

Clear Roads Snow and Ice Control Chemical Product Program

Guidance Document for Material Qualified Products List, Specifications, Test Methods, and Product Purchasing

2020

Table of Contents

Int	roduction and Purpose	1
	Introduction	
	Purpose	
	Abbreviations	1
	Definitions	1
	References	
1.	QPL Process	2
	1.1 Overview	2
	1.2 Paperwork/Documentation	
	1.3 Product Sample Submittals	
	1.4 Field Verification	
	1.5 QPL Management	
2.	General Specifications	
	2.1 Specified Limits	
	2.2 Informational Requirements	
	2.3 Chemical Product and Inhibitor Product Categories	
3.	Test Methods	
	3.1 Table of Test Methods	
	3.2 Testing Protocol	
	Test Method 1: Percent Concentration of Active Ingredient in the Liquid	
	Test Method 2: Determination of the Specific Gravity (Weight Per Gallon)	
	Test Method 3: Corrosion Control Inhibitor Presence and Concentration	
	Test Method 4: Determination of the pH	
	Test Method 5: Corrosion Rate	
	Test Method 6: Percent Total Settleable Solids and Percent Solids Passing a 10 Sieve	
	Test Method 7: Total Phosphorus	40
	Test Method 8: Total Cyanide	40
	Test Method 9: Total Arsenic, Barium, Cadmium, Chromium, Copper, Lead, Selenium and Zinc	40
	Test Method 10: Total Mercury	40
	Test Method 11: Milliequivalents OR "meq"	40
	Test Method 12: Moisture Content of Solid Chemical Products	40
	Test Method 13: Gradation	
	Test Method 14: Visual Inspection and Field Observations	
	Test Method 15: Toxicity Test	
	Test Method 16: Ammonia - Nitrogen	
	Test Method 17: Total Kjeldahl Nitrogen	
	Test Method 18: Nitrate and Nitrite as Nitrogen	
	Test Method 19: Biological Oxygen Demand	
	Test Method 20: Chemical Oxygen Demand	
	Test Method 21: Frictional Analysis	
	Test Method 22: Insoluble Material	
	Test Method 23: Chloride	
	Test Method 24: Eutectic Temperature	
4.	Purchasing	
	4.1 Bidding	
	4.2 Deliveries and Invoicing of Products	
	4.3 Field Inspection, Unloading, Sampling and Testing	
	4.4 Product Rejection and Price Adjustments	
	4.5 Bid Evaluation Process	

4.6	Bid and Sample Delivery	48
4.7	Bid Schedule	48

Tables

Table 1. Specification and Method of Determination	7
Table 2. Information and Method of Determination	8
Table 3. Test Methods	26
Table 4. Example of Chemical Product Corrosion Test Results	36

Figures

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Introduction and Purpose

Introduction

Clear Roads provides guidance and administration of the Qualified Products List (QPL), which was originally established through the Pacific Northwest Snowfighters. The QPL was created to review, test, and approve products used for winter maintenance and to provide guidance in the selection and purchasing of environmentally responsible products.

Clear Roads is a national research consortium consisting of several state departments of transportation. Its focus is to fund research and technology transfer efforts in relation to winter maintenance materials, equipment, and methods.

Purpose

The purpose of this document is to define the submittal and maintenance process for the QPL, provide product specifications and testing protocol, and to provide guidance on purchasing winter maintenance products.

Abbreviations

- **QA** Quality Assurance
- **QPL** Qualified Products List
- SDS Safety Data Sheet

Definitions

- **Independent Laboratory** Laboratory not affiliated with manufacturer, vendor, or purchaser that is tasked with performing analyses listed in this document.
- Lot Number A specific number assigned to that particular product as delivered.
- Manufacturer Entity responsible for the production of the material.
- Purchaser Entity responsible for material purchasing.
- Qualification Process Process followed for product inclusion on the QPL.
- **Qualified Products List (QPL)** List of products that have been tested and evaluated and determined to meet requirements stated in this document.
- Vendor Entity responsible for marketing and/or delivery of the material to purchasers.

References

Qualified Products List (QPL) ASTM International

1. QPL Process

1.1 Overview

The QPL is composed of products that have been tested and found to be in conformance with the specifications found in Table 1, and all other necessary product or testing documentation (e.g., Table 2 for liquid products or products intended to be applied as liquid). Clear Roads reserves the right to deny qualification based on all product information provided. Though the information required for "informational purposes" doesn't have set parameters, the tests may reveal information about a product that requires further attention or inquiry.

All products submitted for inclusion on the QPL shall be tested by an independent laboratory and the results shall conform to the specified limits contained within Table 1 and Table 2, as appropriate, and test results sent to Clear Roads. Once a product passes the required specification testing limits and has passed the Clear Roads review, Clear Roads will request any additional information including vendor product samples. Once product samples are received, Clear Roads will conduct appropriate Quality Assurance testing. If QA testing is satisfactory and field testing is deemed unwarranted, the product shall be listed on the Clear Roads QPL. Figure 1 on page 3 details the process in a flowchart.

Incomplete submittals will not be reviewed. Clear Roads will review submitted paperwork in a timely manner and report discrepancies. It is not the role of Clear Roads to do a complete review and find all things erroneous about a product. For example, if a solid doesn't meet the gradation specification, it will be denied and no assurance that any other parameter is met or not met will necessarily be provided. It is prudent that vendors are familiar with the specifications and check their product against the specifications prior to submittal. A product that fails to meet the standard limits as specified will be failed.

Clear Roads 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 or risk to the environment. The right to qualify or disqualify, accept or reject a product based on manufactured composition rest solely with Clear Roads. The Clear Roads assessment shall be final and in the best interest of Clear Roads.

Any material changes to a product that is listed on the QPL by either the manufacturer or the vendor, 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 placed back on the QPL.

Each vendor 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.

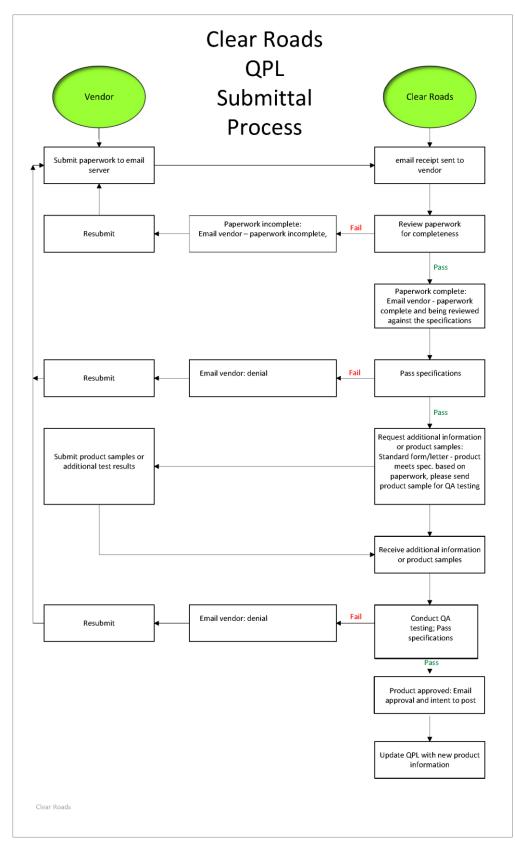


Figure 1. QPL Submittal Process

1.2 Paperwork/Documentation

- A. Manufacturers/vendors will use the "Product Sample Checklist" on the Clear Roads Website to request a product to be listed on the QPL.
- B. In addition to the checklist, the following information is required:
 - (1) Product data/information sheet.
 - (2) Laboratory test results.
 - (3) Safety data sheet (for product and inhibitor, as appropriate).
 - (4) Proprietary information (if applicable); proprietary information will be held confidential by the Clear Roads QPL. Submit in a separate .pdf document titled "confidential," or for non-electronic submittals, a sealed envelope to maintain confidentiality.
- C. All paperwork must clearly identify the product and will be matched up to the product sample submittal when that is requested.
- D. All documents must be clear and legible. Illegible documents will be rejected.
- E. Clear Roads will review submitted documentation/paperwork for completeness.
 - (1) Vendor/manufacturer will be notified by Clear Roads that a complete/incomplete package was received.
 - (2) If incomplete, vendor/manufacturer will be required to resubmit a complete package for review.
- F. Complete packages will be reviewed against the product category specifications.
 - (1) If paperwork indicates the product meets specifications, then Clear Roads will request a product sample from the vendor/manufacturer for Clear Roads QA testing.
 - (2) If paperwork indicates the product doesn't meet specifications, it will be rejected. The vendor/manufacturer will receive notification from Clear Roads that the product failed to meet specifications and resubmittal is required for placement on the QPL.

