

**PERFORMANCE OF TACONITE  
AGGREGATES IN THIN LIFT HMA  
REPORT NUMBER FHWA-HIF-12-025  
Final Report - January 31, 2012**

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**TECHNICAL REPORT  
NRRI/TR-2012/04  
January 2012**

**Funded by U.S. Department of Transportation/Federal  
Highway Administration**

**Project Nos. 3002-10416-00013020  
1750-10416-20090-00013020-1000004147-CS**

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This publication is accessible from the home page of the Economic Geology Group of the Center for Applied Research and Technology Development at the Natural Resources Research Institute, University of Minnesota Duluth (<http://www.nrri.umn.edu/egg>) as a PDF file readable with Adobe Acrobat 6.0.

*Date of release: January 2012*

*Cover Photo Caption*

Unbound taconite fine aggregate (coarse taconite tailings) and project mix design gyratory specimens.

*Recommended Citation*

Zanko, L.M., Johnson, E., Marasteanu, M., Patelke, M.M., Linell, D., Moon, K.H., Oreskovich, J.A., DeRocher, W., Betts, R., Nadeau, L., Johanneck, L., and Turos, M., 2012, Performance of Taconite Aggregates on Thin Left HMA, Report Number FHWA-HIF-12-025, Final Report – January 31, 2012: Natural Resources Research Institute, University of Minnesota Duluth, Technical Report NRRI/TR-2012/04, 125 p.

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**TECHNICAL DOCUMENTATION PAGE**

|   |  |  |  |  |           |
|---|--|--|--|--|-----------|
| 1. Report No.<br>FHWA-HIF-12-025  |  | 2. Government Accession No.                          |  | 3. Recipient's Catalog No.   |           |
| 4. Title and Subtitle<br>Performance of Taconite Aggregates on Thin Left HMA  |  |  |  | 5. Report Date<br>April 25, 2012   |           |
|   |  |  |  | 6. Performing Organization Code  |           |
| 7. Author(s)<br>Lawrence M. Zanko, Ed Johnson, Mihai Marasteanu, Marsha M. Patelke, Dave Linell, Ki Hoon Moon, Julie A. Oreskovich, Ray Betts, Lynnette Nadeau, Luke Johanneck, Mugur Turos, and Will DeRocher  |  |  |  | 8. Performing Organization Report No.  |           |
| 9. Performing Organization Name and Address<br>Natural Resources Research Institute, University of Minnesota Duluth<br>5013 Miller Trunk Hwy<br>Duluth MN 55811   |  |  |  | 10. Work Unit No. (TRAIS)  |           |
|   |  |  |  | 11. Contract or Grant No.<br>DTFH61-09-H-00017   |           |
| 12. Sponsoring Agency Name and Address<br>Federal Highway Administration<br>Office of Pavement Technology, HIPT-10<br>1200 New Jersey Avenue, SE<br>Washington, DC 20590  |  |  |  | 13. Type of Report and Period Covered<br>Final Report  |           |
|   |  |  |  | 14. Sponsoring Agency Code   |           |
| 15. Supplementary Notes<br>The Contracting Officer's Technical Representative (COTR) was Victor (Lee) Gallivan  |  |  |  |  |           |
| 16. Abstract:<br>This project was undertaken to advance the knowledge of the beneficial uses of taconite mining coarse tailings (taconite fine aggregate) for thin lift hot mix asphalt (HMA), to facilitate technical information gathering and marketing of such uses and properties, and to encourage the beneficial use of recycled/byproduct materials like durable and wear- and skid-resistant taconite (Mesabi) aggregates, recycled asphalt pavement (RAP), and asphalt shingles. In combination, the use of each is highly desirable because it promotes resource conservation, safety, and energy-saving. Outcomes of this study suggest that Mesabi rock and tailings products show promise as components of 4.75-mm Dense-graded, Stone Matrix Asphalt, and Ultra-Thin Bonded Wearing asphalt mixtures. Laboratory and field investigations of taconite tailings should continue. The Mesabi rock can be incorporated in standard Superpave, SMA, and fine/sand asphalt mixtures in upcoming construction projects. In each case construction and long term field performance should be evaluated. The investigators conclude that taconite-based thin lift HMA mixes that also incorporate RAP should be recognized as an environmentally sound, i.e., combining the use of byproduct and recycled/reclaimed materials, and high-quality option for HMA pavement rehabilitation and preservation. Collectively, the material testing results suggest that thinner wear-course pavements made from appropriately designed taconite-based mixes can match or exceed the service life of conventional MnDOT Level 4 mixtures. If extended service life is realized, then taconite fine aggregate could be a cost-effective choice at end-user locations where high-quality local aggregate sources are lacking or absent. These enhanced performance attributes can add intrinsic value to taconite materials and make them more desirable to use and more cost-effective to transport longer distances, thereby improving and broadening their near- and long-term potential for regional and national highway infrastructure projects. |  |  |  |  |           |
| 17. Key Words<br>Taconite, Mesabi, Granite, Tailings, Recycle, Leachate, Leaching, Chemistry, RAP, Mesabi Select Aggregate, Mineral Filler, Stone Matrix Asphalt Mixture, SMA, Superpave Asphalt Mixture, Ultra Thin Bonded Wearing Course, Aggregate, MnROAD.  |  |  |  | 18. Distribution Statement<br>No restrictions. This document is available to the public through NTIS:<br>National Technical Information Service<br>5301 Shawnee Road<br>Alexandria, VA 22312 |           |
| 19. Security Classif. (of this report)<br>Unclassified  |  | 20. Security Classif. (of this page)<br>Unclassified |  | 21. No. of Pages   | 22. Price |

# SI\* (MODERN METRIC) CONVERSION FACTORS

## APPROXIMATE CONVERSIONS TO SI UNITS

| Symbol   | When You Know              | Multiply By                 | To Find                     | Symbol            |
|--|----------------------------|-----------------------------|-----------------------------|-------------------|
| <b>LENGTH</b>  |                            |                             |                             |                   |
| in   | inches                     | 25.4                        | millimeters                 | mm                |
| ft   | feet                       | 0.305                       | meters                      | m                 |
| yd   | yards                      | 0.914                       | meters                      | m                 |
| mi   | miles                      | 1.61                        | kilometers                  | km                |
| <b>AREA</b>  |                            |                             |                             |                   |
| in <sup>2</sup>  | square inches              | 645.2                       | square millimeters          | mm <sup>2</sup>   |
| ft <sup>2</sup>  | square feet                | 0.093                       | square meters               | m <sup>2</sup>    |
| yd <sup>2</sup>  | square yard                | 0.836                       | square meters               | m <sup>2</sup>    |
| ac   | acres                      | 0.405                       | hectares                    | ha                |
| mi <sup>2</sup>  | square miles               | 2.59                        | square kilometers           | km <sup>2</sup>   |
| <b>VOLUME</b>  |                            |                             |                             |                   |
| fl oz  | fluid ounces               | 29.57                       | milliliters                 | mL                |
| gal  | gallons                    | 3.785                       | liters                      | L                 |
| ft <sup>3</sup>  | cubic feet                 | 0.028                       | cubic meters                | m <sup>3</sup>    |
| yd <sup>3</sup>  | cubic yards                | 0.765                       | cubic meters                | m <sup>3</sup>    |
| NOTE: volumes greater than 1000 L shall be shown in m <sup>3</sup> |                            |                             |                             |                   |
| <b>MASS</b>  |                            |                             |                             |                   |
| oz   | ounces                     | 28.35                       | grams                       | g                 |
| lb   | pounds                     | 0.454                       | kilograms                   | kg                |
| T  | short tons (2000 lb)       | 0.907                       | megagrams (or "metric ton") | Mg (or "t")       |
| <b>TEMPERATURE (exact degrees)</b>                                 |                            |                             |                             |                   |
| °F   | Fahrenheit                 | 5 (F-32)/9<br>or (F-32)/1.8 | Celsius                     | °C                |
| <b>ILLUMINATION</b>  |                            |                             |                             |                   |
| fc   | foot-candles               | 10.76                       | lux                         | lx                |
| fl   | foot-Lamberts              | 3.426                       | candela/m <sup>2</sup>      | cd/m <sup>2</sup> |
| <b>FORCE and PRESSURE or STRESS</b>                                |                            |                             |                             |                   |
| lbf  | poundforce                 | 4.45                        | newtons                     | N                 |
| lbf/in <sup>2</sup>  | poundforce per square inch | 6.89                        | kilopascals                 | kPa               |

## APPROXIMATE CONVERSIONS FROM SI UNITS

| Symbol                              | When You Know               | Multiply By | To Find                    | Symbol              |
|-------------------------------------|-----------------------------|-------------|----------------------------|---------------------|
| <b>LENGTH</b>                       |                             |             |                            |                     |
| mm                                  | millimeters                 | 0.039       | inches                     | in                  |
| m                                   | meters                      | 3.28        | feet                       | ft                  |
| m                                   | meters                      | 1.09        | yards                      | yd                  |
| km                                  | kilometers                  | 0.621       | miles                      | mi                  |
| <b>AREA</b>                         |                             |             |                            |                     |
| mm <sup>2</sup>                     | square millimeters          | 0.0016      | square inches              | in <sup>2</sup>     |
| m <sup>2</sup>                      | square meters               | 10.764      | square feet                | ft <sup>2</sup>     |
| m <sup>2</sup>                      | square meters               | 1.195       | square yards               | yd <sup>2</sup>     |
| ha                                  | hectares                    | 2.47        | acres                      | ac                  |
| km <sup>2</sup>                     | square kilometers           | 0.386       | square miles               | mi <sup>2</sup>     |
| <b>VOLUME</b>                       |                             |             |                            |                     |
| mL                                  | milliliters                 | 0.034       | fluid ounces               | fl oz               |
| L                                   | liters                      | 0.264       | gallons                    | gal                 |
| m <sup>3</sup>                      | cubic meters                | 35.314      | cubic feet                 | ft <sup>3</sup>     |
| m <sup>3</sup>                      | cubic meters                | 1.307       | cubic yards                | yd <sup>3</sup>     |
| <b>MASS</b>                         |                             |             |                            |                     |
| g                                   | grams                       | 0.035       | ounces                     | oz                  |
| kg                                  | kilograms                   | 2.202       | pounds                     | lb                  |
| Mg (or "t")                         | megagrams (or "metric ton") | 1.103       | short tons (2000 lb)       | T                   |
| <b>TEMPERATURE (exact degrees)</b>  |                             |             |                            |                     |
| °C                                  | Celsius                     | 1.8C+32     | Fahrenheit                 | °F                  |
| <b>ILLUMINATION</b>                 |                             |             |                            |                     |
| lx                                  | lux                         | 0.0929      | foot-candles               | fc                  |
| cd/m <sup>2</sup>                   | candela/m <sup>2</sup>      | 0.2919      | foot-Lamberts              | fl                  |
| <b>FORCE and PRESSURE or STRESS</b> |                             |             |                            |                     |
| N                                   | newtons                     | 0.225       | poundforce                 | lbf                 |
| kPa                                 | kilopascals                 | 0.145       | poundforce per square inch | lbf/in <sup>2</sup> |

\*SI is the symbol for the International System of Units. Appropriate rounding should be made to comply with Section 4 of ASTM E380.  
(Revised March 2003)

## EXECUTIVE SUMMARY

This project was undertaken to advance the knowledge of the beneficial uses of taconite mining coarse tailings (taconite fine aggregate) for thin lift hot mix asphalt (HMA), to facilitate technical information gathering and marketing of such uses and properties, and to encourage the beneficial use of recycled/byproduct materials like durable and wear- and skid-resistant taconite aggregates, recycled asphalt pavement (RAP), and asphalt shingles. In combination, the use of each is highly desirable because it promotes resource conservation, safety, and energy-saving.

Project findings are presented for the following five Study Areas.

- Study Area 1 – Taconite Tailings Literature Review
- Study Area 2 – Thin Lift HMA Literature Review
- Study Area 3 – Leaching Potential of Taconite Tailings
- Study Area 4 – Mix Design Testing for Thin Layer Asphalt Made with Taconite Tailings
- Study Area 5 – Develop and Evaluate Mix Design for Thin Layer Asphalt Made with Taconite Tailings

The project's three lead organizations were given primary responsibility for completing work associated with each Study Area as follows:

- Study Areas 1 through 3 – University of Minnesota Duluth, Natural Resources Research Institute (NRRI);
- Study Area 4 – University of Minnesota Civil Engineering Department (UM-CE) and Minnesota Department of Transportation (MnDOT); and
- Study Area 5 – MnDOT, in collaboration with UM-CE.

Key project findings include the following:

- Laboratory experimental testing using standard testing procedures available to all material testing laboratories on the same fine graded mixtures showed that taconite mixes resulted in:
  - Excellent rutting capabilities as shown by experimental results obtained on APA asphalt mixture specimens at MnDOT materials laboratory;
  - Enhanced creep stiffness as measured by indirect tensile test (IDT) in mixes with and without RAP, with higher stiffness values at all three test temperatures relative to granite mixes; IDT strength comparable to granite mixes; and low temperature fracture toughness – as determined by semi-circular bend (SCB) tests – comparable to or slightly better than granite-containing mixes, as obtained on asphalt mixture specimens at the University of Minnesota pavement laboratory;
  - One order of magnitude increase in fatigue life measured in fatigue tests performed on asphalt mixture beams at Iowa State University.
- Overall, laboratory testing has shown that taconite-based thin lift mix designs performed as well or better than the granite-based (reference) mix;

- Taconite-based mixes used less asphalt binder than anticipated, 5.8% to 7.5%;
- Mix designs containing 20% recycled asphalt pavement (RAP) performed satisfactorily; and
- Leachate derived from the taconite fine aggregate used in this project, in both an as-is (unbound) state and in an asphalt-bound state, meets EPA standards for all Resource Conservation and Recovery Act (RCRA) metals, with the exception of selenium (Se) for tailings used in an unbound state. This single result – 7.46 µg/L (ppb) – is 2.46 µg/L (ppb) above the EPA recommended water quality standard of 5.0 µg/L and occurred in a sample where the liquid-solid (L/S) ratio test condition was 0.5:1.0:
  - In comparison, leachate derived from limestone, gravel, and dirty sand samples exceeded water quality standards for more than one metal and for more than one liquid-to-solid (L/S) ratio test condition, suggesting that these aggregates – when used in an unbound state – are more likely to release metals in a larger variety of environmental conditions than would taconite tailings.

