, 15, 20, 25, 30 ml Volumetric Pipets (Class A) 100 microliter Eppendorf Pipet

## **III. Reagents**

ASTM D 1193 Type II Distilled Water 1000 ppm Calcium Stock Solution 1000 ppm Magnesium Stock Solution Concentrated Hydrochloric Acid (HCl) Concentrated Nitric Acid (HNO<sub>3</sub>) Lanthanum Oxide (La<sub>2</sub>O<sub>3</sub>)

## IV. Preparation of Lanthanum Chloride; Calcium Chloride and Magnesium Chloride Calibration Standards and Blanks; Quality Control Solutions; and Calcium Chloride and Magnesium Chloride Deicer Solutions.

#### 1. Preparation of 10% Lanthanum Chloride Stock Solution

In a 2000 ml beaker add 200 ml of distilled water to 117.28 g of Reagent Grade Lanthanum Oxide. While stirring, **very slowly** add 500 ml of concentrated HCl (25 ml at a time). <u>CAUTION!</u> This reaction is extremely violent. Care should be taken so the solution does not overflow the beaker. When the solution has cooled to room temperature, transfer to a 1000 ml volumetric flask and dilute to volume with distilled water. (Lanthanum Chloride is the Ionization Suppressant used in determining Calcium and Magnesium concentrations by Atomic Absorption).

## 2. Calcium and Magnesium Chloride Calibration Standards

Calcium

## A. <u>100 ppm Calcium Stock Solution for Dilutions</u>

Pipet 10 ml of the 1000 ppm Calcium reagent solution into a 100 ml volumetric flask. Using an Eppendorf pipet add 0.1 ml concentrated HNO3 acid and dilute to volume with distilled water.

B. Calcium Standards for Calibration (20, 25, 30 ppm)

Pipet aliquot's of 20, 25, and 30 ml of the above 100 ppm Calcium stock solution into three different 100 ml volumetric flasks. Add 5 ml of the 10% Lanthanum Chloride solution to each flask before diluting to volume with distilled water. The standard solutions should be prepared daily.

## Magnesium

- A. <u>100 ppm Magnesium Stock Solution for Dilutions</u> Pipet 10 ml of the 1000 ppm Magnesium reagent solution into a 100 ml volumetric flask. Using an Eppendorf pipet add 0.1 ml concentrated HNO<sub>3</sub> acid and dilute to volume with distilled water.
- B. <u>Magnesium Standards for Calibration (10, 15, 20 ppm)</u> Pipet aliquot's of 10, 15, and 20 ml of the above 100 ppm Magnesium solution into three separate 100 ml volumetric flasks. Add 5 ml of the 10% Lanthanum Chloride solution to each flask before diluting to volume with distilled water. The standard solutions should be prepared daily.

## 3. Blank Solution

A. <u>Blank Solution for Calibration</u> Pipette 5 ml of 10% Lanthanum Chloride solution into a 100 ml volumetric flask and dilute to volume with distilled water. The blank solution should be prepared daily.

## 4. Quality Control Solutions

- A. <u>Calcium Quality Control Check</u> Weigh 0.6762 g pre-dried CaCO<sub>3</sub> and place into a 1000ml volumetric flask. Add 1 ml of concentrated HNO<sub>3</sub> and dilute to volume with distilled water. From this solution, pipette 10 ml into a 100 ml volumetric flask, add 5 ml of the 10% Lanthanum Chloride solution and bring to volume with distilled water. This will be the working Quality Control Standard and have a value of **27.10 ppm Calcium**. (Note: The 27.10 ppm Calcium concentration is equal to a 30% brine concentration of Calcium Chloride based on a 2.5 gram sample size.)
- B. <u>Magnesium Chloride Quality Control Check</u> Weigh 1.5056g (nondried) MgCl<sub>2</sub>-6H<sub>2</sub>O and place into 1000 ml volumetric flask. Add 1 ml of concentrated HNO<sub>3</sub> and dilute to volume with distilled water. From this solution, pipette 10 ml into a 100 ml volumetric flask, add 5 ml of the 10% Lanthanum Chloride solution and bring to volume with distilled water. This will be the working Quality Control Standard and have a value of **18.00 ppm Magnesium**.

Note: (Note: The 18.00 ppm Magnesium concentration is equal to a 28.2% brine concentration of Magnesium Chloride based on a 2.5 gram sample size.)