1.3 Product Sample Submittals

When a product sample is requested by Clear Roads, two one-gallon samples of the bid product shall accompany the required information for qualification testing. Clear Roads has the right to test for verification or to accept the product as qualified based on the documentation submitted.

- A. A copy of the Product Sample Checklist and the product Safety Data Sheet (SDS) must be included with all product samples to be considered for the QPL.
- B. Product samples must be clearly labeled as to product content and in order to match product samples to the paperwork.
- C. Clear Roads will conduct QA testing on sample submittals to verify submitted documentation by vendor/manufacturer. Results of the QA testing shall be verifiable and final.
- D. All Clear Roads QA test results will be kept on file and will be used to establish product information as appropriate. If Clear Roads QA testing is different than test results provided by the vendor/manufacturer, the Clear Roads test results will be the official record of product specifications.

1.4 Field Verification

- A. Most products will not require field verification testing by Clear Roads. Field performance should be relatively predictable based on the percentage of the active chemical constituent. If vendor/manufacturer is claiming a unique application method or rate, Clear Roads may require field testing to verify product safety and effectiveness.
- B. 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 field-testing lies solely with the Clear Roads.
- C. 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 QPL.

1.5 QPL Management

Purchased products that appear on the QPL may be tested for compliance to the material that was originally submitted for qualification. The purchaser has the right to conduct this testing at its own will. The most current QPL can be viewed at the Clear Roads website or by contacting one of the Clear Roads QPL members.

A. Expiration

Listing on the Clear Roads QPL will expire 5 (five) years from the QPL approval on December 31st of the expiration year. If the producer wants their product to remain on the Clear Road QPL beyond the expiration date they can submit a letter to the QPL subcommittee's chemists stating the product still meets specifications and is still being produced, and that the formulation hasn't changed. If there are no concerns from the QPL committee the product will be remain on the QPL until the next expiration date at which time this process can be repeated. The committee may request product samples for testing.

B. Random Testing and Auditing

The Clear Roads QPL reserves the right to sample materials on the QPL for testing and to perform audits of test reports. Clear Roads and its representatives may sample material from the manufacturing plant, the member state sites, and other locations. Clear Roads reserves the right to test samples to verify compliance with these specifications. Producers must maintain a complete record of all test reports for the previous two years and current calendar year.

C. Product Name Changes, Producer Changes

If a product name changes but is produced by the same producer in the same manner when first qualified, then the producer will submit a letter to the QPL subcommittee verifying the new products name and that it still meets the Clear Roads QPL Specifications. If the producer changes its name but the product is still produced in the same manner when first qualified, then the producer will submit a letter to the QPL subcommittee verifying the new producer name and that it still meets the Clear Roads QPL Specifications. If a <u>different</u> producer name and that it still meets the Clear Roads QPL Specifications. If a <u>different</u> producer is producing a qualified product, then that producer shall submit the product for QPL review.

D. Product Formulation Modification

If a product currently listed on the QPL is modified, the product must be resubmitted for approval on the QPL, with the accompanying Product Sample Checklist. Exceptions to this rule may be considered upon request.

E. Disqualification

Products that are found to be non-compliant with Clear Roads specifications may be removed from the QPL at any time. Additionally, if member states' testing results are greater than a 10% failure rate in a year and confirmed by third-party testing, the member states can submit those results to the QPL subcommittee for disqualification. The QPL subcommittee will provide in writing reasons for disqualification. These items will remain unlisted until adherence to the specifications can be verified.

2. General Specifications

This section provides the product specifications. Products that contain any constituent in excess of the following established total concentration limits (as tested in accordance with the listed test methodology from Section 3) will not be acceptable.

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 the parameters as compared to the specification limits. Do not back calculate the concentration of the parameters to the dry weight of the material.

All liquid deicers must successfully complete the National Association of Corrosion Engineers (NACE) Standard TM0169-95, as modified by Clear Roads, and found to have a Corrosion Value of at least 70% less than that of Sodium Chloride (salt).

Section 2.1 contains Table 1, which lists the general specification limits, which are applicable to all products on the QPL. Section 2.2 contains Table 2, which lists the informational requirements for the liquid products and solids products to be used as liquids for application purposes. Section 2.3 provides category-specific specifications.

Table 1. Specification and Method of Determination

Test	Specified Limits	Method Name
Corrosion Rate (corrosion inhibited products only)	≥ 70% less corrosive than NaCl	Test Method 5
Phosphorus	≤ 2500. ppm	Test Method 7
Cyanide ¹	≤ 0.20 ppm	Test Method 8
Arsenic	≤ 5.0 ppm	Test Method 9
Barium	≤ 100.0 ppm	Test Method 9
Cadmium	≤ 0.20 ppm	Test Method 9
Chromium	≤ 1.0 ppm	Test Method 9
Copper	≤ 1.0 ppm	Test Method 9
Lead	≤ 1.0 ppm	Test Method 9
Selenium	≤ 5.0 ppm	Test Method 9

2.1 Specified Limits

Vendor may bid this product with or without the anti-caking agent. Vendors must note on the Sample Checklist if the sample does contain anti-caking agent or not, and if so, what type. If the Vendor chooses not to add the anti-caking agent it does not prevent the vendor from assuring that the delivered product is in a free-flowing state.

¹ 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.

Test	Specified Limits	Method Name
Zinc	≤ 10.00 ppm	Test Method 9
Mercury	≤ 0.05 ppm	Test Method 10

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.

In addition to the above testing requirements, all liquid products must be tested for pH. 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 its constituents may be waived by the Clear Roads 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 Clear Roads. The Clear Roads decision to waive the pH requirement shall be in the best interest of the Clear Roads and shall be final.

2.2 Informational Requirements

For all liquid products and solid products intended to be used as liquid for application purposes.

Test	Specified Limits	Method Name
Toxicity	Sample measurement as determined by an Independent Laboratory	Test Method 15
Ammonia – Nitrogen	Sample measurement as determined by an Independent Laboratory	Test Method 16
Total Kjeldahl Nitrogen	Sample measurement as determined by an Independent Laboratory	Test Method 17
NO ₃ - & NO ₂ - as Nitrogen	Sample measurement as determined by an Independent Laboratory	Test Method 18
Bio. Oxygen Demand	Sample measurement as determined by an Independent Laboratory	Test Method 19
Chem. Oxygen Demand	Sample measurement as determined by an Independent Laboratory	Test Method 20
Frictional Analysis	Sample measurement as determined by an Independent Laboratory	Test Method 21
Specific Gravity ²	N/A	Test Method 2

Table 2. Information and Method of Determination

² Specific gravity chart 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.