Outcomes of this study suggest that Mesabi rock and tailings products show promise as components of 4.75-mm Dense-graded, Stone Matrix Asphalt, and Ultra-Thin Bonded Wearing asphalt mixtures. Laboratory and field investigations of taconite tailings should continue. The Mesabi rock can be incorporated in standard Superpave, SMA, and fine/sand asphalt mixtures in upcoming construction projects. In each case construction and long term field performance should be evaluated.

The investigators conclude that taconite-based thin lift HMA mixes that also incorporate RAP should be recognized as an environmentally sound, i.e., combining the use of byproduct and recycled/reclaimed materials, and high-quality option for HMA pavement rehabilitation and preservation. Collectively, the material testing results suggest that thinner wear-course pavements made from appropriately designed taconite-based mixes can match or exceed the service life of conventional MnDOT Level 4 mixtures. If extended service life is realized, then taconite fine aggregate could be a cost-effective choice at end-user locations where high-quality local aggregate sources are lacking or absent. These enhanced performance attributes can add intrinsic value to taconite materials and make them more desirable to use and more cost-effective to transport longer distances, thereby improving and broadening their near- and long-term potential for regional and national highway infrastructure projects.

## **ACKNOWLEDGEMENTS**

The Federal Highway Administration (FHWA) is gratefully acknowledged for providing project funding. Special thanks also go to NRRI chemist Dr. Igor Kolomitsyn for his project assistance and laboratory guidance with respect to leachate generation, NRRI scientist/geologist John Heine for conducting X-ray diffraction (XRD) analysis of project samples and for providing X-ray fluorescence (XRF) assistance, and to UMD science and engineering students Katherine Ballandby, Paul Kimpling, and Will DeRocher for their project contributions in the laboratory. Appreciation is also expressed for state, county, and local transportation professionals and engineers (current and retired), and to private sector contractors, for their cooperation and project input. Lastly, in light of unforeseen events over the course of the project, including

complications and delays related to the 2011 Minnesota state government shutdown, the three lead organizations also express their gratitude to FHWA for providing extended time for the completion of this project.

## BACKGROUND

Taconite is a hard, dense iron-bearing sedimentary rock, composed of alternating chert and slate units of varying thickness, that contains an intimate mixture of quartz and magnetite ( $Fe_3O_4$ ), plus varying amounts of iron oxides, carbonates, and silicates (Davis, 1964). Taconite is also a term used for describing the type of iron ore mined on the Mesabi Iron Range of northeastern Minnesota from which the iron, in the form of magnetite, can be profitably extracted after crushing and fine-grinding, followed by magnetic separation and pelletizing (Morey and Southwick, 1993). Geologically, this magnetite-bearing taconite ore is associated with the 1.8 to 2 billion year-old Biwabik Iron Formation.

Presently, six taconite mines are operating on Minnesota's Mesabi Iron Range. Their locations are shown in Figure 1, with the diagonal and sigmoidal-shaped Biwabik Iron Formation depicted in red. The six active operations, from southwest to northeast, are: 1) U.S. Steel Keewatin Taconite (Keetac); 2) Hibbing Taconite Company (Hibtac); 3) U.S. Steel Minntac (Minntac); 4) ArcelorMittal Minorca Mine (Minorca); 5) United Taconite LLC (UTAC); and 6) Northshore Mining Company (Northshore). This study focused on taconite aggregate materials sourced from Minntac and Minorca.

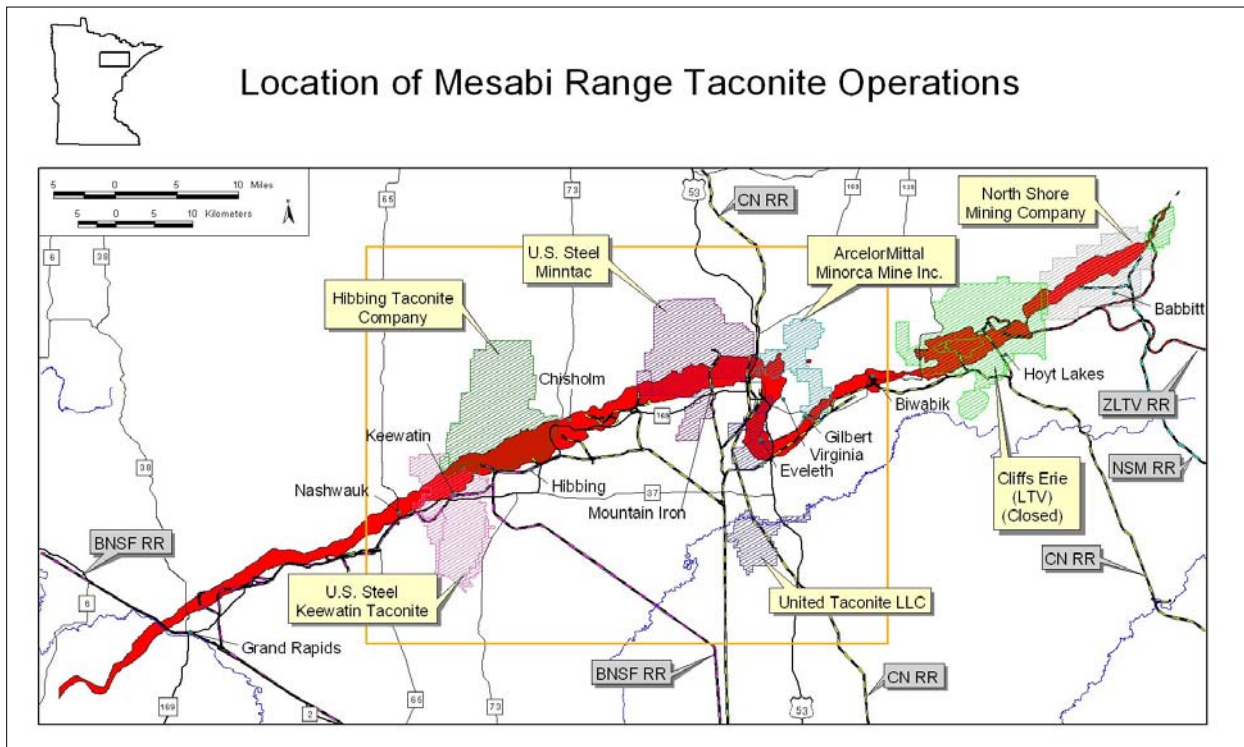


Figure 1. Location map of Minnesota taconite mining operations.

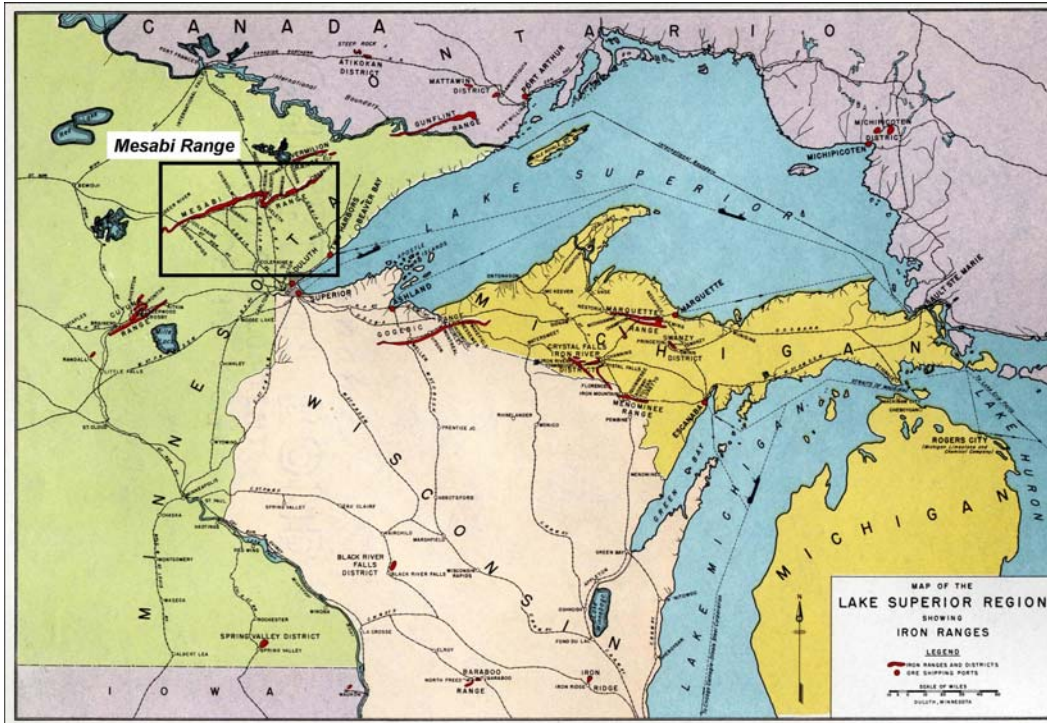


## A Note on Terminology

The term “taconite” is a geologically, mineralogically, and economically imprecise descriptor and prefix for the types of mining byproduct material (i.e., taconite aggregates) on which this FHWA project is focused, because taconite can also refer to the ore that is mined *and* the iron-rich pellets that are produced following the processing of that ore. In other words, “taconite” can mean geological or aggregate material (rock) to one person and a salable, processed product used in steel making (pellets) to someone else.

Taconite is a term that has, for better or worse, become embedded in the vernacular of the region and anything related to the type of iron ore mining and processing practiced in the state of Minnesota during the past half-century. For example, Dr. E.W. Davis (who developed the taconite process) describes taconite as follows in his 1964 book, "Pioneering with Taconite": *"Taconite is the name given by geologists to a type of hard rock containing fine particles of iron ore. The word was first used by Newton H. Winchell, a Minnesota geologist, who in 1892 applied it to the magnetic iron formation on the state's eastern Mesabi Range."* Thus, it is a term that has persisted.

Geologically speaking, these materials are, by definition, “iron-formation” rock. However, “iron-formation” is still too broad and generic a term because it does not describe *where* the aggregate materials originate. To avoid future terminology problems and confusion, it is suggested that the term or prefix “Mesabi” be applied to aggregate materials sourced from Minnesota’s taconite mining operations. This “Mesabi” naming convention is logical because it is based on the specific district from which taconite ore has been mined historically: Minnesota’s Mesabi Range. Similar historic iron mining districts (iron ranges) exist in the Lake Superior region (Fig. 2) such as the Cuyuna and Vermilion Ranges in Minnesota, the Gogebic Range in Wisconsin, and the Marquette Range in Michigan.



**Figure 2. Mesabi Range (boxed area) relative to historic Lake Superior region iron ranges. Map source: Oliver Iron Mining Division, United States Steel Corporation (Goldich and Marsden, 1956).**

Still, the current project is titled, “Performance of *Taconite* Aggregates in Thin Lift HMA.” Consequently, this report continues the “tradition” of using the term “taconite” throughout. In the future, these materials may simply be referred to as “Mesabi” aggregate, “Mesabi iron-formation” aggregate, “Mesabi Rock,” or some variation thereof.

### **Byproduct Types and Potential**

Tens of millions of tons of byproduct (non-ore) taconite rock are generated annually by Minnesota’s taconite producers, in the form of three aggregate-applicable byproducts:

1. **Taconite Blast Rock:** low-grade taconite rock that must be drilled, blasted, and removed to gain access to the underlying taconite ore; it is typically much larger than 6 inches (15cm) in size, and can be used as rip-rap, armor stone, etc.;
2. **Coarse Crushed Taconite Rock:** blast rock that undergoes further crushing and screening to meet a particular size specification, e.g., -2½ in, -¾ in, -½ in (-6.4cm, -1.9cm, -1.3cm, respectively), i.e., coarse aggregate (CA); and
3. **Coarse Taconite Tailings (taconite fine aggregate):** non-magnetic byproduct of taconite ore processing; essentially a manufactured sand, with a typical gradation equivalent to fine aggregate (FA), i.e., 100% finer than 3/8 in (10mm) but containing a low percentage of -200 mesh (0.075mm) particles.

**NOTE:** These byproduct taconite rock size distinctions are important to understand because the term “taconite tailings” is often used improperly when referred to as an aggregate material. Therefore, the following description is presented to add clarity:

- Within the context of paving applications such as HMA, coarse taconite tailings represent the *fine aggregate* (FA) component of an overall mix design; whereas coarse crushed taconite rock represents the coarse aggregate (CA) component. Relative to the thin lift emphasis of this investigation, coarse taconite tailings can also represent the *sole or dominant* aggregate component of the mix. That is why the term “taconite fine aggregate” is a more technically accurate descriptor for coarse taconite tailings, i.e., coarse taconite tailings = fine aggregate.