# V. Preparation of Liquid Chemical Products Sample Solution

## Solution A

1. Weigh approximately 2.500 grams of the liquid chemical product into a tared 500 ml volumetric flask. Record the sample weight to the nearest mg for final calculations. Add l ml HNO<sub>3</sub>. <u>Rinse the neck of the volumetric flask with a slight amount of distilled water and allow the sample to digest for one hour.</u> Dilute to volume with distilled water. Label as solution A.

# Solution B (Working Chemical Product Solution)

- 2. Pipette 5 ml of Solution A into a 100 ml volumetric flask. Add 5 ml of 10% Lanthanum Chloride solution and dilute to volume with distilled water. Label as solution B (Dilution factor of 20).
- 3. Repeat Step 2 so that each chemical product sample has a duplicate working solution.

# VI. Atomic Absorption Spectrophotometer Operation

## Calcium

- 1. Set up the spectrophotometer (absorption) with the Calcium lamp using a wavelength setting of 422.4 nm, and a slit width of 0.2 nm. An Air-Acetylene flame should be used with the 10 cm burner head set at a 45<sup>0</sup> angle. The flame, burner, and instrument are to be optimized for best detection.
- 2. Calibrate the instrument using the blank, 20 ppm, 25 ppm, and 30 ppm standards for Calcium.
- 3. Run the Calcium Quality Control solution. This result must be within plus or minus 0.20 ppm of the known 27.10 ppm concentration before proceeding.
- 4. Once the Quality Control solution is within allowable limits, run the chemical product samples and their duplicates and record the results.
- 5. Run the Calcium Quality Control solution again to assure accurate results.
- 6. Following the analysis calculate the percent concentration of the sample and the duplicate sample for each chemical product using the following formulas. These test results must be repeatable within plus or minus 0.3% concentration of each other to be acceptable for reporting. If the results are outside this allowable limit, perform the dilutions over and retest until the samples are repeatable within the 0.3% limit.

## <u>Magnesium</u>

- 1. Set up the spectrophotometer (absorption) with the Magnesium lamp using a wavelength setting of 285.4 nm, and a slit width of 0.2 nm. An Air Acetylene flame should be used with the 10 cm burner head set at a  $45^{0}$  The flame, burner, and instrument are to be optimized for best detection.
- 2. Calibrate the instrument using the blank, 10 ppm, 15 ppm, and 20 ppm standards for Magnesium.
- 3. Run the Magnesium Quality Control solution. This result must be within plus or minus 0.15 ppm of the known 18.00 ppm concentration before proceeding.

- 4. Once the Quality Control solution is within allowable limits, run the chemical product samples and their duplicates and record the results.
- 5. Run the Magnesium Quality Control solution again to assure accurate results.
- 6. Following the analysis calculate the percent concentration of the sample and the duplicate sample for each chemical product using the following formulas. These test results must be repeatable within plus or minus 0.3% concentration of each other to be acceptable for reporting. If the results are outside this allowable limit, perform the dilutions over and retest until the samples are repeatable within the 0.3% limit.

## **VII.** Calculations

Calculations for CaCl<sub>2</sub> base on a sample weighing 2.550 grams :

 $Factor = (110.99 \text{ CaCl}_2)(1\%)(Dilution \text{ factor})(Initial \text{ vol.}) = 2.7692$   $(40.08 \text{ Ca}) \quad (10,000 \text{ ppm})$ 

% 
$$CaCl_2 = (X ppm from AA)(Factor)$$
  
grams of sample

Example: 
$$(28.20 \text{ PPM})(2.7692) = 30.6\% \text{ CaCl}_2$$
  
2.5500 g chemical product

Calculations for MgCl<sub>2</sub> base on a sample weighing 2.550 grams:

Factor = 
$$\frac{(20) \quad (500 \text{ ml})}{(24.305 \text{ Mg}) \quad (10,000 \text{ ppm})} = 3.9173$$

%  $MgCl_2 = (X ppm from AA)(Factor)$ grams of sample

Example:  $(18.87 \text{ ppm})(3.9173) = 29.0\% \text{ MgCl}_2$ 2.5500 g chemical product

# **TEST METHOD B**

# <u>Corrosion Rate As Conducted From The NACE Standard TM0169-95 (1995 Revision) And As</u> <u>Modified By The Pacific Northwest States</u>

Products that are submitted to meet the Corrosion Rate Test and to have a Percent Effectiveness determined shall be tested according to the National Association of Corrosion Engineers (NACE) Standard TM0169-95 as modified by the PNS. The PNS has modified this procedure so that the test procedure uses 30 ml of a 3% chemical product solution as received per square inch of coupon surface area for the corrosion test. Corrosion inhibited chemical product must prove to have a Percent Effectiveness value of at least 70% less than Sodium Chloride (salt) to be acceptable.