2.3 Chemical Product and Inhibitor Product Categories

- A. Chemical Product Category 1 Corrosion Inhibited Liquid Magnesium Chloride
 - (1) The product must meet the following requirements:
 - a. General Specifications in Tables 1 and 2.
 - b. No less than 25% Magnesium Chloride. (Test Method 1)
 - c. Weight per gallon as indicated by vendor. (Test Method 2)
 - d. Corrosion Inhibitor as indicated by vendor³. (Test Method 3)
 - e. pH must be 6.0 9.0. (Test Method 4)
 - f. Total Settleable Solids/Solids Passing a #10 Sieve. (Test Method 6)
 - Storage temperature: $-17.8^{\circ}C \pm 1^{\circ}C (0^{\circ}F \pm 2^{\circ}F)$
 - Total Settleable Solids (V/V): 1.0% maximum
 - Solids Passing a #10 Sieve: 99.0% minimum

B. Chemical Product Category 2 – Corrosion Inhibited Liquid Calcium Chloride

(1) The product must meet the following requirements:

- a. General Specifications in Tables 1 and 2.
- b. No less than 25% Calcium Chloride. (Test Method 1)
- c. Weight per gallon as indicated by vendor. (Test Method 2)
- d. Corrosion Inhibitor as indicated by vendor³. (Test Method 3)
- e. pH must be 6.0 10.0. (Test Method 4)
- f. Total Settleable Solids/Solids Passing a #10 Sieve. (Test Method 6)
 - Storage temperature: -29°C ± 1°C (-20°F ± 2°F)
 - Total Settleable Solids (V/V): 1.0% maximum
 - Solids Passing a #10 Sieve: 99.0% minimum

C. Chemical Product Category 3 –Liquid Calcium Magnesium Acetate

- (1) The product must meet the following requirements:
 - a. General Specifications in Tables 1 and 2.
 - b. No less than 25% Calcium Magnesium Acetate. (Test Method 1)
 - c. Weight per gallon as indicated by vendor. (Test Method 2)
 - d. pH must be 8.0 10.0. (Test Method 4)
 - e. Total Settleable Solids/Solids Passing a #10 Sieve. (Test Method 6)
 - Storage temperature: -12°C ± 1°C (10°F ± 2°F)

³ The finished deicing product, including corrosion inhibitors, must be completely accomplished at the original manufacturing plant location. Post-addition of corrosion inhibitors or any other ingredients and splash mixing is unacceptable after the product has left the original manufacturing plant.

- Total Settleable Solids (V/V): 4.0% maximum
- Solids Passing a #10 Sieve: 99.0% minimum
- f. Calcium to magnesium mole ratio shall be 3 to 7. (Test Method 1)
- g. Residual base shall be a maximum of 0.30 meq (milliequivalents) base per gram of sample. (Test Method 11)

D. Chemical Product Categories 4A, 4B, 4C, 4D and 4E – Corrosion Inhibited Solid Sodium Chloride Specifications⁴

(1) The Categories shall be defined as follows:

<u>Category 4A</u> – Dry Salt, ASTM D632 Type I, Grade 2 Gradation Corrosion Percent Effectiveness of 30% or less

<u>Category 4B</u> – Wet Salt, ASTM D632 Type I, Grade - Modified Corrosion Percent Effectiveness of 31% to 85%

<u>Category 4C</u> – Wet Salt, ASTM D632 Type I, Grade 2 Gradation Corrosion Percent Effectiveness of 31% to 85%

<u>Category 4D</u> – Dry Salt, ASTM D632 Type I, Grade 1 Gradation Corrosion Percent Effectiveness of 30% or less

<u>Category 4E</u> – Wet Salt, ASTM D632 Type I, Grade 1 Gradation Corrosion Percent Effectiveness of 31% to 85%

(2) The product must meet the following requirements:

- a. General Specifications in Table 1.
- b. Anti-Caking⁵ agent will be included to ensure that the material remains free from hard caking and suitable for its intended purpose. **(Test Method 14)**
- c. 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 14)**

(3) Individual Category requirements:

- a. Category 4A
 - i. Moisture Content. (Test Method 12)

0.5% Maximum

ii. Insoluble Material. (Test Method 22)

10.0% Maximum

iii. Corrosion Control Inhibitor and Concentration. (Test Method 3)

⁴ Category 4 is tested/qualified for use as a solid. If user intends to use products in this category to make brine, tests included in Table 2 (for liquid products) haven't been conducted, evaluated, or reviewed by Clear Roads.

⁵ See footnote for Cyanide in Table 1.

iv. Gradation. (Test Method 13)

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

- b. Category 4B
 - i. Moisture Content. (Test Method 12)

5.0% Maximum

ii. Insoluble Material. (Test Method 22)

10.0% Maximum

- iii. Corrosion Control Inhibitor and Concentration. (Test Method 3)
- iv. Gradation. (Test Method 13)

Sieve Size	Wt.% Passing	
3/4"	100	
1/4"	75 – 85	
#8	50 – 70	
#30	10 - 20	

c. Category 4C

i. Moisture Content. (Test Method 12)

5.0% Maximum

ii. Insoluble Material. (Test Method 22)

10.0% Maximum

- iii. Corrosion Control Inhibitor and Concentration. (Test Method 3)
- iv. Gradation. (Test Method 13)

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

- d. Category 4D
 - i. Moisture Content. (Test Method 12)

0.5% Maximum

ii. Insoluble Material. (Test Method 22)

10.0% Maximum

iii. Corrosion Control Inhibitor and Concentration. (Test Method 3)

iv. Gradation. (Test Method 13)

Sieve Size	Wt.% Passing
1/2"	100
3/8"	95 – 100
#4	20 – 90
#8	10 - 60
#30	0 – 15

- e. Category 4E
 - i. Moisture Content. (Test Method 12)

5.0% Maximum

ii. Insoluble Material. (Test Method 22)

10.0% Maximum

- i. Corrosion Control Inhibitor and Concentration. (Test Method 3)
- ii. Gradation. (Test Method 13)

Sieve Size	Wt.% Passing
1/2"	100
3/8″	95 – 100
#4	20 - 90
#8	10-60
#30	0 – 15

- E. Chemical Product Category 5 Corrosion Inhibited Sodium Chloride Plus 10% Magnesium Chloride (Solid)
 - (1) The product must meet the following requirements:
 - a. General Specifications in Table 1.
 - b. The vendor 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.
 - c. Gradation. (Test Method 13)

Shall be ASTM D632 Type 1, Grade 2, for Sodium Chloride.

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

- d. Anti-Caking agent⁶ will be included to ensure that the material remains free from hard caking and suitable for its intended purpose. **(Test Method 14)**
- e. 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 14)**
- f. Moisture Content. (Test Method 12)
 - Sodium Chloride Only: 0.5% maximum moisture content.
 - Magnesium Chloride Hexahydrate Only: 56% maximum moisture content (both bound and unbound water⁷).
- g. Corrosion Control Inhibitor and Concentration. (Test Method 3)
- h. Product Must Contain No Less Than 10% Magnesium Chloride Hexahydrate by Weight. (Test Method 1)

This product will consist of 10% magnesium chloride hexahydrate (MgCl₂ +6H₂O) as specified by weight. Weight of the magnesium chloride shall be calculated as a percent of the total mixture with zero percent-unbound water ⁷. The manufacturer 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 (MgCl₂) in the total mixture shall be based on the weight of magnesium chloride hexahydrate (MgCl₂+6H₂O).

F. Chemical Product Category 6 – Corrosion Inhibited Sodium Chloride Plus 20% Magnesium Chloride (Solid)

(1) The product must meet the following requirements:

- a. General Specifications in Table 1.
- b. The vendor 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.
- c. Gradation. (Test Method 13)

Shall be ASTM D632 Type 1, Grade 2, for Sodium Chloride.

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

⁶ See footnote for Cyanide in Table 1.

⁷ 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.