### **Why Taconite Tailings and Thin Lift?**

Taconite aggregate products are inherently hard and durable, given the high compressive strength of the iron-formation from which they are derived. As reported in Zanko et al. (2009):

*“...the compressive strength of typical Mesabi Range iron-formation (taconite) rock ranges from 28,000 to 90,000 psi (193 to 621MPa) (Plummer, 1976; Stump and Hetzer, 1999). In comparison, the compressive strength of limestone ranges from 8,000 to 26,000 psi, while crystalline igneous rocks like granite and basalt (aka trap rock) are in the 20,000 psi to 50,000 psi range, respectively (Call and Savely, 1990).”*

Hard, durable aggregates enhance the skid resistance of the pavement, and taconite byproduct rock (aggregate) materials have long been recognized for their superior friction characteristics and wear-resistant properties. A 1976 FHWA report stated the following:

*“The serviceability of these taconite overlays has been exceptional. It has been found that the use of **coarse taconite tailings** definitely improves the skid resistance of pavements in which it is used. In the future, taconite tailings may be specified as the sole material used for surface overlays because of their skid resistance qualities.”*

More recently, a review concerning safety by the Asphalt Concrete Pavement Association cited Snyder (2006), and stated:

*“Pavement texture plays an important role in roadway safety issues. There are more than one million deaths and 50 million injuries annually on highways and roads all over the world, with more than 40,000 deaths and 3 million injuries annually in the U.S. alone. Research indicates that about 14 percent of all crashes occur in wet weather, and that 70 percent of these crashes are preventable with improved pavement texture/friction.”*

Developing thinner (yet stronger and longer-lasting) and more skid-resistant asphalt wear courses – particularly those that utilize taconite fine aggregate – positively affect both pavement construction time, safety and cost, and represent a more environmentally responsible and efficient use of natural resources. The benefits of using the proposed technology start with

producing smaller quantities of mixtures compared to the current thicker overlays and continue to accumulate in time by extending the longevity and the performance level of pavements in general, which in turn translates into less frequent rehabilitation of highway infrastructure and reduced user delays and congestion. Further cost and environmental benefits are similarly realized via reductions in raw materials needed (asphalt binder and aggregate), by improvements in safety imparted by the more skid-resistant taconite materials, and by replacing rougher and compromised pavements with a more durable and essentially smoother driving surface. These enhancements are tangible and quantifiable. For example, a recent MIT study (Santero et al., 2011), suggests that a rehabilitated pavement should improve fuel efficiency by about 5% for automobiles and 3.5% for trucks due to decreased rolling resistance. The 2011 MIT study also determined that excess vehicle fuel consumption related to pavement roughness is a major CO<sub>2</sub> contributor, in most cases second only to cement production.

### **Project Approach and Report Layout**

The objective of this cooperative project is to advance the knowledge of the beneficial uses of taconite mining coarse tailings (taconite fine aggregate) for thin lift hot mix asphalt (HMA), as well as to facilitate technical information gathering and marketing of such uses and properties.

To meet this objective, the project is subdivided into five (5) major Study Areas, with primary duties delegated as follows: Study Areas 1 through 3 – University of Minnesota Duluth, Natural Resources Research Institute (NRRI); Study Area 4 – University of Minnesota Civil Engineering Department, (UM-CE) and Minnesota Department of Transportation (MnDOT); and Study Area 5 – MnDOT, in collaboration with UM-CE.

- **Study Area 1:** Perform a literature review of the existing completed research on taconite's engineering characterization or properties, as well as any uses in highway test sections;
- **Study Area 2:** Perform a literature review on the development, construction and performance of "thin layers" of asphalt materials in pavement applications;
- **Study Area 3:** Perform experimental work to determine the physical and chemical characteristics of aggregates including leaching potential of asphalt mixtures made with taconite aggregates;
- **Study Area 4:** Mix design testing for thin layer of asphalt mixture made with taconite aggregates, and evaluation of laboratory mechanical properties; and
- **Study Area 5:** Develop and evaluate mix designs for thin layer asphalt made with taconite tailings.

The project's three lead organizations were given primary responsibility for completing work associated with each Study Area. As such, this document reflects this division of labor as follows:

- Chapter 1: Study Areas 1 through 3 – University of Minnesota Duluth, Natural Resources Research Institute (NRRI);

- Chapter 2: Study Area 4 – University of Minnesota Civil Engineering Department, (UM-CE); and
- Chapter 3: Study Area 5 – Minnesota Department of Transportation (MnDOT).

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## LIST OF ACRONYMS AND SYMBOLS

|         |  |
|---------|--|
| AASHTO  | American Association of State Highway and Transportation Officials |
| AFT     | Asphalt Film Thickness   |
| APA     | Asphalt Pavement Analyzer  |
| CA      | Coarse Aggregate   |
| CS      | Chronic Standard   |
| DSR     | Dynamic Shear Rheometer  |
| $ E^* $ | Dynamic Modulus  |
| EPA     | Environmental Protection Agency                                    |
| ESAL    | Equivalent Single Axle Load  |
| FA      | Fine Aggregate   |
| FHWA    | Federal Highway Administration                                     |
| $G^*$   | Shear Modulus  |
| GLI     | Great Lakes Initiative   |
| HMA     | Hot Mix Asphalt  |
| HRL     | Health Risk Limit  |
| IDT     | Indirect Tensile Test  |
| L/S     | Liquid-to-Solid  |
| LVR     | Low Volume Road  |
| MCL     | Maximum Contaminant Level  |
| MnDOT   | Minnesota Department of Transportation                             |
| MPCA    | Minnesota Pollution Control Agency                                 |
| MS      | Maximum Standard   |
| NCAT    | National Center for Asphalt Technology                             |
| NHI     | National Highway Institute   |
| NMAS    | Nominal Maximum Aggregate Size                                     |
| NPDS    | National Primary Drinking Standard                                 |
| NPDWS   | National Primary Drinking Water Standard                           |
| NRRI    | Natural Resources Research Institute                               |
| NRWQ    | National Recommended Water Quality                                 |
| PG      | Performance Grade  |
| RAP     | Reclaimed Asphalt Pavement   |
| RAS     | Recycled Asphalt Shingles  |
| RCRA    | Resource Conservation and Recovery Act                             |
| RWQ-C   | Recommended Water Quality – Chronic Standard                       |
| SCB     | Semi-Circular Bend   |
| SMA     | Stone Matrix Asphalt   |
| SPLP    | Synthetic Precipitation Leaching Procedure                         |
| TCLP    | Toxicity Characteristics Leaching Procedure                        |
| TSR     | Tensile Strength Ratio   |
| UM-CE   | University of Minnesota Department of Civil Engineering            |
| UMD     | University Of Minnesota Duluth                                     |
| VCA     | Voids in Coarse Aggregate  |
| VFA     | Voids Filled with Asphalt  |
| VMA     | Voids in the Mineral Aggregate                                     |
| XRD     | X-Ray Diffraction/Diffractometry                                   |
| XRF     | X-Ray Fluorescence   |

## CHAPTER 1: NRRI

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### STUDY AREA 1 – TACONITE TAILINGS LITERATURE REVIEW

#### Synopsis

##### *Research objectives*

A review of existing literature regarding research on taconite tailings engineering properties and uses in highway test sections was completed. The results of this review provide a collection of taconite tailings usages and published engineering testing results.

##### *Scope of Work*

A literature review was completed to understand the engineering properties of taconite tailings. Results from the literature review are then applied to the existing data for aggregate characteristics to evaluate their use as an aggregate source for thin lift Hot Mix Asphalt (HMA) types of pavements.

##### *Summary of Results*

There is a large amount of available data regarding taconite tailings physical characteristics and historical data regarding its use as the aggregate in asphalt pavements in Minnesota. Several field demonstration projects have been undertaken at the Minnesota Department of Transportation (MnDOT) MnROAD facility. MnDOT studies reported placement of HMA was typical when compared with other aggregates. Initial results have been favorable, with friction being characterized as “exceptional” and ride, rutting, and cracking “satisfactory.”

#### Literature Review

A number of research efforts conducted either individually (or in cooperation) by the project’s three lead organizations provide reasonable supporting specifications, guidelines, and/or procedures to support successful national deployment. The results have been published in peer-reviewed journals and in organizational technical reports and have been presented at national conferences. Examples follow.

Two NRRI publications provide the largest amount of published data on the use of taconite tailings as aggregate. The first, *Properties and Aggregate Potential of Coarse Taconite Tailings from Five Minnesota Taconite Operations* (Zanko et al., 2003), also includes the results of physical, geological, chemical, mineralogical, and microscopic testing performed on 18 bulk samples of coarse taconite tailings (taconite fine aggregate) collected over a one-year period between 2000 and 2001. The second NRRI publication, *Documenting the Historical Use of Taconite Byproducts as Construction Aggregates in Minnesota—A GIS-based Compilation of Applications, Locations, Test Data, and Related Construction Information* (Oreskovich et al., 2007), covers a time period from the 1950s to 2006 and includes a database assembled from a

compilation of locations, applications, test data, and related construction information. Over 100 entities, including state, county, and municipal agencies, contractors, testing laboratories, and engineering firms were contacted to obtain the data.

Study Area 1 of the current project builds upon the database included in the 2007 report. Four of the major entities involved with the use of taconite aggregates in bituminous pavements – Minnesota Department of Transportation (MnDOT), St. Louis County Department of Public Works, Northland Bituminous (Duluth), and Ulland Brothers, Inc. (Mesabi Range) – were contacted to update the database. Northland Bituminous and Ulland Brothers are two of the principal paving contractors in the Duluth area and on the Mesabi Range. Each provided updates on taconite products used, applications, locations, and test or mix data. More detail regarding Study Area 1 contacts, contact responses, and references to data and data files compiled as a result of this data acquisition process is presented in Appendix A.

A third NRRI publication, *Final Compendium Report to the Economic Development Administration – Research, Development, and Marketing of Minnesota’s Iron Range Aggregate Materials for Midwest and National Transportation Applications* (Zanko et al., 2010), presents the results of a 4-year taconite aggregate research and demonstration program covering the entire spectrum of byproduct taconite rock materials. The 1,295-page compendium report includes several external reports, including contributions from this project’s partners (MnDOT and UM-CE), and an extensive reference list. The full report is available for download at: <http://www.nrri.umn.edu/egg/REPORTS/TSR201001/TSR201001.html>

In addition to the NRRI reports just cited, MnDOT reports (Zerfas et al., 2005; Olson et al., 2006; Johnson et al., 2009; and Clyne et al., 2010) were specifically reviewed regarding use of the tailings as aggregate for paving.

#### *Usage, Source, and Data Examples*

Taconite aggregates, particularly the coarse taconite tailings, have become a mainstay of nearly every asphalt mix design developed by Northland Bituminous and Ulland Brothers for nearly a decade. Of particular note is a reclaim and resurface job done on Haines road in the city of Duluth in 2003 that used 80% taconite with 20% RAP. St. Louis County put down a 1” skim coat that was to buy an additional five years of time for this high traffic volume road. The surface went approximately five years without developing a crack and continues to hold up well despite problems with the foundation (D. Gustafson, Northland Bituminous; W. Wilmot, St. Louis County, pers. comm., 2010). The skim coat consists of 15% -3/4” rock CA (taconite), 5% -1/2” rock CA (taconite), 40% -1/2” fine FA (taconite), 20% coarse tailings (taconite), and 20% RAP. All of the taconite products came from the United States Steel (USS) Minntac operation in Mountain Iron, MN. The road is now scheduled for a rebuild in 2012 or 2013, nearly doubling the expected life of the temporary fix.

The ArcelorMittal Minorca Mine (also referred to simply as Minorca) has been the source of Northland Bituminous’ coarse taconite tailings for the past four years, as well as a primary source for Ulland Brothers, which operates a crushing operation on ArcelorMittal property (as well as on Minntac property). Northland Bituminous’ coarse tailings deliveries are the result of a back haul on coal delivered to ArcelorMittal from the Port of Superior.

Physical properties of ArcelorMittal coarse taconite tailings obtained from MnDOT, Northland Bituminous, and Ulland Brothers are presented in Table 1. Similar data from these and other laboratories has been compiled and tabled for each of the remaining westernmost taconite mines on the Mesabi Range: Hibbing Taconite (Hibtac), Keewatin Taconite (Keetac), Minntac, and United Taconite (UTAC) (refer to Fig. 1). ArcelorMittal data are shown here, as they contain the greatest number of laboratory test results other than gradations.

**Table 1. Physical properties of ArcelorMittal (Minorca) coarse taconite tailings.**

| ArcelorMittal MINORCA MINE COARSE TAILINGS                   |      |      |      |      |      |      |      |      |      |      |      |
|--|------|------|------|------|------|------|------|------|------|------|------|
| SMP  | 15   | 16   | 18   | 19   | 20   | 21   | 32   | 33   | 34   | 35   | 36   |
| Year   | 2006 | 2006 | 2007 | 2007 | 2008 | 2008 | 2009 | 2009 | 2009 | 2009 | 2010 |
| Lab  | M    | U    | M    | N    | M    | N    | M    | M    | U    | N    | N    |
| GRADATIONS (% Passing)                                       |      |      |      |      |      |      |      |      |      |      |      |
| 3/8"   | 100  | 100  |      | 100  | 100  | 100  | 100  | 100  | 100  | 100  | 100  |
| NO. 4  | 99   | 99   | 100  | 100  | 98   | 97.8 | 98   | 98   | 99   | 97.5 | 97.7 |
| NO. 8  | 87   | 90   | 90   | 89.7 | 89   | 85.2 | 89   | 90   | 90   | 84.5 | 87.8 |
| NO. 10   |      |      |      |      |      |      |      |      |      |      |      |
| NO. 16   | 62   | 62   | 84   | 84.5 | 68   | 61   | 68   | 70   | 69   | 60.6 | 70.4 |
| NO. 20   |      |      |      |      |      |      |      |      |      |      |      |
| NO. 30   | 38   | 38   | 52   | 51.5 | 46   | 39.2 | 46   | 47   | 46   | 39.1 | 45.3 |
| NO. 40   |      |      |      |      |      |      |      |      |      |      |      |
| NO. 50   | 18   | 18   | 28   | 27.8 | 26   | 21.4 | 26   | 24   | 25   | 21   | 26.2 |
| NO. 80   |      |      |      |      |      |      |      |      |      |      |      |
| NO. 100  | 9    | 9    | 6    | 6.5  | 9    | 8.6  | 9    | 9    | 9    | 8.5  | 7.8  |
| NO. 200  | 2.4  | 4.4  | 3.2  | 3.2  | 3.7  | 4.3  | 3.7  | 4.4  | 4.6  | 4.3  | 4    |
| LABORATORY TEST RESULTS                                      |      |      |      |      |      |      |      |      |      |      |      |
| % Absorp (-4)  | 0.8  |      | 0.9  |      | 0.9  |      | 0.9  | 1    |      |      |      |
| Bulk SpG (-4)  | 2.89 | 2.9  | 2.9  |      | 2.93 |      | 2.91 | 2.89 | 2.96 |      |      |
| Bulk SpG (BA +4)   |      | 2.9  |      |      |      |      |      |      | 2.96 |      |      |
| Total % Bulk SpG   |      | 2.9  |      | 2.92 |      | 2.93 |      |      | 2.96 | 2.95 | 2.95 |
| Avg % FAA  |      |      |      |      |      |      |      |      | 46   |      |      |
| Laboratories: M=MnDOT, N=Northland Bituminous, U=Ulland Bros |      |      |      |      |      |      |      |      |      |      |      |

Coarse tailings from any given taconite operation are very consistent over time. One gradation per year (per mine) is submitted to MnDOT by the paving contractor. While MnDOT accepts the contractor's gradation, both MnDOT and the contractor run specific gravity on the sample. Results from the two labs must be within 0.030 for acceptance (D. Gustafson, Northland Bituminous, pers. comm., 2010). Due to the quality (i.e., durability and hardness) of the taconite aggregates and familiarity with the product, the MnDOT District 1 laboratory no longer runs Los Angeles Rattler/Abrasion (LAR) and Magnesium Sulfate tests on the material, as taconite aggregates have consistently exceeded the minimum specification. The aggregate cannot absorb enough moisture to be affected by magnesium sulfate (R. Garver, Materials Engineer, pers. comm., 2010).

