



Transportation Synthesis Report

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Anti-Caking Admixtures to Road Salt

Prepared for
Bureau of Highway Operations
Division of Transportation Infrastructure Development

Prepared by
CTC & Associates LLC
WisDOT RD&T Program
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Transportation Synthesis Reports (TSRs) are brief summaries of currently available information on topics of interest to WisDOT technical staff in highway development, construction and operations. Online and print sources include NCHRP and other TRB programs, AASHTO, the research and practices of other state DOTs, and related academic and industry research. Internet hyperlinks in TSRs are active at the time of publication, but changes on the host server can make them obsolete.

REQUEST FOR REPORT

Wisconsin is the lead state for a new pooled fund study, TPF-5(092) Test and Evaluation of Materials, Equipment and Methods for Winter Highway Maintenance. As part of the research selection process for the upcoming year, members of the pooled fund Technical Advisory Committee are considering whether a study of anti-caking agents for road salt would be an appropriate research topic. WisDOT's RD&T Program was asked to survey state practices in this regard and review available literature.

SUMMARY

A survey of state DOT practices was carried out through the AASHTO Research Advisory Committee listserv with the following questions:

- Have you had significant problems with crusting of stored road salt?
- Do you require anti-caking agents to be added to stored road salt?
- Do you specify the anti-caking agent to be used? If so, what is the specification?
- Do you specify the concentration of the anti-caking agent? If so, what is the concentration?
- Have you taken other measures to address caking problems in stored salt? If so, please describe.
- Other comments.

Survey responses are summarized below (**SURVEY**) and tabulated in **Appendix A: Road Salt Anti-Caking Survey**. We then review related specifications from the Pacific Northwest Snowfighters and Maine DOT (**SPECIFICATIONS** and **Appendix B: Maine DOT Specifications**). Following this section, we highlight several Web sites devoted to **SALT MANAGEMENT PRACTICES**. Finally we review some of the issues related to **FERROCYANIDES AND THE ENVIRONMENT**.

SURVEY

In the April 16, 2004 survey of state practices, 19 agencies responded. See full responses in Appendix A.

Anti-caking Agent Type. Of the 19 respondents, 13 require anti-caking agents in road salt they use, and eight of these specify the agent to be used. Six states require sodium ferrocyanide, known as Yellow Prussiate of Soda (YPS), and the other two require either YPS or an equivalent, typically ferric ferrocyanide, known as Prussian Blue. Clearly, then, YPS is the standard anti-caking agent employed.

Concentration. Specified concentrations of YPS vary widely from as little as 10 parts per million to 250 ppm. Prussian Blue ranges from 70 to 165 ppm. Though it doesn't specify a particular anti-caking agent, Virginia DOT does specify concentration of whatever agent is used to be from 0.1 to 0.2 pounds per ton (50 ppm to 100 ppm). Oklahoma does require that YPS is used, but does not specify the concentration.

Moisture and Fines. Moisture content seems the clearest factor in caking or crusting problems with stored road salt. Fully 11 of the 13 states reporting that they require anti-caking agents in their road salt are coastal or Midwestern states, areas in which atmospheric humidity is common year-round. Four respondent states—Arkansas, Maine, New Jersey and Virginia—monitor moisture content in salt, and all except Arkansas consider price adjustments against the unit bid price for high moisture contents. Of states that monitor moisture content, however, thresholds for acceptability without penalty range from 0.5% to 2%, and some states accept, according to an Iowa survey, 3%. Iowa DOT notes that handling of salt can also increase levels of fines, which the agency considers a contributor to caking or crusting problems.

SPECIFICATIONS

Canada, Finland, Sweden and some U.S. states appear to have conducted research on anti-caking ferrocyanides. However, specifications, standards of treatment for anti-caking road salt, and road-testing studies of such treatments have not been made readily available online. One consortium of Northwest states and provinces—Pacific Northwest Snowfighters—posts its specifications for road salt mixtures on its Web site, and Maine has sent us its specifications.