- d. Anti-Caking agent⁸ will be included to ensure that the material remains free from hard caking and suitable for its intended purpose. **(Test Method 14)**
- e. 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 14)**
- f. Moisture Content. (Test Method 12)
 - Sodium Chloride Only: 0.5% maximum moisture content.
 - Magnesium Chloride Hexahydrate Only: 56% maximum moisture content (both bound and unbound water⁷).
- g. Corrosion Control Inhibitor and Concentration. (Test Method 3)
- h. Product Must Contain No Less Than 20% Magnesium Chloride Hexahydrate by Weight. (Test Method 1)

This product will consist of 20% magnesium chloride hexahydrate (MgCl₂ +6H₂O) as specified by weight. Weight of the magnesium chloride shall be calculated as a percent of the total mixture with zero percent-unbound water ⁷. 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 (MgCl₂) in the total mixture shall be based on the weight of magnesium chloride hexahydrate (MgCl₂ +6H₂O).

G. Chemical Product Category 7 – Solid Calcium Magnesium Acetate (CMA)

(1) The product must meet the following requirements:

- a. General Specifications in Tables 1 and 2.
- b. Product will consist of Calcium Magnesium Acetate. (Test Method 14)

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 ingredient(s) result in an improvement to the product.

- c. Calcium to magnesium mole ratio shall be 3 to 7. (Test Method 1)
- d. Total Settleable Solids/Solids Passing a #10 Sieve. (Test Method 6)

When liquefied at or near a 25% concentration:

- Storage temperature: -12°C ± 1°C (10°F ± 2°F)
- Total Settleable Solids (V/V): 4.0% maximum
- Solids Passing a #10 Sieve: 99.0% minimum
- e. Moisture (free and hydration) shall not exceed 10%. (Test Method 12)
- f. Product attrition shall be less than 2.5% with minimum dust generated on handling. **(Test Method 14 and any other tests deemed necessary)**

⁸ See footnote for Cyanide in Table 1.

CLEAR ROADS SNOW AND ICE CONTROL CHEMICAL PRODUCT PROGRAM

- g. Residual base shall be 0.30 milliequivalents base per gram of sample. (Test Method 11)
- h. The pH of product in a 10% solution shall be 8 to 10. (Test Method 4)

H. Chemical Product Categories 8A, 8B, 8C, 8D – Non-Corrosion Inhibited Solid Sodium Chloride

(1) The Categories shall be defined as follows:

Category 8A – Dry Salt, ASTM D632 Type I, Grade 2 Gradation

Category 8A-B Brining Salt

Category 8A-R Road Salt

Category 8B – Wet Salt, ASTM D632 Type I, Grade 2 Gradation

<u>Category 8C</u> – Dry Salt, Fine Gradation

Category 8C-B Brining Salt

Category 8C-R Road Salt

Category 8D – Dry Salt, ASTM D632 Type I, Grade 1 Gradation

(2) The Categories must meet the following requirements:

- a. General Specifications in Table 1.
- b. Anti-Caking agent⁹ will be included to ensure that the material remains free from hard caking and suitable for its intended purpose. **(Test Method 14)**
- c. 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 14)**

(3) Individual Category requirements:

- a. Category 8A
 - i. Moisture Content. (Test Method 12)

0.5% Maximum

ii. Insoluble Material. (Test Method 22)

Category 8A-B – 1.0% Maximum

Category 8A-R – 10.0% Maximum

iii. Gradation. (Test Method 13)

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

⁹ See footnote for Cyanide in Table 1.

CLEAR ROADS SNOW AND ICE CONTROL CHEMICAL PRODUCT PROGRAM

b. <u>Category 8B</u>

i. Moisture Content. (Test Method 12)

5.0% Maximum

ii. Insoluble Material. (Test Method 22)

10.0% Maximum

iii. Gradation. (Test Method 13)

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

- c. Category 8C
 - i. Moisture Content. (Test Method 12)

0.5% Maximum

ii. Insoluble Material. (Test Method 22)

Category 8C-B – 1.0% Maximum

Category 8C-R – 10.0 % Maximum

iii. Gradation. (Test Method 13)

Sieve Size	Wt.% Passing
#4	20 - 100
#100	0 - 3

- d. Category 8D
 - i. Combined Moisture Content (Test Method 12) and Insoluble Material. (Test Method 22)

5.0% Maximum

ii. Gradation. (Test Method 13)

Sieve Size	Wt.% Passing
1/2"	100
3/8"	95 – 100
#4	20 – 90
#8	10 - 60
#30	0 - 15

- I. Chemical Product Category 9 Corrosion Inhibited Liquid Sodium Chloride
 - (1) The product must meet the following requirements:
 - a. General Specifications in Tables 1 and 2.
 - b. Product must contain no less than 21% sodium chloride. **(Test Method 1 or Test Method 23)**
 - c. Weight per gallon as indicated by vendor. (Test Method 2)
 - d. Corrosion Inhibitor as indicated by vendor. (Test Method 3)
 - e. pH must be 6.0 9.0. (Test Method 4)
 - f. Total Settleable Solids/Solids Passing a #10 Sieve. (Test Method 6)
 - Storage temperature: $-17.8^{\circ}C \pm 1^{\circ}C (0^{\circ}F \pm 2^{\circ}F)$
 - Total Settleable Solids (V/V): 1.0% maximum
 - Solids Passing a #10 Sieve: 99.0% minimum
- J. Chemical Product Category 10 Corrosion Inhibited Liquid Sodium Chloride Plus Calcium Chloride
 - (1) The product must meet the following requirements:
 - a. General Specifications in Tables 1 and 2.
 - b. Product must contain no less than 15% sodium chloride plus no less than 2% calcium chloride. **(Test Method 1 or Test Method 23)**
 - c. Weight per gallon as indicated by vendor. (Test Method 2)
 - d. Corrosion Inhibitor as indicated by vendor. (Test Method 3)
 - e. pH must be 6.0 9.0. (Test Method 4)
 - f. Total Settleable Solids/Solids Passing a #10 Sieve. (Test Method 6)
 - Storage temperature: $-17.8^{\circ}C \pm 1^{\circ}C (0^{\circ}F \pm 2^{\circ}F)$
 - Total Settleable Solids (V/V): 1.0% maximum
 - Solids Passing a #10 Sieve: 99.0% minimum
- K. Chemical Product Category 11 Corrosion Inhibited Liquid Chloride Blended Brines

- a. General Specifications in Tables 1 and 2.
- 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 1 or Test Method 23)
- c. Weight per gallon as indicated by vendor. (Test Method 2)
- d. Corrosion Inhibitor as indicated by vendor. (Test Method 3)
- e. pH must be 6.0 9.0. (Test Method 4)

- f. Total Settleable Solids/Solids Passing a #10 Sieve. (Test Method 6)
 - Storage temperature: $-17.8^{\circ}C \pm 1^{\circ}C (0^{\circ}F \pm 2^{\circ}F)$
 - Total Settleable Solids (V/V): 1.0% maximum
 - Solids Passing a #10 Sieve: 99.0% minimum

L. Chemical Product Category 12 – Blended Acetates (¹⁰New Category)

(1) The product must meet the following requirements:

- a. General Specifications in Tables 1 and 2.
- b. No less than 25% Active Acetate Ingredient. (Test Method 1)
- c. Weight per gallon as indicated by vendor. (Test Method 2)
- d. pH must be 8.0 10.0. (Test Method 4)
- e. Total Settleable Solids/Solids Passing a #10 Sieve. (Test Method 6)
 - Storage temperature: $-12^{\circ}C \pm 1^{\circ}C (10^{\circ}F \pm 2^{\circ}F)$
 - Total Settleable Solids (V/V): 4.0% maximum
 - Solids Passing a #10 Sieve: 99.0% minimum
- f. Residual base shall be a maximum of 0.30 meq (milliequivalents) base per gram of sample. **(Test Method 11)**

M. Chemical Product Category 13 – Liquid Products with Greater than 30% Organics (¹⁰New Category)

(1) The product must meet the following requirements:

- a. General Specifications in Tables 1 and 2.
- b. Product must contain no less than 30% organic content.
- c. Weight per gallon as indicated by vendor. (Test Method 2)
- d. Corrosion Inhibitor as indicated by vendor (if applicable). (Test Method 3)

The finished deicing product, including corrosion inhibitors, must be completely accomplished at the original manufacturing plant location. Post-addition of corrosion inhibitors or any other ingredients and splash mixing is unacceptable after the product has left the original manufacturing plant.