# I. PREPARATION OF THE COUPONS

The coupons used are 1/2" (approximately 1.38 in. x 0.56 in. x 0.11 in.) flat steel washers displaying a density of approximately 7.85 grams per cubic centimeter. (Note: No galvanized coupons are allowed to be used even after removing the zinc with acid. Hot dipped galvanization creates a Fe-Zn metallurgical surface bond that changes the characteristics of the steel. Coupons must meet ASTM F 436, Type 1, with a Rockwell Hardness of C 38-45. Each coupons used in the test procedure is subjected to the following process to assure accuracy in test results.

- Examine each coupon for metallurgical abnormalities and reject those that are suspect to flaws.
- All coupons are tested for Rockwell Hardness of C 38-45; coupons having hardness outside of this range are rejected.
- Acceptable coupons are stamped for identification.
- Wipe with suitable solvent to remove grease and oil.
- Coupons are placed in a 1 Liter plastic bottle with 1 tablespoon of phosphate free labware grade detergent (such as Contrex) and 200 mL of warm tap water. Gently agitated for fifteen minutes then rinse coupons with tap water. Coupons are then placed into a 1 Liter plastic bottle with 3 tablespoons of Bon Ami cleaning powder and 200 ml warm tap water. Gently agitate the coupons for fifteen minutes drain and rinse with distilled water and wipe dry.
- Coupons are acid etched with 1 + 1 HCl for 3 minutes.
- The coupons are then quickly rinsed with tap water, distilled water, wiped dried and placed in chloroform.
- When the coupons are removed from the chloroform for use, they are place on a paper-lined tray (not touching each other) and allowed to air dry in a ventilated hood for a minimum of 15 minutes.
- Coupons are measured as specified. (Note: If latex gloves are not worn during measuring, the coupons should be rinsed again and dried as prescribe above prior to weighing. This will remove any oils that may be transferred to the coupons.)
- Each coupon shall be weighed to a constant weight. The constant weight shall be two consecutive weighings of each coupon within a minimum of 0.5 milligrams of each other. Removal of incidental flash rusting prior to weighing is not necessary.

Three coupons are used in each flask for testing the chemical product solutions, and determining the distilled water and sodium chloride control standard values.

# **II. MEASURING OF THE COUPONS**

The outside diameter, inside diameter, and the thickness of each coupon is measured twice at 90 degrees from each initial reading and the averages calculated for each measurement. The averages are then used to calculate the surface area of each coupon with the following formula:

 $A = (3.1416/2)(D^2 - d^2) + 3.1416(t)(D) + 3.1416(t)(d)$ Where D = average outside diameter d = average inside diameter t = average thickness

Example:

$$\begin{split} A &= (1.5708)(1.9044 - 0.3136) + 0.4768949 + 0.1935226\\ A &= (1.5708)(1.5908) + 0.4768949 + 0.1935226\\ A &= 2.4988286 + 0.4768949 + 0.1935226\\ A &= 3.1692461 \text{ square inches (Total surface area of the coupon.)}\\ A &= 3.17 \text{ square inches} \end{split}$$

# **III. PREPARATION OF THE SOLUTIONS**

ASTM D 1193 Type II distilled water is used to prepare each solution, blank, and control standard. The Sodium Chloride (NaCl) used to prepare the salt standard shall be of "ANALYZED REAGENT GRADE" quality.

A 3% solution of NaCl is prepared by weight, using the reagent grade salt and distilled water (W/V).

A 3% solution of each chemical product to be tested is prepared using distilled water to dissolve and or dilute the chemical product. For liquid chemical products, three parts liquid chemical product (as received) is mixed with 97 parts distilled water to produce the test solution (V/V). If the chemical product is a dry product, then the 3% solution is made by weight (W/V).

All solutions including the distilled water blank are covered an allowed to sit a minimum of 12 hours to stabilize and reach equilibrium, ensure solubility and to account for any reactivity that may occur.