Gradations and physical properties of various coarser taconite aggregate (rock) fractions from ArcelorMittal are presented in Table 2. Unlike coarse taconite tailings, which are a market-ready fine aggregate byproduct of taconite ore processing, these coarser taconite aggregate products must be crushed to spec by the contractor.



**Table 2. Gradations and physical properties of various coarser taconite aggregate fractions from ArcelorMittal (Minorca).**

| ArcelorMittal BITUMINOUS TACONITE AGGREGATE   |           |           |           |           |           |            |                    |            |                    |            |            |            |            |            |            |            |               |
|---|-----------|-----------|-----------|-----------|-----------|------------|--------------------|------------|--------------------|------------|------------|------------|------------|------------|------------|------------|---------------|
| SMP   | 1         | 2         | 3         | 4         | 5         | 6          | 7                  | 8          | 9                  | 10         | 11         | 12         | 13         | 14         | 15         | 16         |               |
| Year  | 2006      | 2006      | 2006      | 2008      | 2008      | 2008       | 2008               | 2009       | 2009               | 2006       | 2006       | 2008       | 2008       | 2008       | 2009       | 2008       |               |
| Field ID  | 3/4" ROCK | 3/4" ROCK | 3/4" ROCK | 3/4" ROCK | 3/4" ROCK | 3/4" MINUS | 3/4" MINUS         | 3/4" MINUS | 3/4" MINUS         | 1/2" MINUS | 1/2" MINUS | 1/2" MINUS | 1/2" MINUS | 1/2" MINUS | 1/2" MINUS | 1/2" MINUS | CRUSHER FINES |
| Use   | BA        | BA        | BA        | BA        | BA        | BA         | BA                 | SP NW      | SP NW              | SP BA      | SP BA      | BA         | BA         | BA         | BA         | BA         |               |
| Lab   | ULLAND    | MNDOT     | MNDOT     | ULLAND    | MNDOT     | ULLAND     | MNDOT <sup>1</sup> | ULLAND     | MNDOT <sup>1</sup> | ULLAND     | MNDOT      | ULLAND     | ULLAND     | MNDOT      | ULLAND     | MNDOT      |               |
| GRADATIONS (% Passing)  |           |           |           |           |           |            |                    |            |                    |            |            |            |            |            |            |            |               |
| 1.00"   | 100       | 100       |           | 100       |           | 100        |                    | 100        |                    | 100        |            | 100        | 100        |            | 100        |            |               |
| 3/4"  | 100       | 91        | 100       | 100       | 100       | 100        | 100                | 100        | 100                | 100        | 100        | 100        | 100        | 100        | 100        |            |               |
| 5/8"  |           | 71        |           |           |           |            |                    |            |                    |            |            |            |            |            |            |            |               |
| 1/2"  | 41        | 44        | 41        | 45        | 45        | 82         | 82                 | 81         | 81                 | 100        | 100        | 100        | 99         | 100        | 100        | 100        | 100           |
| 3/8"  | 13        | 22        | 11        | 15        | 11        | 68         | 68                 | 65         | 65                 | 96         | 96         | 97         | 94         | 94         | 94         | 94         | 97            |
| NO. 4   | 1         | 1         | 1         | 1         | 2         | 31         | 31                 | 37         | 37                 | 75         | 64         | 68         | 60         | 59         | 59         | 61         | 61            |
| NO. 8   | 1         |           | 1         | 1         | 2         | 26         | 26                 | 26         | 26                 | 48         | 35         | 46         | 43         | 40         | 42         | 34         | 34            |
| NO. 10  |           |           |           |           |           |            |                    |            |                    |            |            |            |            |            |            |            |               |
| NO. 16  | 1         |           | 1         | 1         | 1         | 17         | 17                 | 16         | 16                 | 25         | 22         | 31         | 27         | 29         | 26         | 21         | 21            |
| NO. 30  | 1         |           | 1         | 1         | 1         | 11         | 11                 | 11         | 11                 | 16         | 14         | 22         | 18         | 19         | 17         | 14         | 14            |
| NO. 40  |           |           |           |           |           |            |                    |            |                    |            |            |            |            |            |            |            |               |
| NO. 50  | 1         |           | 1         | 1         | 1         | 7          | 7                  | 7          | 7                  | 14         | 14         | 15         | 11         | 13         | 10         | 11         | 11            |
| NO. 100   | 1         |           | 1         | 1         | 1         | 5          | 5                  | 4          | 4                  | 7          | 7          | 11         | 7          | 9          | 7          | 8          | 8             |
| NO. 200   | 1         |           | 1         | 1.1       | 1.1       | 4.2        | 4.2                | 2.9        | 2.9                | 5.5        | 5          | 8.9        | 6.3        | 7.2        | 4.7        | 6.3        | 6.3           |
| LABORATORY TEST RESULTS   |           |           |           |           |           |            |                    |            |                    |            |            |            |            |            |            |            |               |
| % Absorp (-4)   |           |           |           |           |           |            | 1.6                |            | 2.9                |            |            | 1.5        |            |            | 2.5        |            | 2.3           |
| % Absorp (BA +4)  |           |           | 0.44      |           | 1.26      |            | 1.03               |            | 1.66               |            |            | 0.84       |            |            | 1.62       |            | 1.73          |
| Total % Absorption  |           |           |           |           |           |            | 1.21               |            | 2.11               |            |            | 1.24       |            |            | 2.15       |            | 2.1           |
| Bulk SpG (-4)   | 3.352     |           |           | 3.087     |           | 3.022      | 3.008              | 2.847      | 2.825              | 3.064      | 3.043      | 2.947      | 2.985      | 2.878      | 2.985      | 2.967      | 2.967         |
| Bulk SpG (BA +4)  | 3.352     |           | 3.332     | 3.087     | 3.056     | 3.124      | 3.124              | 2.959      | 2.928              | 3.211      | 3.187      | 2.993      | 3.032      | 2.951      | 3.032      | 3.014      | 3.014         |
| Total % Bulk SpG  | 3.352     |           |           | 3.087     |           | 3.092      | 3.088              | 2.917      | 2.89               | 3.099      | 3.095      | 2.962      | 3.004      | 2.908      | 3.004      | 2.985      | 2.985         |
| LAR B-Pct Loss  |           | 16        |           |           |           |            |                    |            |                    |            |            |            |            |            |            |            |               |
| % Clay Balls  |           |           | 0         |           |           |            |                    |            |                    |            | 0          |            |            |            |            |            |               |
| % Other Rock  |           |           | 100       |           |           |            | 100                |            |                    |            | 100        |            |            | 100        |            |            |               |
| % Misc. Spall   |           |           | 0         |           |           |            |                    |            |                    |            | 0          |            |            |            |            |            |               |
| % TotalSampleSpall  |           |           | 0         |           |           |            |                    |            |                    |            | 0          |            |            |            |            |            |               |
| % BA Spall +4   |           |           | 0         |           |           |            |                    |            |                    |            | 0          |            |            |            |            |            |               |
| CAA   | 100       |           |           | 100       |           | 100        |                    | 100        |                    | 100        |            | 100        | 100        |            | 100        |            |               |
| FAA   |           |           |           |           |           |            |                    | 47.6       |                    |            |            |            |            |            |            |            |               |
| Mag%Lost 3/4-1/2  |           | 0.41      |           |           |           |            |                    |            |                    |            |            |            |            |            |            |            |               |
| Mag%Lost 1/2-3/8  |           | 2.09      |           |           |           |            |                    |            |                    |            |            |            |            |            |            |            |               |
| Mag%Lost 3/8-4  |           | 1.76      |           |           |           |            |                    |            |                    |            |            |            |            |            |            |            |               |
| % Mag Total Loss  |           | 1         |           |           |           |            |                    |            |                    |            |            |            |            |            |            |            |               |
| 1 MNDOT LAB POSTS THE CONTRACTOR'S GRADATIONS (SMPS 6 & 7, 8 & 9).  |           |           |           |           |           |            |                    |            |                    |            |            |            |            |            |            |            |               |
| Test Procedures: AASHTO T-19,T-21, T-27 (M), T-30 (M), T-84 (M), T-85 (M), T-96 (M), T-104 (M), T-113 (M), T-176 (M), T-248 (M), T-304 Method A, ASTM C123, ASTM C535, ASTM D3042, ASTM D4791 (M), Litho (MP), Micro Deval (MP), Percent Crushing (MP)<br>M=Mn/DOT Modified; MP=Mn/DOT Procedures |           |           |           |           |           |            |                    |            |                    |            |            |            |            |            |            |            |               |

Non-bituminous usage of taconite aggregate materials is still common, but some changes have occurred in recent years. For example, MnDOT District 1 (Duluth, MN) reports that their office now uses coarse taconite tailings only in bituminous (paving) applications (P. Houston, Resident Engineer, pers. comm., 2010). Prior to and throughout the 2003 Piedmont Avenue project, coarse taconite tailings had also been used as base or sub-grade (fill) material in projects managed out of MnDOT's Duluth office. This change to bituminous-only usage was transportation cost-driven and occurred after a rail haul was no longer available for transporting larger quantities of coarse tailings for lower value fill uses, and more expensive trucking became the only alternative. St. Louis County reported use of Hibbing Taconite's cobber rejects as filter aggregate in road bed construction and reconstruction in the Hibbing area (NOTE: cobber rejects are a 1" to 2" rounded byproduct of autogenous, i.e., rock-on-rock, grinding of taconite ore at the Hibbing Taconite operation). In addition, crushed taconite rock was used as Class 5 (6,854 yds<sup>3</sup>), Select Granular Embankment Mod 7% (8,000 yds<sup>3</sup>), and Granular Embankment Mod 7% (2,550 yds<sup>3</sup>) in the base of Hoover Road in Virginia during 2007-2008 (E. Wilkins, Resident Engineer, pers. comm., 2010).

Since 2004, MnDOT has undertaken several studies on the use of taconite byproduct rock and tailings as aggregate in asphalt paving mixtures. In the first demonstration project, taconite byproduct rock (coarse aggregate) and coarse tailings (fine aggregate) were used in test Cell 31 at MnDOT's MnROAD facility in an HMA mix that would meet MnDOT specifications of a low volume road (LVR). The next study looked at design modifications to produce a Superpave asphalt pavement, design a Stone Matrix Asphalt (SMA) using only taconite byproduct rock, and to evaluate the potential for the use of fine aggregate asphalt using only coarse and fine taconite tailings. Results for this study were published in 2006 (Olson et al., 2006). Taconite materials were used in studies at MnROAD in 2008 in Cells 6 and 23. In Cell 6, a 2 inch HMA fine aggregate (FA) mixture was used over concrete to evaluate the use of thin lift overlays made with a blend of coarse taconite tailings and sand. Railroad ballast-sized taconite byproduct rock was used in Cell 23 as an aggregate base under the asphalt. The 2010 report (Clyne et al., 2010) included results from performance monitoring of test cells, use of taconite tailings for pothole patching, and laboratory testing of the materials.