- e. pH must be 6.0 9.0. (Test Method 4)
- f. Total Settleable Solids/Solids Passing a #10 Sieve. (Test Method 6)
 - Storage temperature: $-17.8^{\circ}C \pm 1^{\circ}C (0^{\circ}F \pm 2^{\circ}F)$
 - Total Settleable Solids (V/V): 1.0% maximum
 - Solids Passing a #10 Sieve: 99.0% minimum

¹⁰ Clear Roads added categories for like-products that were in the Experimental Category, therefore specifications in these categories are subject to change.

CLEAR ROADS SNOW AND ICE CONTROL CHEMICAL PRODUCT PROGRAM

N. Chemical Product Category 14 – Liquid Salt Blends (¹¹New Category)

(1) The product must meet the following requirements:

- a. General Specifications in Tables 1 and 2.
- b. Product must contain no less than 10% sodium chloride plus no less than 3% of calcium chloride, magnesium chloride, or a combination of both. Total chloride concentration shall be no less than 20%. **(Test Method 1 or Test Method 23)**
- c. Weight per gallon as indicated by vendor. (Test Method 2)
- d. Corrosion Inhibitor as indicated by vendor (if applicable). (Test Method 3)
- e. pH must be 6.0 9.0. (Test Method 4)
- f. Total Settleable Solids/Solids Passing a #10 Sieve. (Test Method 6)
 - Storage temperature: $-17.8^{\circ}C \pm 1^{\circ}C (0^{\circ}F \pm 2^{\circ}F)$
 - Total Settleable Solids (V/V): 1.0% maximum
 - Solids Passing a #10 Sieve: 99.0% minimum

O. Inhibitor Product Category A-1 – Corrosion Inhibitor for Sodium Chloride Brine

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

- a. The finished corrosion inhibited sodium chloride shall have a minimum sodium chloride concentration of no less than 21%. **(Test Method 1 or Test Method 23)**
- b. Corrosion Percent Effectiveness Rating of 30% or less. (Test Method 5)
- c. This liquid corrosion inhibitor when added to concentrated sodium chloride brine will provide a finished product that is compliant to all the General Specifications listed in Tables 1 and 2.
- d. 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.
- e. 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.
- f. 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.

¹¹ Clear Roads added categories for like-products that were in the Experimental Category, therefore specifications in these categories are subject to change.

- g. 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.
- h. 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
- i. Total Settleable Solids/Solids Passing a #10 Sieve. (Test Method 6)
 - Storage temperature: Designated Storage Class ± 2°F
 - Total Settleable Solids (V/V): 1.0% maximum
 - Solids Passing a #10 Sieve: 99.0% minimum

P. 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.

- a. The finished corrosion inhibited product shall have a minimum concentration of 15% sodium chloride and a minimum concentration of 2% calcium chloride. (Test Method 1 or Test Method 23)
- b. 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. (Test Method 5)
- c. This liquid corrosion inhibitor when added to concentrated sodium chloride/calcium chloride brine will provide a finished product that is compliant to all the General Specifications listed in Tables 1 and 2.
- d. 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.
- e. The finished product is classified into the following Types:
 - i. Type I The corrosion inhibitor contains sufficient calcium chloride that additional calcium chloride is not required to be added to the salt brine.
 - ii. Type II The corrosion inhibitor, salt brine, and calcium chloride are added separately.
- f. 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 in a guidelines.

- g. 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.
- h. 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.
- i. 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
- j. Total Settleable Solids/Solids Passing a #10 Sieve. (Test Method 6)
 - Storage temperature: Designated Storage Class ± 2°F
 - Total Settleable Solids (V/V): 1.0% maximum
 - Solids Passing a #10 Sieve: 99.0% minimum

Q. Inhibitor Product Category A-3 – Corrosion Inhibitor for Sodium Chloride Brine

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

- a. The finished corrosion inhibited sodium chloride shall have a minimum sodium chloride concentration of no less than 15%. **(Test Method 1 or Test Method 23)**
- b. The product shall 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. (Test Method 5)
- c. 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.
- d. This liquid corrosion inhibitor when added to concentrated sodium chloride brine will provide a finished product that is compliant to all the General Specifications listed in Table 1.
- e. 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.

- f. 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.
- g. 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.
- h. 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
- i. Total Settleable Solids/Solids Passing a #10 Sieve. (Test Method 6)
 - Storage temperature: Designated Storage Class ± 2°F
 - Total Settleable Solids (V/V): 1.0% maximum
 - Solids Passing a #10 Sieve: 99.0% minimum

R. Inhibitor Product Category A-4 – Additive for Liquid Deicing Products and Pre-Wet Materials (¹²New Category)

This specification is for a liquid additive to brine or for use as a pre-wetting material only. It is not qualified as a product to be used as a direct liquid application.

- a. The vendor shall provide the active ingredient concentration and the recommended dilution rate(s). **(Test Method 1 or Test Method 23)**
- b. Corrosion Percent Effectiveness Rating of 30% or less. (Test Method 5)
- c. The additive, when added to liquid deicing products or solid products as a pre-wet, will provide a finished product that is compliant to all the General Specifications listed in Tables 1 and 2.
- d. The finished product shall provide eutectic temperature points equal to or lower than that of the standard liquid deicing product without the additive.
- e. The additive shall be capable of being homogenously mixed with the liquid deicing product and resulting in a finished product that does not separate or settle out.
- f. The additive shall be flowable and have the capability to be mixed fully into the liquid deicing product at a minimum temperature of 15° F.
- g. Temperature Storage Class of Additive: The additive 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

¹² Clear Roads added categories for like-products that were in the Experimental Category, therefore specifications in these categories are subject to change.

- Class 2: 0° F
- h. Total Settleable Solids/Solids Passing a #10 Sieve for concentrated additive. **(Test Method 6)**
 - Storage temperature: Designated Storage Class ± 2°F
 - Total Settleable Solids (V/V): 1.0% maximum
 - Solids Passing a #10 Sieve: 99.0% minimum

S. Experimental Category for Liquid Products (Category L)

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

- (1) The product must meet the General Specifications list in Table 1 and Table 2.
- (2) 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 Clear Roads. The manufacturer shall submit all test results as required along with the following information:
 - a. Define the active ingredient that can be analytically measured.
 - b. Define the concentration of the active constituent at which the product will be manufactured. State the test protocols used for analyzing the primary constituent.
 - c. 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.
 - d. Once the testing information is completed the manufacturer shall then follow the protocols for submitting samples and testing information to Clear Roads for Quality Assurance Testing. Upon request of Clear Roads, 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.
 - e. Clear Roads reserves the right to provisionally approve unique products that satisfactorily meet stated standards to allow for field testing. Provisionally approved products will be noted as such on the QPL. Provisional approval will remain until field testing and evaluations are complete and determined successful. If successful, the product will then be classified as a Qualified Product in the Experimental Category or an appropriate category if one is available for the product. If not successful, the product will be removed from the QPL.
 - f. Field testing of the products for this category shall be conducted by Clear Roads members or by agencies within the Associations' domain. If other than a Clear Roads member is conducting the testing themanufacture shall be responsible for collecting the field data and submitting it to Clear Roads 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 Clear Roads can not only review questions of performance but also handling, storage, application information and any other information that Clear Roads feels is relevant regarding a productand its use.