Pacific Northwest Snowfighters. Participants in this consortium include transportation agencies from Washington, Oregon, Montana, Idaho and British Columbia. All sodium chloride road salts—with or without corrosion inhibitors, with or without 10 or 20 percent magnesium chloride—face the same requirements for anti-caking agents. Such agents used are ferric ferrocyanide (Prussian Blue), or Sodium Ferrocyanide (Yellow Prussiate of Soda-YPS). The PNS specification notes that YPS is used in table salt. Prussian Blue can be added at 70 to 165 parts per million (0.33 to 1.14 pounds per ton of salt), and YPS at 50 to 250 ppm (0.1 to 0.5 pounds per ton). Furthermore, use of such additives is not assessed against total cyanide concentration levels acceptable to these road salt specifications. See Chemical Product Categories 4, 5, 6 and 8, beginning on page 13 of the specifications, at http://www.wsdot.wa.gov/partners/pns/pdf/PNS_SPECS_2002_FINAL.pdf. See also the PNS site, <http://www.wsdot.wa.gov/partners/pns/>.

Maine. MaineDOT specifications vary the acceptable level of sodium ferrocyanide according to moisture content. For salt with moisture content below 0.5%, the anti-caking agent must be present at concentrations of 10 to 50 ppm. For salt with moisture over 0.5%, the anti-caking agent concentration must be 50 to 100 ppm. Maine also specifies the sampling method and test method to be employed for determining YPS concentrations. See attached specifications below in Appendix B.

SALT MANAGEMENT PRACTICES

Research in Progress. *Determination of Moisture Content of De-Icing Salt at Point of Delivery*, finished December 2003 – <http://rip.trb.org/browse/dproject.asp?n=8382>. Investigators aim to develop a quick, simple and affordable method to measure moisture content of road salt in the field in a range of 3% to 5% moisture content with an accuracy of +/- 0.5%. See University of Connecticut investigator's details at http://www.netc.uconn.edu/ti/Research/netc_fy02.htm#netc025.

Salt Institute. The Salt Institute offers a variety of fact sheets, white papers, and training programs in salt management.

- The *Salt Storage Handbook* claims that most producers add anti-caking agents, and while there is much information regarding storage of salt here, further reference to anti-caking agents is absent – <http://www.saltinstitute.org/snowfighting/sshandbook.zip>. (Very large—78 MB—Zip file.)
- “Snowfighters,” the Salt Institute’s salt management training program, is detailed on the site. See <http://www.saltinstitute.org/news04-mar.html> (March issue of SI Report).

Road Salt Management Site, Transportation Administration of Canada. This site lists contacts, a brochure on practices, and details regarding online courses and road salt management manuals developed by the TAC. See <http://www.tac-atc.ca/roadsalt/roadsalt.htm>.

American Public Works Association. APWA created a CD-ROM, *Salt Management Guide*, which contains details on the chemical components of road salt, their impact, and use. See <http://www.apwa.net/bookstore/detail.asp?PC=PB.X308>.

FERROCYANIDES AND THE ENVIRONMENT

At this time, sodium ferrocyanide and ferric ferrocyanide appear to be the only additives used to impede caking or crusting in stored road salt. States that deal with anti-caking agents in road salts share certain practices, according to our Internet search and survey of state winter operations. Sodium ferrocyanide—usually in the form of the product Yellow Prussiate of Soda (YPS)—seems to be the favored anti-caking agent employed around the country. The alternative to YPS sometimes employed is Prussian Blue, a ferric ferrocyanide product.

Concern over the environmental impact of ferrocyanides has been most acute in Canada. In the U.S., the FHWA has joined with the Environmental Protection Agency to designate ferric ferrocyanides as toxic, but the agencies have stopped short of banning its use in road salt, arguing that concentrations are not significant enough to cause ecological or public health concern. Unlike Canada, official U.S. concerns do not include sodium ferrocyanides, which may explain the widespread popularity of YPS over Prussian Blue. Scandinavian countries and several U.S. states concerned with the environmental impact of road salt typically focus on salinity or chloride levels in groundwater, and encourage restrained use of road salt. Restrictions on specific levels of ferrocyanides from anti-caking additives do not obtain.

While there is some disagreement on the toxicity of ferric ferrocyanide (in Prussian Blue) being matched by that of sodium ferrocyanide (in YPS), scientific studies seem to support the FHWA and EPA position that it is the former, not the latter, that poses the most risk.