Results from these studies that apply to thin lift HMA include the following:

- 2004 test cell 31 (Zerfas et al., 2005; and Clyne et al., 2010):
  - Superpave HMA contained 80% coarse taconite aggregate and taconite fine aggregate (tailings); the remaining material was sand;
  - Cell designed for LVR, 20 years, and equivalent single axle loads (ESALs) of 110,000 (level 2 traffic), resulting in a 4 mat inch thickness based on MnDOT software calculations;
  - Taconite coarse aggregate exceeded flatness and elongate particle parameters, so the cell was also designed for a traffic level 2 pavement;
  - Binder used was PG64-34;
  - The unit weight of the asphalt is about 160 pounds per cubic foot;
  - Heavy rollers were used for compaction due to angularity and density of the taconite HMA;
  - Paving was described as "typical" with a "tender spot identified between 220°F and 180°F." Field inspectors would have noticed a tender zone in this temperature range;

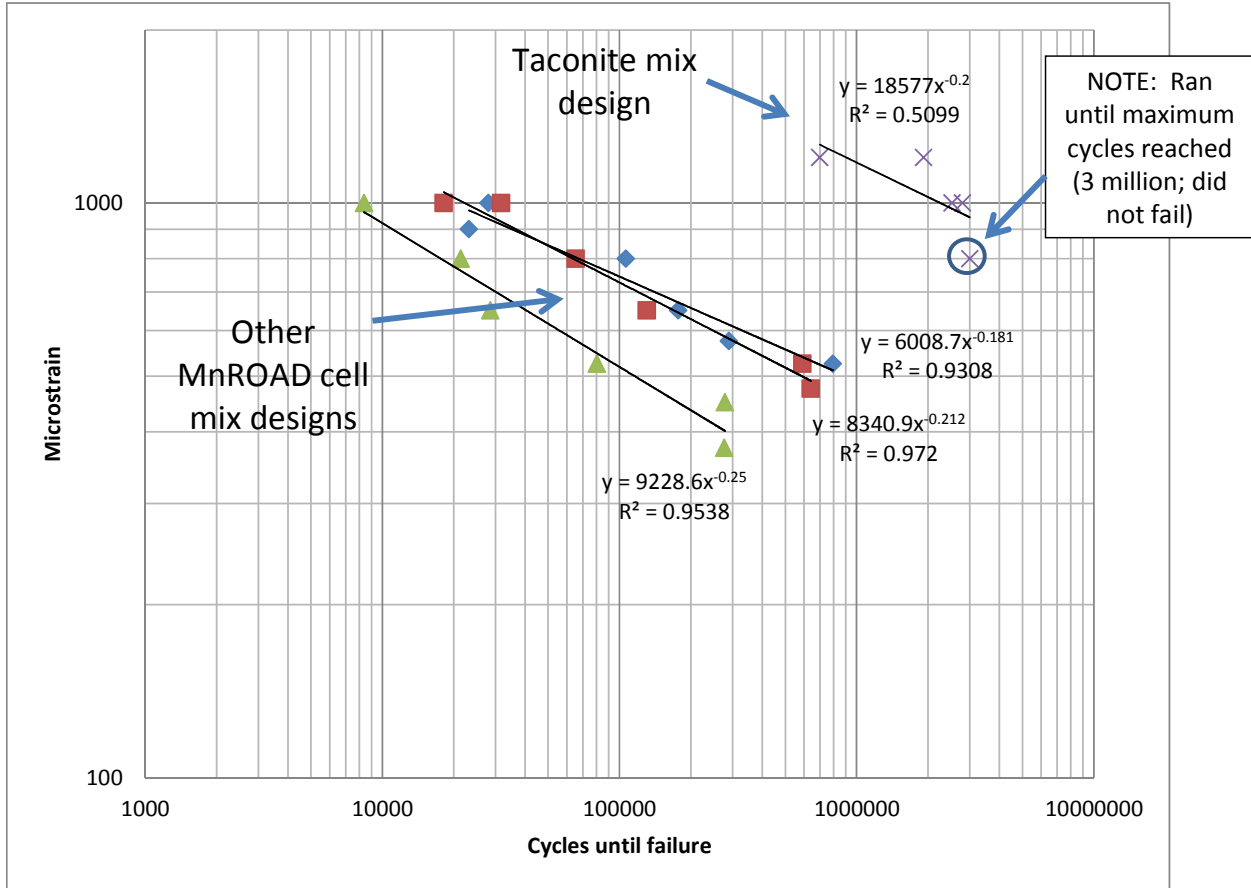
- Problems noted “pneumatic rollers, which picked up the HMA because of the rich mixture and lack of time to adequately warm tires because of the cells short length”;
  - ESALs over a 5 year period were about 75,000 (Clyne et al., 2010); and
  - By 2009, the following were observed during monitoring: two transverse cracks, working cracks from shoulder extended into cell 31, rutting depth was measured as below 0.25 inches and above 0.15 inches (below the average rut depth of ½”), ride quality as fair, friction number was very good (over 50) but declining between 2004 and 2009;
- 2008 test cell 6 (Johnson et al., 2009; Clyne et al., 2010):
    - Cell 6 was subdivided to test the performance of a thin layer fine aggregate HMA over two types of concrete;
    - The pavement was a 4.75 mm Superpave HMA applied in a single layer with a thickness of 2 inches;
    - Aggregate included two sources of taconite tailings and manufactured granite sand;
    - “...was only able to achieve about 90% to 91% density in the field, which was typical of other 4.75 mm mixtures. . .”;
    - The mix design was done by MnDOT’s Trial Mix Lab in Maplewood, MN;
    - Over one year’s, time cell 6 was exposed to about 1 million ESALs (Clyne 2010); and
    - Performance monitoring results in 2009: average rutting depth below 0.1 inches, ride quality index was rated at fair, and the friction number was above 50, very good;
  - Laboratory testing (Clyne et al., 2010):
    - Clyne references the current FHWA (see also MnDOT’s Task F report to NRRI; Clyne et al., 2009) testing and provides a listing of analyses. However, no results are provided.

Clyne’s 2010 report concludes that when properly crushed and sized, taconite tailings can be used in HMA pavements. Skid resistance is listed as “exceptional,” and other performance measurements have been satisfactory.

Further laboratory experimental testing, conducted in 2011 using standard testing procedures available to all material testing laboratories on the same fine graded Cell 6 mixtures, resulted in:

- Excellent rutting capabilities as shown by experimental results obtained on asphalt mixture specimens at MnDOT materials laboratory;
- An increase in low temperature fracture toughness obtained on asphalt mixture specimens at University of Minnesota pavement laboratory; and
- *One order of magnitude increase* in fatigue life measured in fatigue tests performed on asphalt mixture beams at Iowa State University (Fig. 3).

These latest testing findings are presented in greater detail in the Study Area 4 and 5 portions of this report (Sections 2 and 3).



**Figure 3. Beam fatigue testing.**  
 (Sources: Prof. Chris Williams, Iowa State; Tim Clyne, MnDOT.)

## **STUDY AREA 2 – THIN LIFT HMA LITERATURE REVIEW**

### **Synopsis**

#### *Research objectives*

This literature review of development, construction and performance of thin layer hot mix asphalt (HMA) materials in pavement application that require compaction was completed to fulfill requirements for Study Area 2 of the FHWA contract. The results of this review provide a collection of thin HMA types and their specifications. In addition, data regarding taconite tailings usage are compared to the requirements for thin layer HMAs.

#### *Scope of Work*

A literature review was completed to understand the existing requirements for compacted thin layer/overlay HMA. Results from the literature review are then applied to the existing data for taconite tailings characteristics to evaluate their use as an aggregate source for these types of pavements. This literature review has also shown that on-line information sources are ever-expanding (one example being “Pavement Interactive”/<http://www.pavementinteractive.org>) and provide growing access to paving-related issues and data at the state and national levels.

#### *Summary of Results*

Thin lift HMAs are used in several states in the US. Published requirements are available from Maryland, Georgia, Utah, Ohio, Texas, and Michigan and have similar design components and requirements. In general, thin lift overlays can extend the life of a pavement by 8 to 15 years if constructed per specification. Rock types most frequently used in HMAs include limestone and granite. In the last few years, Minnesota has been researching and doing demonstration projects specifically using taconite tailings for the bulk of the aggregate in Superpave and 4.75 mm HMAs. Results from these studies show that taconite aggregate materials can be used in HMAs with few differences in design

### **Literature Review**

#### *Pavement Treatment Types*

Thin lift HMA mixes are utilized for pavement preservation, which means extending the life of a road way before total reconstruction is required. In addition to lengthening pavement life, preservation is conducted to improve the quality of the ride, correct road surface imperfections, and improve road safety. Several types of pavement preservation considered for the literature review are provided in Table 3. These methods include pavements that are or are not compacted as part of the construction process. Further investigation for this review was conducted only on the pavements that require compaction.

**Table 3. Types of Pavement Preservation.**

| <b>Type</b>  | <b>Use</b>   | <b>Application</b>  | <b>Compacted</b> |
|--|--|---|------------------|
| Chip Seal  | Skid resistance, Durability improvement, Raveling  | Spray binder, Spread aggregate, Roll to embed aggregate, Remove excess aggregate                                    | N                |
| Fog Seal   | Coat existing surface to revive asphalt, Surface protection, Raveling                    | Spray   | N                |
| Slurry Seal  | Seal sound oxidized pavements, Improve skid resistance, Waterproofing, Raveling Bridges  | Slurry Machine, slurry seal spreader box (drag box)<br><br>Thin surface treatment. Thickness = largest stone        | N                |
| Micro-Surfacing  | Minor surface irregularities, rutting, improve durability                                | Micro-surface spreader, special spreader box for rutting.<br><br>Thin surface, can be applied at 2-3x largest stone | N                |
| Thin Functional and Maintenance Overlays – Dense Graded                          | Skid resistance, Raveling, Oxidation, Cracking, Surface irregularities                   | Windrowed, spread by paver, rolled – vibratory roller, pneumatic roller, static roller                              | <b>Y</b>         |
| Thin Functional and Maintenance Overlays – Open Graded                           | Skid resistance, Splash, Noise, Raveling, Oxidation, Surface irregularities, Reflections | Windrows or end-dump, spread by paver, rolled – static rollers  | <b>Y</b>         |
| Thin Functional and Maintenance Overlays – Gap Graded (Stone Matrix Asphalt-SMA) | Skid Resistance, Raveling, Oxidation, Surface irregularities                             | Windrowed, paver, rolled – vibratory roller, static roller  | <b>Y</b>         |
| Ultra Thin HMA Bonded Overlays   | Skid resistance, Noise, Splash control   | Spray emulsion, spreader box applies HMA, Compaction required, static steel drum rollers                            | <b>Y</b>         |
| References: NHI Pavement Preservation training documents                         |  |   |                  |

## **Thin Overlay HMA Types**

The Texas Transportation Institute reviewed literature regarding thin overlays and concluded that the following mixes can be utilized: micro surfacing, Nova Chip<sup>RM</sup>, SMA with 3/8" Nominal Maximum Aggregate Size (NMAS), Superpave with 3/8" NMAS, Smoothseal and several Texas DOT mixes (Walubita, 2008). The National Center for Asphalt Technology (NCAT) evaluated a 4.75 mm SMA and had favorable results for its use as a thin overlay.

### *History*

Development of thin lift or thin overlay hot mix asphalts HMAs has been in response to a move from complete reconstruction of all roadways to pavement preservation of structurally sound road pavements. The 1980s saw improvements in polymers to help resist rutting. In the 1990s, stone matrix asphalt (SMA), which combines angular stone on stone contact and crack-resistant binders, was introduced. The Superpave mix design was also developed in the 1990s. By 1999, according to an American Association of State Highway and Transportation Officials (AASHTO) survey, thin lift asphalt overlays had become the most popular treatment for extending the life of a road (Newcomb, 2009). Improvements to technologies, methods, materials, designs, and construction methods continue to advance pavement preservation.

### *General Descriptions*

Hot mix asphalt is defined as consisting of between 93% to 97% aggregate (weight percent) that has been dried at a temperature of 300 degrees Fahrenheit and bound with asphalt binder at a mix plant, and then transported to the road construction site. Compaction is completed at the project site using mechanical spreaders and rollers while the asphalt is still hot. It is placed either on the road sub-base or applied on top of existing HMA surfaces (New Jersey Asphalt Pavement Association, 2009). Typical thicknesses of Thin HMAs are generally less than 1.5 inches (37.5 mm) but can range between 3/4 inches (19 mm) and 2 inches (51 mm) (Cooley, 2002, NCAT 02-04). Ultra-thin HMAs have a thickness of 3/4 inch. Thickness of the overlay and the nominal maximum aggregate size (NMAS) are related. The stone size is limited by the designed thin overlay thickness.

There are three types of Thin HMA, according to National Highway Institute (NHI) training, and they are briefly described below. The difference between these mixes is related to the aggregate, binder, and voids in a particular pavement mix.

1. Dense Graded – aggregate structure is graded from largest to smallest aggregate (grain size);
2. Gap Graded – has a missing size fraction from the gradation, typically the finer aggregate size, to improve stone to stone contact. Stone matrix asphalt (SMA) is a gap graded mix. Fine SMAs have either a 4.75 mm or 9.5 mm NMAS (Cooley, 2003); and
3. Open Graded – aggregate gradation provides voids (between 15-25%) to produce a permeable pavement.

*Beneficial properties*

Use of thin overlay HMAs as method of pavement preservation can extend the life of an asphalt pavement up to 5-10 years by delaying further deterioration of a road and restoring pavement smoothness. The HMA mix can be designed for the specific roadway weather and distress condition. It is a non-structural layer applied for road preservation projects that can correct problems such as:

- Raveling (separation of the aggregate from the pavement and leads to erosion of larger particles, leaving the road surface pitted);
- Oxidation (contributes to hardening of asphalt binder, which results in loss of adhesion properties and viscosity);
- minor cracking;
- some surface irregularities; and
- skid problems (NHI training document, Chap. 9).

Other benefits associated with the thin overlay HMA include: ease of construction, can be feathered to match existing roadway structures, and noise reduction.

Table 4 outlines the various graded asphalts and the distresses for which they can be used to correct.

**Table 4. Graded HMA – Application for Condition.**

| <b>Distress</b>              | <b>Dense Graded</b> | <b>Gap Graded</b> | <b>Open Graded</b> |
|------------------------------|---------------------|-------------------|--------------------|
| Raveling                     | x                   | x                 | x                  |
| Oxidation                    | x                   | x                 | x                  |
| Minor Cracking               | x                   |                   |                    |
| Minor Surface Irregularities | x                   |                   |                    |
| Skid Problems/<br>Hydroplane | x                   | x                 | x                  |
| Splash & Spray               |                     |                   | x                  |
| Noise                        | x                   | x                 | x                  |
| Reflection Cracking          | x                   | x                 |                    |
| Flushing Surfaces            | x                   | x                 |                    |
| Surface Reflection           | x                   |                   | x                  |
| Bleeding Surfaces            | x                   |                   | x                  |



## Components

HMA consists of three components that can be manipulated to obtain a mix design with qualities appropriate for the overlay project. They include the aggregate, asphalt binder, and the ratio of aggregate to binder.