# IV. THE CORROSION TEST

Approximately 300 milliliters (actual volume is determined by the surface area of test coupons) of each solution as mixed with distilled water and is put into a 500 milliliter Erlenmeyer flask. Each flask is stoppered with a rubber stopper that has been drilled to allow a monofilament line to run through it. The hole in the rubber stopper is 3-4 millimeters in diameter. The hole should not be greater than 4 REVISION 12-10

millimeters as it will allow the vapor phase within the neck of the flask to vent excessively and can skew the results. One end of the line is attached to the up/down bar of the corrosion machine and the other end of the line is attached to a plastic frame made to hold coupons inside the flask where the three coupons are attached to each plastic frame. The bar is controlled by an electric timer that lowers the coupons into the solution for 10 minutes then raises the coupons out of the solution for 50 minutes, but still keeps the coupons inside the sir space of the flask for the entire duration of the test. This allows the coupons to be exposed to the test solution 10 minutes of each hour. The corrosion test runs for 72 hours starting with the coupons being lowered into the solution. No agitation of the solution is made during the corrosion test.

Corrosion tests are conducted at 21-23 degrees Centigrade. The room temperature is to be recorded daily during the operation of the test. The room temperature shall be taken with a calibrated thermometer located next to the corrosion-testing instrument.

# V. CLEANING OF THE CORRODED COUPONS

The coupons are removed from the solution after 72 hours on the end cycle where the coupons are suspended in the air space of the flask. The coupons are pre-washed under running tap water to remove any loosely adherent corrosion products. They are then placed into glass beakers containing the cleaning acid, which is composed of concentrated hydrochloric acid (HCL) containing 50 grams/liter SnCl<sub>2</sub> (stannous chloride) and 20 grams/liter SbCl<sub>3</sub> (antimony trichloride). The two salts are added to the HCL to stop the reaction of the HCL with the steel once the rust or corrosion is removed. (Note: The gas fumes given off by the acid during this cleaning process contain antimony and are extremely hazardous; this portion of the cleaning must be conducted under a well ventilated hood.)

Allow the coupons to soak in the cleaning acid for a total of 15 minutes. Remove the coupons from the acid and rinsed with tap water followed by distilled water. Wipe with a paper towel or cloth to clean any residual deposit from the coupons. They are then returned to the cleaning acid and the procedure is repeated. After cleaning, the coupons are rinsed in chloroform, air dried for 15 minutes and weighed. Each coupon shall be weighed to a constant weight. The constant weight shall be two consecutive weighings of each coupon within a minimum of 0.5 milligrams of each other.

# VI. EVALUATION OF CORROSION

The weight loss of each coupon is determined by subtracting the final weight from the original weight. The corrosion rate for each coupon is expressed as mils penetration per year (MPY) by the following formula:

MPY = (weight loss (milligrams)) (534) / ((area) (time) (metal density))

# OR

MPY = (weight loss (milligrams)) (534) divided by ((area) (time) (metal density\*))

(Density is 7.85 g/cc for steel\*)

The final MPY value for each solution is determined by calculating the average MPY of the three individual coupons. The average MPY from this point forward will be referred to as only MPY of the solution being tested. (Note: Wide variation of MPY of individual coupons inside the same flask typically indicates contamination of a coupon. If variation of individual MPY is too great to determine consistent data the test should be run over again. Typically coupon variation may run plus or minus 3 MPY.)

# **VII. EXPLANATION**

To put the information into perspective it is necessary to briefly recap the corrosion test process. The corrosion value of the distilled water and the reagent grade sodium chloride is critical to this whole process. These are the two base lines used to determine products acceptability in terms of corrosion value only.

In the following table the distilled water proved to have a corrosion value of 5.00 MPY and the salt had a value of 55.00 MPY. The chart shows that the reagent grade sodium chloride has a water corrected corrosion value of 50.00 MPY. This means that the original corrosion value of the reagent grade sodium chloride and the distilled water (in a 3% solution) was 55.00 MPY. That is, 5.00 MPY for the distilled water and 50.00 MPY for the reagent grade sodium chloride. The 5.00 MPY value for the distilled water is subtracted from the original 55.00 MPY for the reagent grade sodium chloride and distilled water solution to arrive at the distilled water corrected value of 50.00 MPY for the reagent grade sodium chloride.

The corrosion value of 5.00 MPY for the distilled water is subtracted from the total MPY for each of the 3% solutions for each product tested. When this calculation is completed for each product being tested the resulting value indicates the corrected corrosion value.

According to criteria adopted by PNS; "Only corrosion inhibited chemical products that are at least 70% less corrosive than reagent grade sodium chloride may be used". To determine if a product is acceptable, take the corrected corrosion value of the reagent grade sodium chloride and multiply it by 30%. In this case, 50.00 MPY multiplied by 30% equals 15.00 MPY which is the highest acceptable corrected corrosion value for any product in this test. Any product in this test that produces a MPY value higher than 15.00 MPY is not acceptable.