Road salt itself is a potentially problematic toxin. The EPA's recent declaration of ferric ferrocyanide (Prussian Blue) as a "toxic pollutant" and "hazardous substance" does not in the short-term preclude the use of it in road salts. But there is potential for future determinations of FFC-laden road salt damage that could have implications for litigation and regulation; hence, its use in highway programs should be carefully considered. See FHWA memo from Oct. 2003, and its links to official EPA pronouncements on FFC—<http://www.fhwa.dot.gov/environment/toxsalt.htm>.

The Salt Institute cites and echoes EPA and FHWA positions. In *Highway Salt and Our Environment* (2004), the Salt Institute claims ferrocyanides pose no health risks, citing in footnotes various publications. See §5.4 on p. 23 of <http://www.saltinstitute.org/publications/saltandenvironment-english.pdf>. See also its summary of the recent EPA findings on FFC at <http://www.saltinstitute.org/ffc.html>.

Minnesota. An environmental watch group says the ferrocyanides are a problem not in the widely spread salt, in which its levels are too small to be dangerous, but in runoff from salt storage piles—see http://www.duluthstreams.org/understanding/impact_salt_2.html. Note the above claim's source—Minnesota Environment November 2000: "Worth His Salt," <http://www.pca.state.mn.us/publications/mnenvironment/fall2000/salt.html>.

Canada and Sodium and Ferric Ferrocyanides. In Canada, both sodium and ferric ferrocyanides are seen as problematic. Environment Canada determined in January 2001 that the ferrocyanide content of road salts needed to be reduced. This was one element of findings that various road salt constituents have adverse impacts on groundwater, flora and fauna. See a clear, detailed article at <http://www.esemag.com/0101/salt.html> and more at <http://www.esemag.com/0901/pickling.html> and <http://www.esemag.com/0102/news.html>. See also the Canadian Web site www.ec.gc.ca/CEPARRegistry.

Environment Canada. The August 2000 draft for public comments, at 156 pages, is a huge summary of information. [Priority Substances List Assessment Report: Road Salts](http://www.ec.gc.ca/nopp/roadsalt/reports/en/rms.cfm). A road salt risk management strategy details environmental impacts—see <http://www.ec.gc.ca/nopp/roadsalt/reports/en/rms.cfm>.

Effects of Sodium Ferrocyanide Derived From Road Salting on the Ecosystem. Assessment and Appendices, summary at <http://trisonline.bts.gov/sundev/detail.cfm?ANNUMBER=00943816&bypass='y'&CFID=496950&CFTOKEN=2195156>. This May 2000 study focused on sodium ferrocyanide, finding that it breaks down too quickly to significantly endanger terrestrial or aquatic biota, save in urban roadside ditches, where it collects.

Swedish National Road Administration. Because of concerns for groundwater quality, the SNRA's environmental plan for 2002-2005 aims at reducing sodium chloride use. Note that constituents of road salt are nowhere mentioned; concern for ferrocyanides does not seem to be a factor in setting the goal of reducing road salt use. http://www.vv.se/for_lang/english/publications/Miljoprogram_english_KO.pdf.