T. Experimental Category for Solid Products (Category S) (¹³New Category)

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

- (1) The product must meet the General Specifications list in Table 1 and Table 2 (this is not a typo; experimental solids must provide information required in Table 2).
- (2) 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 Clear Roads. The manufacturer shall submit all test results as required along with the following information:
 - a. Define the active ingredient that can be analytically measured.
 - b. Define the concentration of the active constituent at which the product will be manufactured. State the test protocols used for analyzing the primary constituent.
 - c. The manufacturer shall designate the appropriate gradation the product adheres.
 - ASTM D632 Type 1 Grade 1
 - ASTM D632 Type 1 Grade 2
 - ASTM D632 Type 1 Modified
 - d. Once the testing information is completed the manufacturer shall then follow the protocols in Section 1 for submitting samples and testing information to Clear Roads for Quality Assurance Testing. Upon request of Clear Roads, 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.
 - e. Clear Roads reserves the right to provisionally approve unique products that satisfactorily meet stated standards to allow for field testing. Provisionally approved products will be noted as such on the QPL. Provisional approval will remain until field testing and evaluations are complete and determined successful. If successful, the product will then be classified as a Qualified Product in the Experimental Category or an appropriate category if one is available for the product. If not successful, the product will be removed from the QPL.
 - f. Field testing of the products for this category may be conducted by the Clear Roads members or by agencies within the Associations' domain. If other than a Clear Roads member is conducting the testing themanufacturer shall be responsible for collecting the field data and submitting it to Clear Roads 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 Clear Roads can not only review questions of performance but also handling, storage, application information and any other information that Clear Roads feels is relevant regarding a productand its use.

¹³ Clear Roads added categories for like-products that were in the Experimental Category, therefore specifications in these categories are subject to change.

g. Anti-Caking agent¹⁴ will be included to ensure that the material remains free from hard caking and suitable for its intended purpose. **(Test Method 14)**

¹⁴ See footnote for Cyanide in Table 1.

CLEAR ROADS SNOW AND ICE CONTROL CHEMICAL PRODUCT PROGRAM

3. Test Methods

This section provides a list of the methods and the testing protocol. The list of all methods used or accepted by Clear Roads is provided in Table 3. The testing protocol for each method is provided in Section 3.2.

3.1 Table of Test Methods

Test Method	Description
Test Method 1	Percent Concentration of Active Ingredient in The Liquid
Test Method 2	Specific Gravity by ASTM D 1429 Test Method A - Pycnometer at 20°C ± -1°C.
Test Method 3	Clear Roads may use the test procedures provided by the vendor or manufacturer 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 qualified in the QPL process.
Test Method 4	ASTM D 1293 except a dilution shall be made of 1 part chemical product to 4 parts distilled water before attempting a reading.
Test Method 5	Corrosion Rate Determination as described in NACE Standard TM0169-95 (1995 Revision) and modified by Clear Roads.
Test Method 6	Test Percent Total Settleable Solids and Percent Solids Passing a 10 Sieve.
Test Method 7	Total Phosphorous as described in "Standard Methods for the examination of Water and Wastewater," APHA-AWWA-WPCF.
Test Method 8	Total Cyanide as described in "Standard Methods for the examination of Water and Wastewater," APHA-AWWA-WPCF.
Test Method 9	Atomic Absorption Spectrophotometry or Plasma Emission Spectroscopy as described in "Standard Methods for the examination of Water and Wastewater," APHA-AWWA-WPCF.
Test Method 10	Cold vapor atomic absorption spectrophotometry, as described in "Standard Methods for the examination of Water and Wastewater," APHA-AWWA-WPCF.
Test Method 11	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.
Test Method 12 ¹⁵	ASTM E 534 "Standard Test Methods for Chemical Analysis of Sodium Chloride."

Table 3. Test Methods

¹⁵ The moisture content is judged as available free water. Organic Based 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 prescribed test method but do not reflect the loss of available free water shall be limited to a maximum of 3% by weight. Additionally, the use

Test Method	Description
Test Method 13	Gradation shall be analyzed according to ASTM C 136 per reference from ASTM D632. 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 ensure 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.
Test Method 14	Visual inspection and field observations to assure that the material remains clean and free of extraneous matter, free from hard caking, does notsegregate, and remains suitable for the intended purpose and as otherwise outlined in Section 5.3 <u>NOTE</u> : Purchaser may use any laboratory test method necessary to verify conclusions from visual inspections.
Test Method 15	Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms; 4th ed. (PDF) (350 pp, 5 MB, October 2002, EPA- 821-R-02-013.
Test Method 16	Ammonia as described in "Standard Methods for the examination of Water and Wastewater," APHA-AWWA-WPCF.
Test Method 17	Total Kjeldahl Nitrogen as described in "Standard Methods for the examination of Water and Wastewater," APHA-AWWA-WPCF.
Test Method 18	Nitrate and Nitrite as Nitrogen as described in "Standard Methods for the examination of Water and Wastewater," APHA-AWWA-WPCF.
Test Method 19	Biological Oxygen Demand as described in "Standard Methods for the examination of Water and Wastewater," APHA-AWWA-WPCF.
Test Method 20	Chemical Oxygen Demand as described in "Standard Methods for the examination of Water and Wastewater," APHA-AWWA-WPCF.
Test Method 21	Frictional analysis shall be conducted on products that have been applied at the prescribed 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 frictional coefficient shall be calibrated and certified prior to use on the sample analysis.
Test Method 22	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

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 vendor at therequest of Clear Roads before approval to the Qualified Products List (QPL) 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	Description
	Whatman No. 541(or equal), 125 mm diameter filter paper seated in a Buchner Funnel.
Test Method 23	Chloride as described in "Standard Methods for the examination of Water and Wastewater," APHA-AWWA-WPCF.
Test Method 25	ASTM D1177: "Standard Test Method for Freezing Point of Aqueous Engine Coolant."

3.2 Testing Protocol

Test Method 1: Percent Concentration of Active Ingredient in the Liquid

Atomic Absorption or Inductively Coupled Plasma Spectrophotometry as described in "Standard Methods for the Examination of Water and Wastewater," APHA- AWWA-WPCF is acceptable. A description of use of Atomic Absorption to determine the percent concentration of Calcium Chloride or Magnesium Chloride follows.

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

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 (HNO₃)
- Lanthanum Oxide (La₂O₃)

Solutions

- Lanthanum Chloride
- Calcium Chloride and Magnesium Chloride Calibration Standards and Blanks
- Quality Control Solutions
- Calcium Chloride and Magnesium Chloride Deicer Solutions

(1) Preparation of 10% Lanthanum Chloride Stock Solution

- a. In a 2000 ml beaker add 200 ml of distilled water to 117.28 g of Reagent Grade Lanthanum Oxide.
- b. While stirring, very slowly add 500 ml of concentrated HCl (25 ml at a time). CAUTION! This reaction is extremely violent. Care should be taken so the solution does not overflow the beaker.

c. 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

<u>Calcium</u>

- a. 100 ppm Calcium Stock Solution for Dilutions
 - i. Pipet 10 ml of the 1000 ppm Calcium reagent solution into a 100 ml volumetric flask.
 - ii. 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)
 - i. Pipet aliquots of 20, 25, and 30 ml of the above 100 ppm Calcium stock solution into three different 100 ml volumetric flasks.
 - ii. 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. 100 ppm Magnesium Stock Solution for Dilutions
 - i. Pipet 10 ml of the 1000 ppm Magnesium reagent solution into a 100 ml volumetric flask.
 - ii. Using an Eppendorf pipet add 0.1 ml concentrated HNO₃ acid and dilute to volume with distilled water.
- b. Magnesium Standards for Calibration (10, 15, 20 ppm)
 - i. Pipet aliquots of 10, 15, and 20 ml of the above 100 ppm Magnesium solution into three separate 100 ml volumetric flasks.
 - ii. 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.

Blank Solution

- a. Blank Solution for Calibration
 - i. 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.