### *Mix Designs*

NOTE: much of the information presented in the following discussion is derived from Washington State's DOT (WsDOT) website: <http://training.ce.washington.edu/wsdot/>.

Three different mix design methods are used to evaluate the mix components for HMA and include Hveem, Marshall, and Superpave. The Hveem mix design was developed in California during the 1920s and 1930s and is based on the following concepts: coating of each aggregate particle by binder, resisting traffic loading due to product stability, and using thicker asphalt binder film thickness for durability (WsDOT, website). Hveem is used in the western United States. In the late 1930s the Marshall method for mix design was used and then modified through the 1950s. This method's main focus is evaluating asphalt binder content density to achieve stability and a range of flow values (WsDOT, website). Marshall methods are preferred by laboratories. HMA Superpave mix design method was developed in the 1990s to replace both the Marshall and Hveem methods. Superpave combines aggregate and asphalt binder to site specific conditions, including climate and traffic, to develop the performance required for a specific road pavement (WsDOT, website). Many states are migrating to using the Superpave method. Marshall and Superpave are commonly used for thin overlay HMAs.

Mix design testing for HMA utilizes a combination of the test methods listed in Table 5. In addition, NCAT utilized/recommended using draindown using 2.36 mm wire mesh basket when working with Superpave mix designs using a 4.75 mm NMA SMA mix designs.

**Table 5. Mix design methods and testing.**

| <b>Testing/Method</b>                         | <b>Hveem</b>                                  | <b>Marshall</b>                        | <b>Superpave</b> |
|---|---|--|------------------|
| <i>Aggregate</i>                              |   |  |                  |
| - Gradation                                   | X   | X                                      | X                |
| - Size  | X   | X                                      | X                |
| - Angularity                                  |   |  | X                |
| - Shape & Texture                             | X   | X                                      | X                |
| - Abrasion                                    | X   | X                                      |                  |
| - Durability                                  | X   | X                                      | X                |
| -Soundness                                    | X   | X                                      |                  |
| - Specific Gravity                            | X   | X                                      |                  |
| - Absorption                                  | X   | X                                      |                  |
| - Deleterious/Clay Materials                  | X   | X                                      | X                |
| <i>Asphalt Binder</i>                         | None Specifically Developed – based on agency | None Specifically for binder selection |                  |
| - Superpave PG Binder (AASHTO MP 1)           | X   | X                                      | X                |
| <i>Mix Design</i>                             |   |  |                  |
| - Centrifuge Kerosene Equivalent (CKE) Test   | X   |  |                  |
| - Compaction w/ California Kneading Compactor | X   |  |                  |
| - Compaction with Marshall Hammer             |   | X                                      |                  |
| - Stabilometer                                | X   |  |                  |
| - Cohesimeter                                 | X   |  |                  |
| <i>Performance Tests</i>                      |   |  |                  |
| - Density and Voids                           | X   | X                                      | X                |
| - Marshall Stability and Flow Test            |   | X                                      |                  |

*Aggregate*

Asphalt pavements contain over 90% aggregate. Aggregates used in HMA pavement typically included crushed limestone, granite, and gravel. Shingles and reclaimed asphalt pavements (RAP) can also be incorporated. Types of aggregates evaluated in papers reviewed for this study include: limestone, granites, taconite tailings, RAP, and manufactured screenings. A brief description for each of the aggregates mentioned above is provided below.

- Limestone – the most commonly used rock type in the U.S.;
- Granite – second most commonly used rock type in the U.S.;

- RAP – Reclaimed Asphalt Pavement;
- Manufactured Screenings (granite & limestone for 4.75 mm NMAAS HMA). Manufactured screenings are defined as follows: *Manufactured screenings are the rock material rejected from a aggregate gradation specification due to its size.* Superpave mixes, which are commonly used, require a coarser gradation, and low fines content. The fines are screened off during the creation of the aggregate gradation, which has created a surplus of Manufactured Screenings (Cooley, 2002); and
- Taconite Tailings (use in Superpave designs in Minnesota).

*Aggregate Gradations (grain size distribution):* For thin HMA overlays, the stone size should be limited to one-half (0.5) the thin overlay thickness. The largest aggregate stone size for a one-inch-thick overlay would be 0.5 inches (12.5 mm). Nominal Maximum Aggregate Size (NMAAS) is defined as one sieve size bigger than the first sieve to retain greater than 10% of the aggregate. For thin overlays, the lift thickness NMAAS ratio needs to be between 1:3 and 1:5 so that proper compaction can be applied. Some typical gradations requirements for thin HMA overlays with NMAAS between 9.5mm and 4.75 mm are presented in the table below. Gradation requirements used in several states and mix designs for thin lift HMAs are provided in Table 6.

**Table 6. Gradation requirements for thin lift HMA.**

| NMAAS      |          | 9.5 mm    |        | 6.3 mm   | 4.75 mm        |          |          |         |             |           |          |
|------------|----------|-----------|--------|----------|----------------|----------|----------|---------|-------------|-----------|----------|
| State      |          | Nevada    | Utah   | New York | 3/8" SuperPave | 3/8" SMA | Maryland | Georgia | Ohio Type B | Texas CAM | Michigan |
| Sieve Size |          | % Passing |        |          |                |          |          |         |             |           |          |
| 1/2"       | 12.5 mm  | 100       | 100    | -        | 100            | 100      | -        | 100     | 100         | 100       | 100      |
| 3/8"       | 9.5 mm   | 85-100    | 90-100 | 100      | 90-100         | 90-100   | 100      | 90-100  | 95-100      | 98-100    | 99-100   |
| No. 4      | 4.75 mm  | 50-75     | <90    | 90-100   | 32-90          | 26-100   | 80-100   | 75-95   | 85-95       | 70-90     | 75-95    |
| No. 8      | 2.36 mm  | -         | 32-67  | 37-70    | 32-90          | 20-65    | 36-76    | 60-65   | 53-63       | 40-65     | 55-75    |
| No. 16     | 1.18 mm  | -         | -      | -        | -              | 13-36    | -        | -       | 37-47       | 20-45     | -        |
| No. 30     | 0.60 mm  | -         | -      | -        | -              | 12-28    | -        | -       | 25-35       | 10-30     | 25-45    |
| No. 50     | 0.30 mm  | -         | -      | -        | -              | 12-22    | -        | 20-50   | 9-19        | 10-20     | -        |
| No. 200    | 0.075 mm | 3-8       | 2-10   | 2-10     | 2-10           | 8-15     | 2-12     | 4-12    | 3-8         | 2-10      | 3-8      |

From: Walubita and Scullion (2008) and Newcomb (2009).

For 4.75 mm Stone Matrix Asphalt (SMA), coarse aggregate size is defined as the largest fraction that is retained on 1.18 mm sieve size (#16 sieve), not the 4.75 mm screen typically used (Xie, 2003).

Grain size distribution is just one aggregate characteristic that needs to be evaluated. Qualities such as shape, angularity, soundness, and resistance to abrasion are other characteristics most often assessed. A listing of possible testing is presented in Table 7. Testing and associated

requirements that need to be met may vary depending on the local agency. Xie et al. (2003), notes for 4.75 mm NMAS SMA, aggregate shape, angularity, and texture influence the ability to achieve the required design criteria.

**Table 7. Additional required aggregate physical characteristics.**

| Characteristic                           | Measurement   | Reference   | Pavement Mix Design        | Testing                                       |
|--|---|---|----------------------------|---|
| Angularity, fine aggregate               | Percent air voids   | Walubita, 2008, p2-3; & SHRP-A-408, 1994; NAPA IS 135, 2009 | Superpave                  | AASHTO T304 and ASTM C1252                    |
| Particle Shape and Texture               | Index value   | Washington State DOT  | Hveem, Marshall            | ASTM D3398                                    |
| Fractured Faces                          | % 2 or more faces fractured, or % 1 face fractured, for aggregate 4.75 + mm | NAPA IS 135, 2009; Washington State DOT, Module 3           | Superpave                  | ASTM D5821                                    |
| Flat & Elongation                        | For Coarse Aggregate (larger than 4.75 mm)                                  | SHRP-A-408, 1994; Washington State DOT, Module 3            | Superpave                  | AASHTO D4791 or ASTM D4791                    |
| Toughness/Abrasion Resistance            | LA abrasion loss value of <40   | Walubita, 2008, p2-3; Washington DOT Models 3, 4, 5         | Superpave, Hveem, Marshall | AASHTO T96                                    |
| Soundness                                | Percent degradation/loss  | SHRP-A-408, 1994; NAPA IS 135, 2009                         | Superpave, Hveem, Marshall | Sodium or Magnesium test                      |
| Deleterious Materials, Clay/Dust Content | Percent by weight, 40-45%   | SHRP-A-408, 1994; Rausch, 2006, p9; Newcomb, 2009           | Superpave, Hveem, Marshall | Sand Equivalent Test, AASHTO T176, ASTM D2419 |
| Flakiness                                | Index less than 18  | Walubita, 2008, p2-3  |                            |   |
| Stone Polish                             | 50  | Walubita, 2008, p2-3  |                            |   |
| Aggregate Crushing Value                 | 20  | Walubita, 2008, p2-3  |                            |   |
| Specific Gravity                         |   |   | Superpave, Hveem, Marshall | AASHTO T84, ASTM E12                          |
| Moisture Content                         | Percent   | Washington State DOT, Module 3                              |                            | ASTM C70 - Surface<br>ASTM C566 Total         |
| Water Absorption                         | 1.5% maximum  | Walubita, 2008, p2-3  | Superpave, Hveem, Marshall | AASHTO T84                                    |
| Permeability/Drain Down Test             |   | Walubita, 2008  |                            | AASHTO T305-97 for SMAs                       |

Typical negative effects due to out-of-specification results for characteristics listed in Table 7 can include: increased rutting, stripping, and decreased stability (Rausch, 2006). These are problems that can be corrected with the application of a thin overlay.

## Asphalt Binders

For Superpave projects, asphalt binder selection is based on the project climate (temperatures) and traffic conditions. Other characteristics considered include workability, durability and performance. Superpave uses a performance grade (PG) system of classifying and selecting asphalt binder in relation to site specific conditions including high and low temperatures, traffic speed, and measurement of traffic load using equivalent single axle loads (ESAL). In order to correctly select the asphalt binder, a 20-year ESAL is used. Performance grading (PG) of asphalt binders consists of laboratory testing to determine the best asphalt binder for given climate conditions with regard to rutting and cracking of pavement. Numbers following the PG designation represent the maximum 7-day average temperature and the 1-day minimum temperatures (°C) that are likely to be experienced. A higher first number provides more rut resistance, and a lower second number provides more cracking resistance. Some asphalt binder requirements from several states are provided below. The Hveem and Marshall methods do not have a specific binder selection or testing process but rely on the local agency to specify types and test methods. Superpave's procedures are the most commonly-specified methods. Binder types required in several different states are provided in Table 8.

**Table 8. Example states and asphalt binders.**

| Binder Type   | State            | Comments   |
|---|------------------|--|
| PG 64-22 or PG 76-22  | Ohio             | Polymer modified (PG) graded asphalt   |
| PG 64-22 or PG 76-22  | New York         | Upper State or Lower State   |
| PG 76-22  | New Jersey       |  |
| PG 76-22 or PG 64-22  | North Carolina   | ESAL included in selection criteria. Highest ESAL or Lowest ESAL. 4.75 mm mixes only used for ESAL less than 300,000 so PG 64-22 is specified. |
| AASHTO MP1  | Washington State |  |
| PG 64-34 (Note: PG 58-28 and PG 58-34 are more typical for MN.) | Minnesota        | MnDOT has been using this modified polymer binder for HMA demonstration projects.  |
| References: Newcomb (2009) and Zerfas (2005).                   |                  |  |

Asphalt binders can include addition of modifiers, either natural or synthetic. The purpose of the modifiers is to improve resistance to rutting, cracking, performance at certain temperature ranges, and oxidation. Examples include fillers, polymers, crumb rubber, and fibers, some of which are also added to the HMA at mix design specified quantities.

## Performance

### *Concerns and Typical Distresses*

According to the Walubita et al. (2008) paper, *Thin HMA Overlays in Texas: Mix Design and Laboratory Material Property Characterization*, if done properly, the life expectancy of thin lift HMA is between 8-15 years. The longevity of the pavement is dependent on creating the correct

mix design, structural integrity of the existing pavement structure, and the pavement preparation prior to HMA placement. Common distresses can include “bleeding, reflective cracking, fatting, texture loss, and decrease in skid resistance.”

### **Minnesota HMA and Taconite Tailings**

In the literature, commonly quoted State programs include Maryland, Texas, Utah, and Georgia. However, Minnesota has been working on evaluations of HMAs for some time. Specifically, Minnesota has been investigating the performance and mix designs for HMAs and SMAs containing crushed taconite byproduct rock (also referred to as Mesabi Rock). In MnDOT reporting (Clyne et al., 2010), it was noted that taconite materials performed well as aggregate in the HMAs. State specifications were met for level 2 (ESALs < 1 million). Elongation and flatness of particles exceeded MnDOT requirements for level 4 and level 3 ESAL roads. Specifications for 4.75 mm HMA and SMA containing coarse taconite tailings are the same as for other aggregates, where tailings are considered as another rock type. It should be noted that the shape characteristics of taconite fine aggregate (coarse taconite tailings) will tend to be more angular because the ore from which this fine aggregate is derived comes from the cherty portions of the iron-formation, rather than from the slaty portions.

As part of this project, MnDOT and UM-CE have been working on mix designs utilizing taconite fine aggregate (coarse taconite tailings). Results from their investigation are presented in Sections 2 and 3 (Study Areas 4 and 5) of this report.