Appendix A: Transportation Synthesis Report on Anti-Caking Agents for Road Salt

Wisconsin Department of Transportation RD&T Program

Survey of AASHTO RAC Listserv April 16, 2004

	STATE	Have you had significant problems with crusting of stored road salt?	Do you require anti-caking agents to be added to stored road salt?	Do you specify the anti-caking agent to be used?	If so, what is the anti-caking agent specification?	Do you specify the concentration of the anti-caking agent?	If so, what is the anti-caking agent concentration specification?	Have you taken other measures to address caking problems in stored salt?	If so, please describe	Additional Comments
1	ARKANSAS	No	No	No		No		No		
2	CALIFORNIA	No	Yes, except in Tahoe Basin, which uses only kiln-dried salt	Yes	Yellow Prussiate of Soda (YPS) -- sodium ferrocyanide	Yes	50-80 ppm	Yes	Occasionally work stockpile with loader	
3	COLORADO	No	No	No		No		No		We only stock salt/sand mixtures; we use no pure salt.
4	CONNECTICUT	No	No	No		No		No		All our salt piles are stored in temporary or permanent sheds. Some caking on top layers occurs, but has not been problematic.
5	IOWA	A big problem, at some locations.	Yes	Yes	Yellow Prussiate of Soda (YPS) -- sodium ferrocyanide	Yes	50-250 ppm. Wide range frees salt vendors from customizing their stockpiles, adding expense	Yes	We select a few locations each year to remove old salt. All deliveries covered and meet one percent moisture content requirement. We push for less handling of salt, which can lead to packing.	A survey we conducted showed moisture content varies significantly around the country, with up to three percent moisture content required by some. We have intensified our enforcement of covered loads and inspection to improve initial quality, and suspect that moisture and fines cause most of our problems with caking.
6	KANSAS	Not in recent years	Yes	Yes	Yellow Prussiate of Soda (YPS) -- sodium ferrocyanide	Yes	25 ppm, minimum	Yes	Constructed new salt domes 10-15 years ago; recently added concrete salt bunkers with fabric roofs to protect from weather	
7	KENTUCKY	No	Yes	No		No		No		
8	MAINE	No	Yes	Yes	YPS or equal; colored is preferred	Yes	If moisture content less than 0.5%, 10-50 ppm; if moisture content over 0.5%, 50-100 ppm	Yes	Inspection, sampling, and price adjustment if moisture content too high	
9	MISSOURI	No	No	No		No		No		MoDOT specifications require that the "sodium chloride shall arrive at the delivery point in a free-flowing and usable condition." Most suppliers add sodium ferrocyanide (Yellow Prussiate of Soda).
10	NEBRASKA	No	No	No		No		No		Upon delivery, most yards treat with anti-icing agent -- such as ICEBAN 100, Caliber 1000, GEOMELT, Liquid Corn Salt -- at about 5-7 gallons per ton.
11	NEW JERSEY	No	Yes	No		No		Yes	If moisture 0.5-1.5%, reduce 1% price per 0.5% moisture. Clay size fraction of insoluble residue cannot exceed 1 percent of total material.	Salt gets used almost as fast as delivered. This and contract requirements seem effective in preventing caking.

Appendix A: Transportation Synthesis Report on Anti-Caking Agents for Road Salt

Wisconsin Department of Transportation RD&T Program

Survey of AASHTO RAC Listserv April 16, 2004

	STATE	Have you had significant problems with crusting of stored road salt?	Do you require anti-caking agents to be added to stored road salt?	Do you specify the anti-caking agent to be used?	If so, what is the anti-caking agent specification?	Do you specify the concentration of the anti-caking agent?	If so, what is the anti-caking agent concentration specification?	Have you taken other measures to address caking problems in stored salt?	If so, please describe	Additional Comments
12	NORTH DAKOTA	Yes, before specifying anticaking agent	Yes	Yes	Yellow Prussiate of Soda (YPS) -- sodium ferrocyanide	Yes	25 ppm	No		It is very noticable to us if the anticaking agent is introduced or not. Prior to spec-ing an anticaking agent, we would get 7 in.-10 in. clumps that would not pass through sanders, or loads that would not flow out of the transport vehicle, requiring jack hammers to break up and payloaders to drive on.
13	OKLAHOMA	No	Yes	Yes	Yellow Prussiate of Soda (YPS) -- sodium ferrocyanide	No		No		
14	OREGON	No	No	No		No		No		Do not use NaCl. Use magnesium chloride and some calcium magnesium acetate.
15	TENNESSEE	Not recently	Yes	No		No		No		When our current state-wide contract expires, we plan to provide a more detailed specification.
16	VIRGINIA	Yes, particularly during summer	Yes	No		Yes	0.1-0.2 lbs per ton	Yes	Road salt must contain at least 95 % sodium chloride; moisture content must not exceed 2%; samples allow 5% tolerance on 3/8 in. sieve, but all plus-3/8 in. must pass 1/2 in. sieve	Rejection standards -- when more than 1/3rd of samples show any of following: moisture content over 3%; sodium chloride content under 92%; stockpile deviation from specification over 10%; or presence of 3/4 in. or 1/2 in. material. Penalty at engineer's discretion -- 2% unit bid price reduction for each 1% moisture about 2%; 2% unit price reduction for each 1% deviation from specified gradation on 3/8 in., #4, #8, and #30 sieves; 2% unit price reduction for each 1% sodium chloride below 95 %. Storage -- road salt is stored in non-airtight buildings sealed against groundwater contamination.
17	WASHINGTON	No	Yes	Yes	Prussian Blue (ferric ferrocyanide), or Yellow Prussiate of Soda (YPS -- sodium ferrocyanide)	Yes	Prussian blue, 70-165 ppm. YPS, 50-250 ppm	Yes	Cover stockpiles	
18	WISCONSIN	No	Yes -- we require salt to be "free flowing" with sufficient additive to keep it so	No		No		Yes	Store under cover on an impermeable pad	WisDOT's current contractor, North American Salt, uses sodium ferrocyanide (YPS) as their anti-caking agent
19	WYOMING	No	Yes	Yes	Yellow Prussiate of Soda (YPS) -- sodium ferrocyanide	Yes	200 ppm	No		Wyoming uses abrasives with enough salt to keep piles relatively chunk-free