Quality Control Solutions

- a. Calcium Quality Control Check
 - i. Weigh 0.6762 g pre-dried CaCO₃ and place into a 1000 ml volumetric flask.
 - ii. Add 1 ml of concentrated HNO_3 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. Magnesium Quality Control Check
 - i. Weigh 1.5056 g (non-dried) MgCl₂-6H₂O and place into 1000 ml volumetric flask.
 - ii. Add 1 ml of concentrated HNO₃ and dilute to volume with distilled water.
 - iii. 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: 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.)

(3) Preparation of Liquid Chemical Products Sample Solution

- a. Solution A
 - i. Weigh approximately 2.500 grams of the liquid chemical product into a tared 500 ml volumetric flask.
 - ii. Record the sample weight to the nearest mg for final calculations.
 - iii. Add I ml HNO₃. Rinse the neck of the volumetric flask with a slight amount of distilled water and allow the sample to digest for one hour.
 - iv. Dilute to volume with distilled water. Label as solution A.
- b. Solution B (Working Chemical Product Solution)
 - i. Pipette 5 ml of Solution A into a 100 ml volumetric flask.
 - ii. Add 5 ml of 10% Lanthanum Chloride solution and dilute to volume with distilled water. Label as solution B (Dilution factor of 20).
 - iii. Repeat Step 2 so that each chemical product sample has a duplicate working solution.

(4) Atomic Absorption Spectrophotometer Operation

- a. Calcium
 - i. 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° angle. The flame, burner, and instrument are to be optimized for best detection.
 - ii. Calibrate the instrument using the blank, 20 ppm, 25 ppm, and 30 ppm standards for Calcium.
 - iii. Run the Calcium Quality Control solution. This result must be within plus or minus0.20 ppm of the known 27.10 ppm concentration before proceeding.

- iv. Once the Quality Control solution is within allowable limits, run the chemical product samples and their duplicates and record the results.
- v. Run the Calcium Quality Control solution again to assure accurate results.
- vi. Following the analysis, calculate the percent concentration of the sample and the duplicate sample for each chemical product using the following formulas in Section (5)i. 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.
- b. Magnesium
 - i. 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° angle. The flame, burner, and instrument are to be optimized for best detection.
 - ii. Calibrate the instrument using the blank, 10 ppm, 15 ppm, and 20 ppm standards for Magnesium.
 - iii. 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.
 - iv. Once the Quality Control solution is within allowable limits, run the chemical product samples and their duplicates and record the results.
 - v. Run the Magnesium Quality Control solution again to assure accurate results.
 - vi. Following the analysis, calculate the percent concentration of the sample and the duplicate sample for each chemical product using the following formulas in Section (5)ii. 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.

(5) Calculations

- i. Calculations for $CaCl_2$ on a sample weighing 2.550 grams:
- Factor = (110.99 CaCl₂)(1%)(Dilution Factor)(Initial Vol.) = 2.7692 (40.08 Ca) (10,000 ppm)

Dilution Factor: 20

Initial Vol: 500 ml

- % CaCl₂ = <u>(X ppm from AA)(Factor)</u> grams of sample
- Example: (28.20 PPM)(2.7692) = 30.6% CaCl₂ 2.5500 g chemical product

ii. Calculations for MgCl₂ base on a sample weighing 2.550 grams:

Dilution Factor: 20

Initial Vol: 500 ml

Example: (18.87 ppm)(3.9173) = 29.0% Ca 2.5500 g chemical product

Test Method 2: Determination of the Specific Gravity (Weight Per Gallon)

Determine Specific Gravity by ASTM D 1429 Test Method A Pycnometer at 20°C±1°C.

Test Method 3: Corrosion Control Inhibitor Presence and Concentration

Clear Roads may use the test procedures provided by the vendor or manufacturer 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 submittal process.

Test Method 4: Determination of the pH

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.

Test Method 5: Corrosion Rate

Test Method: NACE Standard TM0169-95 (1995 Revision) as modified by Pacific Northwest Snowfighters.

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 Clear Roads QPL. The Clear Roads QPL 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.

(1) 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 coupon used in the test procedure is subjected to the following process to assure accuracy in test results.

- a. Examine each coupon for metallurgical abnormalities and reject those that are suspect to flaws.
- b. All coupons are tested for Rockwell Hardness of C 38-45; coupons having hardness outside of this range are rejected.
- c. Acceptable coupons are stamped for identification. Wipe with suitable solvent to remove grease and oil.
- d. 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 agitate 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.
- e. Coupons are acid etched with 1 + 1 HCl for 3 minutes.
- f. The coupons are then quickly rinsed with tap water, distilled water, wiped dried and placed in chloroform.
- g. When the coupons are removed from the chloroform for use, they are placed 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 prescribed above prior to weighing. This will remove any oils that may be transferred to the coupons.)
- i. Each coupon shall be weighed to a constant weight. The constant weight shall be two consecutive mass determinations of each coupon within a minimum of 0.5 milligrams of each other. Removal of incidental flash rusting prior to weighing is not necessary.
- j. Three coupons are used in each flask for testing the chemical product solutions and determining the distilled water and sodium chloride control standard values.
- (2) 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:

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 square inches (Total surface area of the coupon.)

A = 3.17 square inches

(3) 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 and allowed to sit a minimum of 12 hours to stabilize and reach equilibrium, ensure solubility and to account for any reactivity that may occur.

- (4) The Corrosion Test
 - a. 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.
 - b. 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 millimeters as it will allow the vapor phase within the neck of the flask to vent excessively and can skew the results.
 - c. 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.
 - d. 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 keeps the coupons inside the air 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.
 - e. 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.
 - f. 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.
- (5) Cleaning of the Corroded Coupons
 - a. 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.

- b. The coupons are pre-washed under running tap water to remove any loosely adherent corrosion products.
- c. They are then placed into glass beakers containing the cleaning acid, which is composed of concentrated hydrochloric acid (HCl) containing 50 grams/liter SnCl₂ (stannous chloride) and 20 grams/liter SbCl₃ (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.)
- d. Allow the coupons to soak in the cleaning acid for a total of 15 minutes.
- e. Remove the coupons from the acid and rinsed with tap water followed by distilled water.
- f. Wipe with a paper towel or cloth to clean any residual deposit from the coupons.
- g. They are then returned to the cleaning acid and the procedure is repeated.
- h. After cleaning, the coupons are rinsed in chloroform, air dried for 15 minutes and weighed.
- i. Each coupon shall be weighed to a constant weight. The constant weight shall be two consecutive mass determinations of each coupon within a minimum of 0.5 milligrams of each other.
- (6) Evaluation of Coupons
 - a. 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)

- b. 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.)
- (7) 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 Table 4, the distilled water proved to have a corrosion value of 5.00 MPY and the salt had a value of 55.00 MPY. The table 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 Clear Roads: "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 an MPY value higher than 15.00 MPY is not acceptable.

(8) Negative Numbers

Some products may 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 Table 4. 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.

(9) 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		
	Acme Melter has a corrected value of 19.99		
Therefore:	(19.99 / 50.00) X 100 = 40.0% Fail		

Table 4. Example of Chemical Product 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	N/A

Note: * represents an Acceptable Product. 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 mayvary from test to test.

Test Method 6: Percent Total Settleable Solids and Percent Solids Passing a 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.

(1) Apparatus

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

(2) Test Method

- a. Place 1000 ml of a well-mixed (non-diluted) liquid chemical product into a graduated one-Liter Imhoff cone.
- b. Place this sample into a freezer, which has been pre-calibrated and stabilized to the correct specified temperature as established in each liquid chemical product category.
- c. Cover the sample with a watch glass.
- d. 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.
- e. After 168 hours the sample is carefully removed from the freezer for testing.
- (3) 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

- a. 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.
- b. 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.
- c. Determine and record the interface layer volumes of the inhibitor and the concentrated amount of material in the bottom of the cone.
- d. 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.
- e. Gently insert the rod into the cone containing the product and gradually lower the rod to the bottom of the cone.
- f. 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.
- g. If the rod goes completely to the bottom of the cone with no resistance record that no dense settlement was found.
- h. 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.
- i. 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.
- (4) 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.