## STUDY AREA 3 – LEACHING POTENTIAL OF TACONITE TAILINGS

### Synopsis

#### *Research objectives*

Physical, mineralogical, and chemical characteristics of coarse taconite tailings and ten other common fine aggregates used in Minnesota road construction were evaluated. Non-taconite samples were provided by the Minnesota Department of Transportation (MnDOT) Office of Materials located in Maplewood, MN. The twelve samples of HMA aggregate included sand, granite, limestone, gravel, and coarse taconite tailings. The purpose of the testing was to compare the various project aggregates as-is, i.e., in a raw unbound (non-bituminous mix) condition. Practically speaking, the particle surfaces of all of the aggregate materials tested would be largely encapsulated by an asphalt binder when used in thin lift HMA applications, and their exposure to ambient environmental conditions would therefore be greatly reduced. Consequently, the analytical results presented in this section could be viewed as “worst-case.”

#### *Scope of Work*

Analyses conducted on the twelve samples to determine physical, mineralogical, and chemical characteristics included gradations, X-ray powder diffraction (XRD), hand held X-ray fluorescence (XRF) elemental analysis, pH, Toxicity Characteristics Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP), variable pH solubility and release, and variable liquid solid ratio (L/S) solubility and release (leaching). Table 9 lists the samples and denotes the tests conducted on each.

#### *Summary of Results*

Taconite fine aggregate (coarse tailings) conform to gradation characteristics for fine aggregates used for HMA requirements and are similar to most other aggregates used for this study.

Chemical analysis evaluation of leachate produced from the aggregates indicates that taconite tailings present minimal risk to surface or drinking water when the results are compared to the U.S. Environmental Protection Agency’s (EPA) published national drinking and surface water standards. Again, the leachate results are primarily for unbound aggregates. Limited leachate testing (SPLP) was performed on three asphalt-bound samples to provide a point of comparison to unbound aggregates.

**Table 9. List of FHWA project aggregate samples and analyses conducted.**

| Sample ID      | Material                               | Gradation | SPLP | TCLP | XRF | XRD | Variable pH | Variable L/S ratio |
|----------------|--|-----------|------|------|-----|-----|-------------|--------------------|
| E1             | RIVER SAND                             | X         | X    | X    | X   | X   |             |                    |
| E4             | MARTIN MARIETTA SC WASH SAND (GRANITE) | X         | X    | X    | X   | X   |             |                    |
| E7             | VONCO BA SAND                          | X         | X    | X    | X   | X   |             |                    |
| E8             | LOKEN MAN SAND (DIRTY SAND)            | X         | X    | X    | X   | X   | X           | X                  |
| E11            | BARTON ELK RIVER FINE SAND             | X         | X    | X    | X   | X   |             |                    |
| E12            | MARTIN MARIETTA WASHED SAND (GRANITE)  | X         | X    | X    | X   | X   | X           | X                  |
| W6             | CLASS 3 (GRAVEL)                       | X         | X    | X    | X   | X   | X           | X                  |
| SHINGLES       | MAN MADE                               | X         | X    | X    | X   | X   |             |                    |
| M-LS           | LIMESTONE                              | X         | X    | X    | X   | X   | X           | X                  |
| G 3/8 BA LS    | LIMESTONE                              | X         | X    | X    | X   | X   |             |                    |
| ARCELOR MITTAL | TACONITE TAILINGS                      | X         | X    | X    | X   | X   | X           | X                  |
| MINNTAC        | TACONITE TAILINGS                      | X         | X    | X    | X   | X   | X           | X                  |

## Aggregate Physical Characteristics

### *Grain Size*

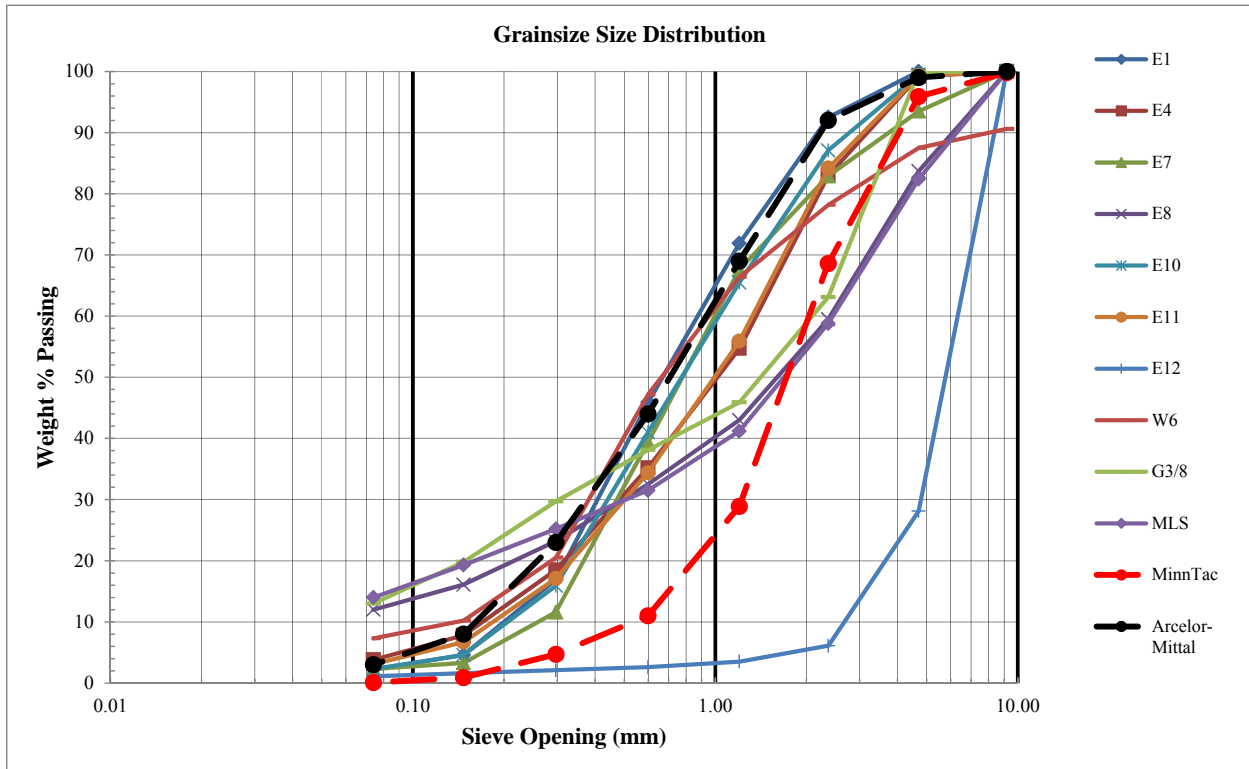
Samples of each MnDOT-supplied aggregate were submitted to Precision Testing in Virginia, MN, for sieve analyses. Grain size analyses on taconite tailings samples were completed at NRRI. Table 10 summarizes the grain size results, which are also plotted graphically in Figure 4. Laboratory testing reports are included in the Appendices.

A comparison of the gradation of taconite tailings to other aggregates (Fig. 4) shows the tailings from ArcelorMittal (Minorca) are similar to those of the other aggregate types, except for E-12, a granite sample. The grain size distribution shown for the 2011 Minntac tailings (highlighted in yellow in Table 10) is not typical of previous (2001) or more recent (2009) analyses (in red text), especially at the smaller-sized fractions. It is possible that particle segregation occurred during handling and unloading at NRRI's Coleraine Laboratory and while the small stockpile was exposed to the elements prior to sampling.



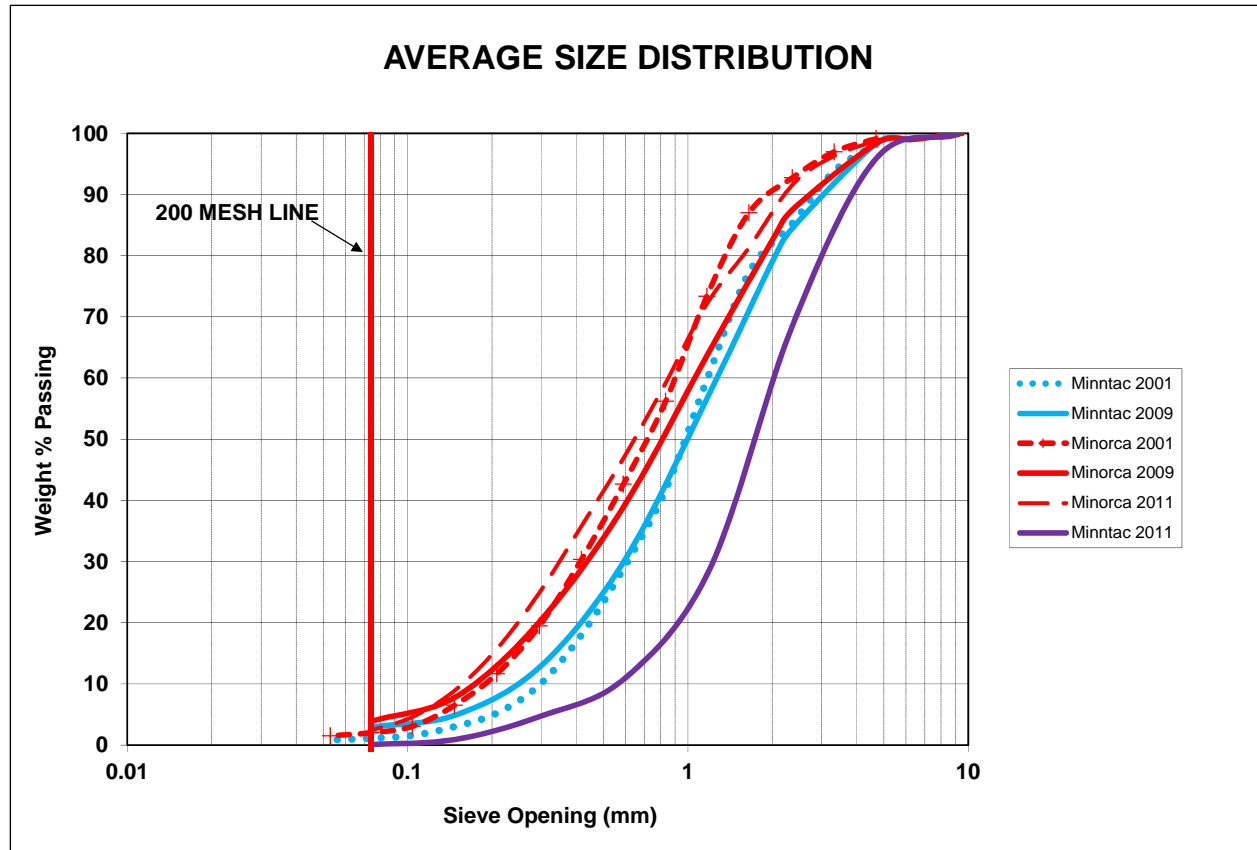
**Table 10. Gradation results for Study Area 3 aggregate samples.**

| Test Material Gradation Results |                  |                    |        |        |       |       |       |       |       |       |        |
|---------------------------------|------------------|--------------------|--------|--------|-------|-------|-------|-------|-------|-------|--------|
| Sample:                         | Date Collected:  | Mesh Size          | 3/8"   | #4     | #8    | #16   | #30   | #50   | #100  | #200  | Bottom |
|                                 |                  | Sieve Opening (mm) | 9.510  | 4.760  | 2.380 | 1.190 | 0.595 | 0.027 | 0.149 | 0.074 | 0.000  |
| E1                              | March 26 2010    | Wt.% Passing       | 100.00 | 100.00 | 92.50 | 71.90 | 45.90 | 16.90 | 4.60  | 2.20  | 0.00   |
| E4                              | March 26 2010    | Wt.% Passing       | 100.00 | 99.20  | 83.00 | 54.70 | 35.20 | 18.50 | 7.80  | 3.80  | 0.00   |
| E7                              | March 26 2010    | Wt.% Passing       | 100.00 | 93.50  | 82.90 | 67.60 | 39.40 | 11.60 | 3.30  | 2.30  | 0.00   |
| E8                              | March 26 2010    | Wt.% Passing       | 100.00 | 83.70  | 59.50 | 43.00 | 32.50 | 23.20 | 16.10 | 12.00 | 0.00   |
| E10                             | March 26 2010    | Wt.% Passing       | 100.00 | 99.50  | 87.10 | 65.50 | 41.00 | 15.90 | 4.60  | 2.30  | 0.00   |
| E11                             | March 26 2010    | Wt.% Passing       | 100.00 | 99.30  | 84.20 | 55.90 | 34.40 | 17.10 | 6.70  | 3.10  | 0.00   |
| E12                             | March 26 2010    | Wt.% Passing       | 100.00 | 28.10  | 6.10  | 3.50  | 2.60  | 2.10  | 1.60  | 1.10  | 0.00   |
| W6                              | March 26 2010    | Wt.% Passing       | 90.60  | 87.50  | 78.20 | 66.40 | 47.20 | 20.50 | 10.20 | 7.30  | 0.00   |
| G3/8 BALS                       | March 26 2010    | Wt.% Passing       | 100.00 | 99.90  | 63.10 | 45.90 | 38.10 | 29.70 | 19.80 | 13.00 | 0.00   |
| MLS MAN SAND                    | March 26 2010    | Wt.% Passing       | 100.00 | 82.40  | 58.80 | 41.20 | 31.50 | 25.20 | 19.30 | 14.00 | 0.00   |
| Min-1-CT                        | February 8, 2011 | Wt.% Passing       | 100.00 | 96.30  | 76.70 | 39.90 | 15.90 | 4.10  | 1.40  | 1.00  | 0.00   |
| MLS MAN SAND                    | March 26 2010    | Wt.% Passing       | 100.00 | 98.80  | 90.10 | 67.40 | 42.70 | 20.80 | 8.00  | 4.70  | 0.00   |
| Minntac                         | Nov 4 2011       | Wt.% Passing       | 99.80  | 95.90  | 68.60 | 28.90 | 11.00 | 4.70  | 0.90  | 0.10  | 0.00   |
| Minntac                         | 2001 data        | Wt.% Passing       | 100.00 | 98.20  | 85.33 | 59.68 | 28.88 | 9.83  | 3.03  | 1.13  | na     |
| Minntac                         | 2009 data        | Wt.% Passing       | 100.00 | 98.50  | 84.50 | 57.00 | 30.50 | 13.00 | 5.00  | 3.00  | na     |
| Arcelor Mittal                  | Nov 4 2011       | Wt.% Passing       | 100.00 | 99.00  | 92.00 | 69.00 | 44.00 | 23.00 | 8.00  | 3.00  | 0.00   |



**Figure 4. Plot of sieve analysis results, log scale.**

Figure 5, which compares the result for the single 2011 test to: a) Minntac gradations summarized in Zanko et al. (2003); and b) to more recent (unpublished) NRRI data collected in 2009 (blue lines), strongly suggests that the 2011 Minntac result (dark purple line) is an anomaly. Similar data (2001 and 2009) for the Minorca tailings (red lines) are also plotted in Figure 5 to further illustrate how the size distribution of both mines' coarse tailings typically falls within a narrow gradation band.