Appendix B Maine DOT Salt Specifications

(Anti-caking provisions are shown in **BOLD**.)

The State shall have the authority when receiving salt not meeting specifications or delivered in a manner not meeting these specifications to shut the vendor down and purchase salt from the nearest suitable source of supply. It shall be left up to the Department of Transportation and the Bureau of Purchases to determine what arrangements can be made with other suppliers to get a supply of salt within a reasonable time frame.

Should the delivered price per ton be the same or relatively close between suppliers, the award shall be determined jointly by the Department of Transportation and the Division of Purchases with consideration being given to the vendor supplying from covered storage.

Reductions: Reductions for moisture will be made for moisture content exceeding 1.0%. Moisture contents over 2.5% are considered unsatisfactory and loads shipped under such circumstances can be returned to the vendor at no cost to the State.

Invoices: Separate invoices are required for each order number. Invoices shall include the stockpile location the order is taken from. Each load is to be listed separately with the date delivered on the invoice. Partial orders will not be paid for unless the State has over-ordered for a particular location. Matching delivery slip numbers for each order should be included on the invoice. Invoices received with the delivery slip numbers will be processed prior to invoices without delivery slip numbers.

Anti-cake: Uniform application of anti-cake is essential to prevent freezing and caking. Evidence on non-uniformity or rates not conforming to the specifications shall be grounds for rejection.

Delivery: It is assumed that companies making deliveries in inclement weather shall cover the load to keep moisture out. Uncovered loads may be rejected at the site if excess moisture is apparent.

Each delivery area's order shall be completed within four (4) hours of delivery of the first load or as otherwise directed by the Division. Initial delivery shall occur within 5 days of an order being placed unless otherwise authorized by the Division.

Deliveries made after working hours will not be accepted unless the vendor gets prior approval from the Division concerned.

June, 2001

STATE OF MAINE
DEPARTMENT OF TRANSPORTATION
BUREAU OF MAINTENANCE & OPERATIONS
Augusta, Maine

Specifications for Sodium Chloride

Classifications

This specification covers sodium chloride obtained from natural deposits (rock salt), or produced by Man (evaporated, solar, other) shall conform to AASHTO M-143-86 (1993) except for specifics as noted below.

Chemical Requirements

The sodium chloride shall conform to a chemical composition of a minimum of 95.0 percent. Variations of less than 95% but above 92% will result in acceptance at reduced payment.

Physical Requirements

A. The gradation when tested by means of laboratory sieves shall conform to the following requirements for particle size without any variations:

<u>Sieve Size</u>	<u>Percent Passing By Weight</u>
1/2 inch	100
3/8 inch	95-100
No. 4	20-90
No. 8	10-60
No. 30	0-15

Because of restrictions used to calibrate salt on Department sanders, the 1/2" gradation requirement is critical. The contractor will be required to re-screen the sodium chloride for continued oversize deliveries.

B. In order to prevent caking while in storage following delivery, all sodium chloride shall be uniformly treated with an anti-caking conditioner (YPS or equal). The conditioner shall be uniformly applied to the sodium chloride at a rate which when sampled shall have minimum of 10 parts per million (PPM) and a maximum of 50 (PPM), for salt with moisture contents less than 0.5%. For salt delivered with greater water content than 0.5%, a minimum of 50 (PPM) and a maximum of 100 parts per million of the conditioner shall have been applied. The use of colored conditioner is desired.

Condition

The sodium chloride shall arrive at the purchaser's delivery point in a free flowing and useable condition.