# VIII. NEGATIVE NUMBERS

Some products actually end up with a negative number as their corrected MPY value. A negative number is exceptionally good and it actually indicates that the product when mixed with distilled water in a 3% solution is less corrosive than distilled water.

To show an example of a negative number, refer to one Table 1. The 3% solution of Wondermelt-A had a corrected corrosion value of -5.18 MPY resulting in a Percent Effectiveness of -10.36. The more negative the number the better a product is in terms of corrosion inhibiting abilities.

# **IX. REPORTING RESULTS**

Results shall be reported in Percent Effectiveness. Percent values equal to or less than 30% are passing. The distilled water corrected values of the chemical product and the salt are used to make this calculation. The corrected value of the chemical product is divided by the corrected value of the salt; this value is then multiplied by 100 to give percent.

Example:	Magic Melter II has a corrected value of 10.15 Salt has a corrected value of 50.00	
Therefore:	(10.15 / 50.00) X 100 = 20.3%	Pass
Therefore:	Acme Melter has a corrected value o (19.99 / 50.00) X 100 = 40.0%	f 19.99 Fail

#### TABLE 1

#### CHEMICAL PRODUCTS CORROSION TEST RESULTS

PRODUCT	INITIAL CORROSION VALUE (MPY)	WATER CORRECTED CORROSION VALUE (MPY)	PERCENT EFFECTIVENESS (%)
*Super Stuff	4.97	-0.03	-0.06
*Ice Melter	5.04	0.04	0.07
*Magic Melter	6.00	1.00	2.00
*Magic Melter II	15.15	10.15	20.30
Acme Melter	24.99	19.99	39.98
Acme Melter-1	28.71	23.71	47.42
Wondermelt	59.07	54.07	108.14
*Wondermelt -A	-0.18	-5.18	-10.36
Stuff	22.00	17.00	34.00
SALT	55.00	50.00	100.00
Distilled Water	5.00	0.00	

## \* ACCEPTABLE PRODUCT

**NOTE:** The results used in the above table are for example only, and they are not firm numbers. The MPY corrosion values of the distilled water and the reagent grade sodium chloride may vary from test to test.

# **TEST METHOD C**

Percent Total Settleable Solids and Percent Solids Passing on a No. 10 Sieve

This test method is used to determine the amount of total settleable solids and the percent solids passing on the No. 10 sieve that are generated from a liquid chemical product when stored at a specified cold temperature without agitation.

Settleable Solids for this procedure are typically formed from chemical precipitation, chemical crystallization, or by the dense settlement of any other components of the deicing product.

Chemical precipitates are formed when specific chemical constituents within the liquid chemical product react together chemically.

Chemical crystallization begins to form when a solution is cooled below its chemical saturation point. Crystallization is the physical characteristic by which a liquid begins to turn to a solid. This physical characteristic is typically used to identify the freezing point of a liquid. This test will determine if the deicing solution can maintain its liquid state at the supplied concentration and at the specified testing temperature with no agitation.

The settlement or separation of additional component(s) (i.e. inhibitors) of the product will be examined for the formation of a dense solid layer and the ability of the chemical product to maintain a non-stratified suspension without agitation.

# Total settleable solids will consist of all described parameters excluding soft settling stratification as outlined in the test methodology.

Percent Solids Passing on the No. 10 Sieve will be measured by subtracting the volume of solids retained on the sieve from the total sample volume.

## I. Apparatus

1-Liter Plastic Graduated Imhoff Cone with bottom plug ASTM E 11 No. 10 sieve Rubber policeman Graduated cylinder Watch glass Freezer

## **II. Test Method**

Place 1000 ml of a well-mixed (non-diluted) liquid chemical product into a graduated one-Liter Imhoff cone. Place this sample into a freezer, which has been precalibrated and stabilized to the correct specified temperature as established in each liquid chemical product category. Cover the sample with a watch glass. The sample shall remain in the freezer unagitated for a period of 168 hours. Record the

temperature of the freezer daily to assure proper testing temperature. After 168 hours the sample is carefully removed from the freezer for testing.

# 1. Total Settleable Solids

This test method will be used to determine if the liquid chemical product is usable and if it requires agitation. It will determine the detrimental amount of settlement formed from chemical precipitation, chemical crystallization, or by the dense settlement of any other component(s) of the deicing product.