- a. 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.
- b. 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.

- c. 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.
- d. 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 side of the sieve the material was place on to pass through.
- e. Material that is trapped in the mesh of 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.
- f. 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.

Test Method 7: Total Phosphorus

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

Test Method 8: Total Cyanide

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

Test Method 9: Total Arsenic, Barium, Cadmium, Chromium, Copper, Lead, Selenium and Zinc

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

Test Method 10: Total Mercury

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

Test Method 11: Milliequivalents OR "meq"

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.

Test Method 12: Moisture Content of Solid Chemical Products

Perform according to ASTM E 534

Test Method 13: Gradation

Gradation shall be run 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 ensure 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.

Test Method 14: Visual Inspection and Field Observations

Visual inspection and field observations to assure that the material remains clean and free of extraneous matter, free from hard caking, does notsegregate, 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.

Test Method 15: Toxicity Test

Test according to <u>Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving</u> <u>Waters to Freshwater Organisms; 4th ed. (PDF)</u> (350 pp, 5 MB, October 2002, EPA-821-R-02-013)

Test Method 16: Ammonia - Nitrogen

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

Test Method 17: Total Kjeldahl Nitrogen

Test Total Kjeldahl Nitrogen as described in "Standard Methods for the examination of Water and Wastewater," APHA-AWWA-WPCF.

Test Method 18: Nitrate and Nitrite as Nitrogen

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

Test Method 19: Biological Oxygen Demand

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

Test Method 20: Chemical Oxygen Demand

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

Test Method 21: Frictional Analysis

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.

Test Method 22: Insoluble Material

Test according to ASTM E534 "Standard Test Methods for Chemical Analysis of Sodium Chloride" with the following modification: dissolve 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.

Test Method 23: Chloride

Test as described in "Standard Methods for the examination of Water and Wastewater," APHA-AWWA-WPCF.

Test Method 24: Eutectic Temperature

Test as per ASTM D1177.

4. Purchasing

The purpose of this section is to provide *suggested* language for agencies to include in their Request for Proposals (RFP) or other bid request documents and in the final contract document, so vendors have an understanding of expectations. In contract language "vendor" would be replaced with the vendor name.

4.1 Bidding

A. Bidding Criteria

To bid a product, that product shall fulfill either of the following criteria:

- (1) Be on the most current Clear Roads QPL, or
- (2) Be "in process" for inclusion on the Clear Roads QPL as part of the 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 Clear Roads QPL committee 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 vendor (some agencies are ok with this provision, others won't accept a product "in process").
- (3) 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 vendor 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 Clear Roads QPL 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.
- (4) An anti-foaming agent will be available from the Vendor for use as needed, at no additional charge to the Purchaser, to control foaming during loading, unloading, and agitation of liquid chemical products.

B. Liability

- (1) The vendor 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.
- (2) The vendor 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.

4.2 Deliveries and Invoicing of Products

A. Deliveries

(1) Vendor 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.

- (2) Each shipment shall be accompanied by a current and clearly legible SDS.
- (3) The bill of lading for each shipment must contain the following information.
 - Name of product.
 - Supplier and manufacturer of product.
 - Delivery Destination.
 - Total number of units being delivered.
 - Total weight of delivery using a certified scale ticket (or certified flow meter if your agency certifies flow meters).

As an option on liquid deliveries only, the vendor can use a legibly printed certified ticket from a flow meter that has been tested and certified a Clear Roads member state. The certification of the meter shall not be older than one year. Any Clear Roads member can request that the meter be re-tested and certified again during the delivery year if the data from the meter is in question. This re-testing and certification shall be done at no extra charge to the Clear Roads member. Reciprocity among Clear Roads members for meter calibration may be employed. <u>The vendor shall provide a copy of the certification and</u> <u>product information about the flow meter at the time of bid.</u> The Clear Roads 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 ensure the accuracy of the flow meter.

- (4) Lot Number for the product being 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.
- (5) Transport information--Name of transporting company, tank, trailer or rail car number, point and date of origin.
- (6) For liquid products include the Vendor Quoted Concentration and Specific Gravity.

B. Invoicing

- (1) The Agency will not process invoices for payment until the vendor has metall requirements under this section. The invoice shall include the following:
 - A copy of the original bill of lading.
 - Contract unit of measure.
 - Total number of units delivered.
 - Contract unit price for product delivered.
 - Total price for units delivered.

4.3 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 are the prerogative of the Purchaser. The vendor shall not off load any material without affording the Purchaser an opportunity to conduct the field inspection, sampling or the testing. Offloading 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 vendor 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:
 - Date of the order.
 - Date and time of delivery.
 - Verification of advance delivery notification.
 - Delivered within allowable times.
 - Name of Delivery Company
 - Are any price adjustment assessments required?
 - Is the product being delivered what you ordered?
 - Document all procedures prior to unloading of product.
 - Verify that all papers required of a delivery are present, complete, and legible.
 - Bill of lading and/or invoice.
 - o Current SDS sheet.
 - o Certified weight slip.
 - Verify separation or non-separation of product.
 - Visually inspect the load to determine if there are any obvious reasons why theload should be rejected.
 - 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.
 - 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 instorage 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

- (1) 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 ensure 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:
 - Manufacturer or vendors 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.

<u>Test results from the appropriate Laboratory will be final and in the best interest of the</u> <u>Purchaser.</u>

- (2) 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.
- (3) 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.

(4) 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.

4.4 Product Rejection and Price Adjustments

PLEASE REFER TO EACH INDIVIDUAL AGENCY'S SPECIFICATIONS FOR PRODUCT REJECTIONS AND PRICE ADJUSTMENTS.

For categories that have specifications that address % insoluble material, the following is suggested contract language:

Example for category 4: 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)
```

For categories that have specifications for moisture content, the following is suggested contract language:

Example for category 8B: 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)
```

4.5 Bid Evaluation Process

A. Bid preferences for higher concentrations

(1) (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. Vendor Quoted Concentrations (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 vendors 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. Bid preferences for superior corrosion inhibition

(1) (Approved Liquid and Solid Chemical Products)

STEP 2: Bid preferences based on the corrosion inhibiting ability of a product as indicated on the QPL. 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.

C. Acceptance of Bids

(1) Will be based on approved Clear Road's QPL. 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 and 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.

4.6 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>."

4.7 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. Vendors can bid their approved products. <u>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 you are not entering a bid for an Area of the selected category enter a "No Bid" for that line item.</u>

A. 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 locations within the Area and will be used in the analysis for the "Final Best Buy Factor." <u>The bid will be award based on the lowest "Final Best Buy Factor" of each category per</u> <u>Area.</u>

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_____

Vendor's Name

Vendor Quoted Concentration of Product_____

ATTACHED AGENCY BID LIST FOR AREAS, LOCATIONS AND QUANTITIES.

B. 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 locations within the Area and will be used in the analysis for the "Final Best Buy Factor." <u>The bid will be awarded based on the lowest "Final Best Buy Factor" of each category</u> <u>per Area (if applicable).</u>

Identify the Category for which you are bidding and provide the product name and the name of your company as well as the information below:

Category _____

Product Name _____

Vendor's Name

Does your Product contain any Anti-Caking agent? (Circle One) YES NO

If your Product does contain an anti-caking agent, please provide the following information:

Amount of anti-caking agent added per ton of product: _____

What is the name of the anti-caking agent you are adding?

ATTACHED AGENCY BID LIST FOR AREAS, LOCATIONS AND QUANTITIES.