Sampling

Each sample submitted for testing shall consist of a minimum of three increments selected at random from the material. Each increment shall be at least a pound in weight and not include the top inch of stored material. Every attempt shall be made to insure a composite sample representative of the pile. Samples for chemical composition, gradation and anti-caking may also be obtained at the time of delivery to the purchaser's delivery destination.

Method of Testing

1. Chemical composition testing for sodium chloride shall conform to the rapid method of testing given in Appendix A1 of AASHTO M-143-86. A second method of rapid analysis could be the use of Gran's plot titration method developed by Orion Research Incorporated. In case that the coarse salt sample is used 100.00 g sample is taken and dissolved in a 2-liter volumetric flask quantitatively. An aliquot of 5.00 ml or 2.00 ml of this solution is then pipetted out to the titrated according to either of these two methods.

If controversy occurs, analysis shall be made in accordance with the method specified for "Salt" in the current "Official Methods of Analysis for the Association of Official Agricultural Chemists".

2. Particle size analysis shall consist of using sieves with square openings mounted on substantial frames or interlocked in a manner as to prevent loss of material during sieving. Woven wire cloth shall conform to AASHTO M92. Gradation analysis shall be made on oven dried material and the sample shall weigh after drying at least 500 grams. In no case shall the fraction retained on any sieve at the completion of the sieving operation weigh more than four grams per square inch of sieving surface (this is 200 grams for 8 inch diameter round sieves).

If sieving is completed by hand, the procedure will include shaking with lateral and vertical motion with occasional jarring action so as to keep the material moving continuously over the surfaces of the sieves for a time of not less than three minutes. The balance or scale shall be sensitive to within 0.2% of the weight of the sample to be tested.

3. Moisture content shall be determined by a method of weighing before and after oven drying at $110^{\circ} + 5^{\circ}$ C ($230^{\circ} + 9^{\circ}$ F) for a minimum of four hours.
4. **The test for anti-caking material shall be made following the attached method for colorimetric determination of YPS or YPP treated salt. For an anti-caking material other than YPS or YPP the successful bidder shall indicate the chemical nature of the material and shall furnish a laboratory procedure for determining the**

**amount of anti-caking material to the Maine Department of Transportation,
Testing Engineer, P.O. Box 1208, Bangor, Maine 04401 within 30 days of the award
of the bid by the Bureau of Purchases.**

Inspection

The purchaser or his representatives shall be provided free entry and access at the storage areas for sampling. It is anticipated that determination for initial compliance will be made from samples obtained from the supplier's storage areas or from rail cars if no storage areas are provided within the State. Shipments to the purchaser's points of delivery may be randomly sampled for moisture content and gradation control. If in doubt, complete analysis may be made on these samples. It is the intent of the purchaser to provide inside storage facilities for all sodium chloride to insure a minimizing of increased moisture content. The supplier must be responsible for insuring a similar concern over moisture absorption between his storage area and that of the purchaser's point of delivery.

Penalty and Price Adjustment

- A. For each percent by which sodium chloride is less than 95%, but greater than 92%, the supplier will be penalized by reducing the payment of salt delivered by one percent (1%).
- B. Reductions for moisture content of the sodium chloride shall be made when the moisture exceeds 1.0%. Sodium chloride with a moisture content up to 2.5% will be accepted with a reduction as shown below:

M.C. = Moisture Content

- 0 to 1.0% moisture = No reduction
- 1.0 to 1.5% moisture reduction = 1 times (M.C. minus 0.5%)
- 1.5 to 2.5% moisture reduction = 2.0 times (M.C. minus 0.5%)
- Over 2.5% moisture - rejected - unless emergency conditions exist whereby the State elects to keep the salt - then the moisture content reduction will be 3.5 times (M.C. minus 0.5%).

- C. For each percent by which gradations exceed the maximums allowed, the contractor will be penalized by reducing the price of the salt by one percent (1%).

Payment

Random sampling and testing of sodium chloride shall be obtained and tested by Maine Department of Transportation personnel. Failing test reports shall be used to calculate reduced payments when penalties and price adjustments are to be applied. The reduction in payment shall continue until such time as a new passing test is obtained.

When on site inspections and tests indicate salt being supplied is an inferior product, the supplier will be given fourteen (14) calendar days to supply a product satisfactory to the State. After that time the M.D.O.T. will request the Bureau of Purchases to make

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Wisconsin Department of Transportation - May 6, 2004***

arrangements with other suppliers to finish out the original supplier's obligation with specification material at prices negotiated at the time.