The formation of chemical precipitation and/or chemical crystallization above the prescribed limit is cause for rejection. These characteristics are observed by a dense formation of precipitate and/or crystals in the cone. Various levels of crystallization may be present if the chemical product concentration is at or near its freezing point.

The settlement of other chemical product components that can produce a dense solid layer above the prescribed limit will be cause for rejection. Stratification of material exhibited by phase separation or exhibiting a soft settlement is not to be interpreted as a dense solid layer. This type of separation is a result of the chemical product not staying homogenous through the test conditions. Samples submitted that exhibit stratification but pass all other specifications will be passed and will be categorized as "Requires Agitation".

The time used to evaluate each sample should be kept to a minimum because as the deicing solutions warm the physical characteristics within the solution change

Remove the sample contained in the Imhoff cone from the freezer. Determine readings as soon as possible because sample temperature begins to rise immediately after being removed. Measure and record the volume of settleable solids using the calibrated gradations on the cone. (Note: If the settled matter contains pockets of liquid between large settled particles, estimate the volume of these and subtract them from the volume of settled solids.)

# For transparent liquids the determinations are easily determined by directly reading the volume of the settleable solids in the bottom of the cone.

For liquids that are not transparent due to the addition of organic matter type inhibitors, the following method shall be used.

Determine and record the interface layer volumes of the inhibitor and the concentrated amount of material in the bottom of the cone.

Determine if the settlement in the bottom portion of the cone is a dense formation or soft settling due to a phase separation. This is done by using an eight-millimeter diameter solid glass rod of sufficient length to reach the bottom of the cone. The rod diameter should allow the rod to be inserted to the bottom of the cone and large enough to be able to determine the slightest resistance. Gently insert the rod into the cone containing the product and gradually lower the rod to the bottom of the cone. If resistance is such that the rod does not reach the bottom of the cone, mark the rod level at the top of the cone and remove it. Place the rod on the outside of the cone with the mark even with the top of the cone. Read and record the volume gradation from the cone that corresponds to the tip of the rod. This will represent the volume inside the cone where resistance was encountered in the product. This volume reading is to be interpreted as a dense settlement and must not exceed the specification limit. If the rod goes completely to the bottom of the cone with no resistance record that no dense settlement was found.

If stratification is present, gently hand stir the chemical product in a clockwise direction for 45 revolutions in one minute to see if the sample will re-homogenize. Examine the chemical product again, with the light if necessary, to determine phase stratification interface levels remaining, if any. Record new levels if present. If no levels are detectable and the solution is returned to a homogenous state exhibiting no stratified layers the chemical product will be marked "Requires Agitation". If levels of stratification are still present, mark as "Requires Extreme Agitation."

The total settleable solids volume shall consist of the accumulated amounts of chemical precipitation, chemical crystallization, and the dense portion of any other constituents. The total settleable solids are reported in percent based upon the volume to volume (V/V) ratio of the settleable solids to the initial sample size.

2. Percent Solids Passing the 10 Sieve

This procedure must be conducted as fast as possible after determining the total settleable solids so that any frozen chemical crystalline materials are adequately evaluated.

Immediately after determining the total settleable solids remove the tip on cone and pour the sample through an ASTM E 11 certified Number 10 sieve. The sieve should be kept in a mixture of ice and water to keep it cold before using and between samples. Rinse the sieve with water to remove any traces of the previous sample prior to placing in the ice bath. Before using the sieve briefly shake excess water from the sieve. The sample should be poured through one-quarter section of the sieve if possible to reduce the surface area from which the sample must be retrieved. The sample on the sieve is not rinsed or pushed through the sieve by any means. All material not flowing through the sieve is rubber policed from the sieve into a graduated cylinder and the volume measured and recorded. Rubber police only the sieve and does not come loose on the face of the sieve is considered passing and is not included. This volume is subtracted from the total volume of the sample to calculate the sample volume passing. The solids passing the No. 10 sieve are reported in percent based upon the volume to volume (V/V) ratio of sample volume passing to the initial sample size.

# SNOW AND ICE CONTROL CHEMICAL PRODUCTS SPECIFICATIONS

# AND TEST PROTOCOLS – SUMMARY OF CHANGES

Date of Change	Change Description	Notes
March 9, 2019	Replaced Chemical Product	Category 4 section was updated July
	Category 4 section with updated	2016 and posted on the PNS website as a
	content.	standalone document. The updated
		Category 4 section was incorporated into
		the main document, replacing the outdated
		Category 4 section, in March 2019.