METHOD FOR THE COLORIMETRIC DETERMINATION

YPS TREATED ROCK SALT

Scope of Method

This colorimetric procedure is applicable in determination range 0-100 ppm of Sodium Ferro cyanide $\text{Na}_4\text{Fe}(\text{CN})_6 \cdot 10 \text{H}_2\text{O}$ (YPS) utilizing a colorimeter such as the Coleman Jr. II spectrophotometer.

Reagents

- 1. Sodium Ferro cyanide $\text{Na}_4\text{Fe}(\text{CN})_6 \cdot 10 \text{H}_2\text{O}$**
- 2. Ferrous Sulfate $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$**
- 3. Sulfuric Acid, concentrated**
- 4. Sodium Chloride, reagent grade**
- 5. Sodium Hydroxide, reagent grade**

Reagent Solutions

A. Sodium Ferro cyanide Solutions

- 1. 0.1% solution - weigh exactly 1.000 gram of Sodium Ferro cyanide, dissolve in distilled water and dilute to 1 liter.**
- 2. 0.05% (500 ppm) solution - take 50 ml aliquot of the above 0.1% solution and dilute to 100 ml.**

B. Ferrous Sulfate (5% solution)

Dissolve 5 grams $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$ in approximately 50 ml of water, add 2 ml concentrated Sulfuric Acid and dilute to 100 ml.

C. Sodium Hydroxide Solution (2%)

Dissolve 20 grams of NaOH in 1 liter of water.

D. Sulfuric Acid, 1:5

Add 20 ml of conc. H_2SO_4 slowly into 100 ml of water, mix well and let cool.

Note: Solutions A and B should be prepared fresh daily or as required.

Preparation of Standards

Six 25 gram samples of reagent grade NaCl are weighed (to 0.01 g) and placed in 250 ml beakers. To each of the samples add 0, 1, 2, 3, 4 and 5 ml of 0.05% sodium Ferro cyanide solution, respectively, (0 ml being a blank) mix well. Prepare standard solution by adding NaOH and H₂SO₄ as described in the sample preparation except NO filtration is needed. To all six flasks add 5 ml of the Ferrous Sulfate solution, bring to 100 ml mark with water, cover flasks with stoppers and mix well. Maximum intensity of color develops in 15 minutes. The standards now indicate 0-100 ppm of Sodium Ferro cyanide in increments of 20 ppm with respect to the original salt sample.

Determination of Sodium Ferro cyanide in Rock Salt

The bulk sample is split down to representative sample of about 300 grams. This portion is then pulverized such that it will all pass a 70 mesh sieve and is mixed thoroughly to ensure good homogeneity. A 25 gram portion of this material is mixed with 5 ml 2% NaOH, stirred and let set for 10 min., 60 ml of water is then added into this solution and the pH is adjusted to 2 with H₂SO₄ (1:5). The solution is filtered through a #1 Whatman filter paper into a 100 ml volumetric flask. Five ml of Ferrous Sulfate solution is added, the volume brought to mark, the flask stoppered, the contents well mixed and allowed to stand 15 minutes. The absorbance of each of the standard solutions is determined against the blank solution at 775 nm. A calibration curve of absorbance vs. ppm is constructed. The absorbance of the samples is then determined and the concentration is read off the calibration curve.

DIVISION OFFICE PHONE NUMBERS FOR SALT DELIVERY NOTIFICATION

DIVISION 1 – Presque Isle	Phone #: (207)764-2060	Contact: Dana Chasse
DIVISION 2 - Ellsworth	Phone #: (207)667-5556	Contact: Randy Gray
DIVISION 3 - Bangor	Phone #: (207)941-4500	Contact: Bill Gormely
DIVISION 4 - Fairfield	Phone #: (207)453-7377	Contact: Norm Richardson
DIVISION 5 - Rockland	Phone #: (207)596-2230	Contact: Doug Carlson
DIVISION 6 - Scarborough	Phone #: (207)885-7000	Contact: Bob Slocum
DIVISION 7 - Dixfield	Phone #: (207)562-4228	Contact: Bob Spencer

FOR BILLING QUESTIONS CONTACT LORI HOWLETT AT (207) 624-3600.