**Figure 5. Minntac and Minorca gradation comparison.**

Table 11 is a modification of Table 6 and compares the taconite tailings gradation results to the various state specifications.

**Table 11. Gradation of taconite tailing relative to state specifications.**

| NMAAS  |          | 9.5 mm    |        | 6.3 mm   | 4.75 mm        |          |          |         |             |           |          | Taconite Tailings |                     |                            |
|--|----------|-----------|--------|----------|----------------|----------|----------|---------|-------------|-----------|----------|-------------------|---------------------|----------------------------|
| State  |          | Nevada    | Utah   | New York | 3/8" SuperPave | 3/8" SMA | Maryland | Georgia | Ohio Type B | Texas CAM | Michigan | Minntac 2011      | Arcelor Mittal 2011 | Minntac 2001, 2009 average |
| Sieve Size   |          | % Passing |        |          |                |          |          |         |             |           |          |                   |                     |                            |
| 1/2"   | 12.5 mm  | 100       | 100    | -        | 100            | 100      | -        | 100     | 100         | 100       | 100      | 99.8              | 100                 | 100                        |
| 3/8"   | 9.5 mm   | 85-100    | 90-100 | 100      | 90-100         | 90-100   | 100      | 90-100  | 95-100      | 98-100    | 99-100   | 95.9              | 99                  | 100                        |
| No. 4  | 4.75 mm  | 50-75     | <90    | 90-100   | 32-90          | 26-100   | 80-100   | 75-95   | 85-95       | 70-90     | 75-95    | 68.6              | 92                  | 98                         |
| No. 8  | 2.36 mm  | -         | 32-67  | 37-70    | 32-90          | 20-65    | 36-76    | 60-65   | 53-63       | 40-65     | 55-75    | 28.9              | 69                  | 85                         |
| No. 16   | 1.18 mm  | -         | -      | -        | -              | 13-36    | -        | -       | 37-47       | 20-45     | -        | 11                | 44                  | 58                         |
| No. 30   | 0.60 mm  | -         | -      | -        | -              | 12-28    | -        | -       | 25-35       | 10-30     | 25-45    | 4.7               | 23                  | 30                         |
| No. 50   | 0.30 mm  | -         | -      | -        | -              | 12-22    | -        | 20-50   | 9-19        | 10-20     | -        | 0.9               | 8                   | 11                         |
| No. 200  | 0.075 mm | 3-8       | 2-10   | 2-10     | 2-10           | 8-15     | 2-12     | 4-12    | 3-8         | 2-10      | 3-8      | 0.1               | 3                   | 2                          |
| From: Walubita and Scullion (2008), and Newcomb (2009)<br>Numbers in bold indicate non-compliance with state requirements. |          |           |        |          |                |          |          |         |             |           |          |                   |                     |                            |

## Aggregate Mineralogical and Chemical Characteristics

### *Mineralogy by powder x-ray diffractometry*

Powder x-ray diffractometry (XRD) is the analytical method of choice for identifying and determining the proportions of minerals and/or other crystalline or metallic materials present in geological materials such as aggregates. The XRD results can also provide a “mineralogical signature” of sorts to indicate the type of deposit or material from which the aggregate was sourced.

XRD was completed on all study samples at the University of Minnesota Duluth (UMD) Research Instrumentation Laboratory. Sub-samples of each aggregate sample were ground to a fine homogeneous powder and analyzed using a Phillips XPert MPD powder x-ray diffractometer. The resulting data were interpreted and compared to standard reference patterns to identify each sample’s mineralogy.

Results are presented in Table 12. With the exception of the two limestone aggregate samples, quartz is the dominant mineral in all samples. The taconite tailings samples are elevated in iron oxides, iron carbonates, and iron silicates, which is typical of these iron ore byproduct materials. Interestingly, a small amount of brass was identified in the recycled asphalt shingles (RAS) sample, most likely from trace amounts of metallic fasteners ground up in the shingle recycling process.

**Table 12. Powder XRD results: mineral percentage by sample.**

|                        | E1         | E4      | E7   | E8         | E10       | E11     | E12     | G 3/8 BA<br>LS | MLS<br>MAN<br>Sand | Shingles | W6     | Arcelor-<br>Mittal | Minntac    |
|------------------------|------------|---------|------|------------|-----------|---------|---------|----------------|--------------------|----------|--------|--------------------|------------|
| Mineral<br>Name        | River sand | Granite | Sand | Dirty Sand | Fine Sand | Granite | Granite | Limestone      | Limestone          | Shingles | Gravel | Tailings-AM        | Tailings-M |
| Quartz                 | 70         | 65      | 75   | 70         | 75        | 65      | 75      | 2              | 3                  | 35       | 100    | 88                 | 85         |
| Albite                 | 25         | 15      | 15   | 15         | 15        | 12      |         |                |                    |          |        |                    |            |
| Wustite                | 3          |         |      |            |           |         |         |                |                    |          |        |                    |            |
| Microcline             | 2          |         |      |            |           |         |         |                |                    |          |        |                    |            |
| Biotite                |            | 5       |      |            |           |         |         |                |                    |          |        |                    |            |
| Augelite               |            | 5       |      |            |           |         |         |                |                    |          |        |                    |            |
| Anorthite              |            | 5       | 3    |            |           | 10      | 20      |                |                    | 10       |        |                    |            |
| Sanidine               |            | 3       |      | 7          |           |         | 3       |                |                    |          |        |                    |            |
| Pargasite              |            | 2       |      |            |           |         |         |                |                    |          |        |                    |            |
| Calcite                |            |         | 5    |            | 10        |         |         |                |                    | 25       |        |                    |            |
| Dolomite               |            |         | 2    | 6          |           | 4       |         | 95             | 97                 | 25       |        |                    |            |
| Clinochlore            |            |         |      | 1          |           |         |         |                |                    |          |        |                    |            |
| Tosudite               |            |         |      | 1          |           |         |         |                |                    |          |        |                    |            |
| Orthoclase             |            |         |      |            |           | 8       |         |                |                    |          |        |                    |            |
| Illite                 |            |         |      |            |           | 1       |         |                |                    |          |        |                    |            |
| Magnesian<br>Chamosite |            |         |      |            |           |         | 2       |                |                    |          |        |                    |            |
| Microcline             |            |         |      |            |           |         |         | 3              |                    |          |        |                    |            |
| Brass                  |            |         |      |            |           |         |         |                |                    | 5        |        |                    |            |
| Hematite               |            |         |      |            |           |         |         |                |                    |          |        | 10                 | 6          |
| Minnesotaite           |            |         |      |            |           |         |         |                |                    |          |        | 1                  | 2          |
| Geothite               |            |         |      |            |           |         |         |                |                    |          |        | 1                  |            |
| Sepiolite              |            |         |      |            |           |         |         |                |                    |          |        |                    | 3          |
| Clinochlore-<br>1M11b  |            |         |      |            |           |         |         |                |                    |          |        |                    | 2          |
| Siderite               |            |         |      |            |           |         |         |                |                    |          |        |                    | 2          |

*Element composition by X-ray fluorescence*

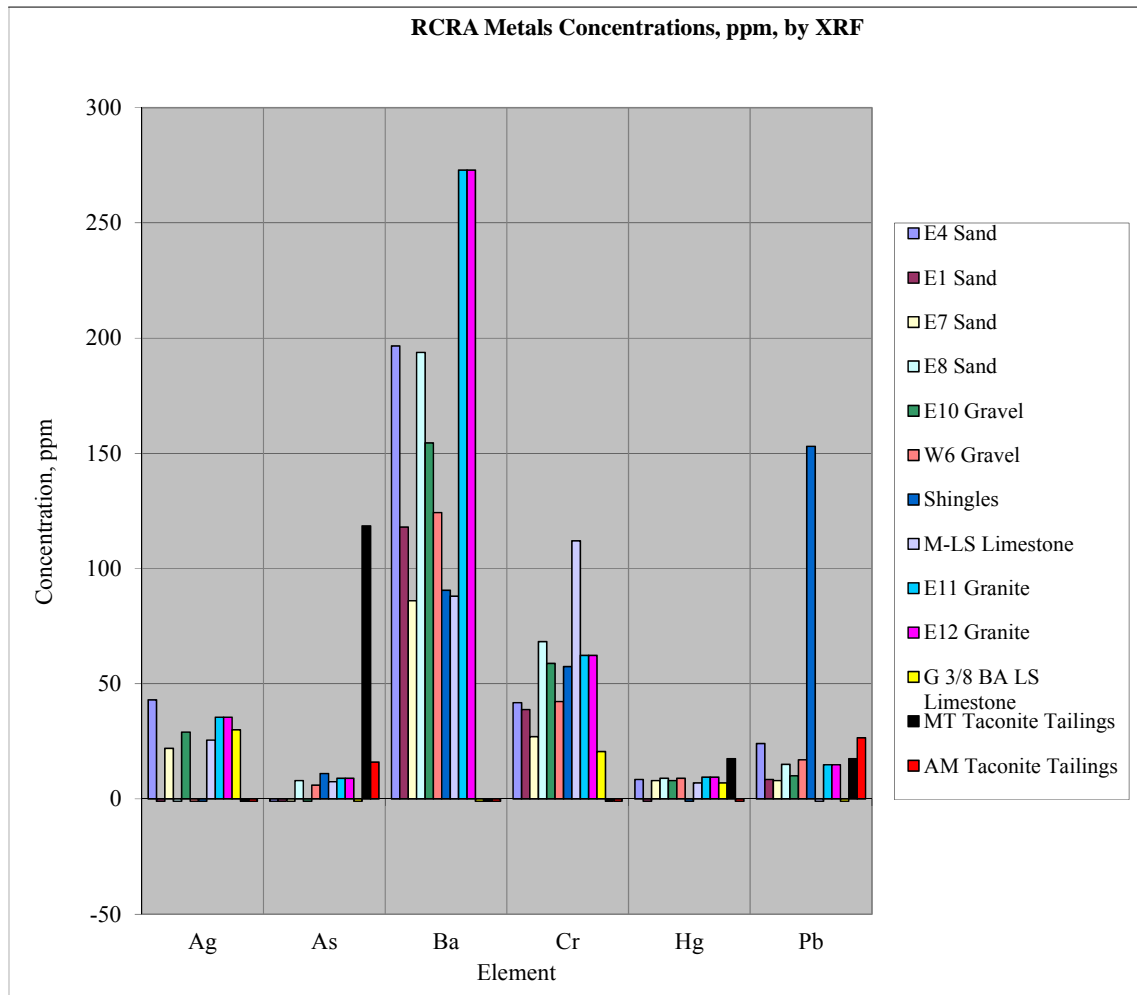
Elemental analysis of each aggregate was completed using NRRI’s handheld Olympus Innov-X XRF. To determine elemental composition, a subsample (approximately 15 grams) of each aggregate material was placed on the stage of the laboratory work station and exposed to the device’s x-rays. Multiple readings (3 to 5) were taken and recorded for each subsample, and the XRF device’s soils application software was used to qualitatively determine the concentration of elements present, in parts per million (ppm). These data were then downloaded into an Excel spreadsheet. The average of the Resource Conservation and Recovery Act (RCRA) Metals for each sample are presented in Table 13, while Mn, Fe, and Co averages are presented in Table 14. Figures 6 and 7 display the results for both groups of metals. Selenium (Se) reported below the

detection limit (<1 ppm) for each sample and is *not* plotted in Figures 6 and 7. Minntac and ArcelorMittal (Minorca) tailings are abbreviated MT and AM, respectively, in both figures.

**Table 13. XRF results: RCRA metals.**

| Description       | Sample #       | Material             | Cr     | As     | Se    | Ag    | Cd    | Ba     | Hg    | Pb     |
|-------------------|----------------|----------------------|--------|--------|-------|-------|-------|--------|-------|--------|
| martin-marietta   | E4             | Sand                 | 41.80  | -1.00  | -1.00 | 43.00 | -1.00 | 196.60 | 8.50  | 24.00  |
| sand              | E1             | Sand                 | 38.80  | -1.00  | -1.00 | -1.00 | -1.00 | 118.00 | -1.00 | 8.50   |
| sand, ba vonco    | E7             | Sand                 | 27.00  | -1.00  | -1.00 | 22.00 | -1.00 | 86.00  | 8.00  | 8.00   |
| sand, loken       | E8             | Sand                 | 68.25  | 8.00   | -1.00 | -1.00 | -1.00 | 193.75 | 9.00  | 15.00  |
| sand-fine-barton  | E10            | Sand                 | 58.83  | -1.00  | -1.00 | 29.00 | -1.00 | 154.50 | 8.00  | 10.00  |
| gravel, class 3   | W6             | Gravel               | 42.25  | 6.00   | -1.00 | -1.00 | -1.00 | 124.25 | 9.00  | 17.00  |
| shingles          | Shingles       | Shingles             | 57.40  | 11.00  | -1.00 | -1.00 | -1.00 | 90.60  | -1.00 | 153.00 |
| limestone         | M-LS           | Limestone            | 112.00 | 7.50   | -1.00 | 25.50 | -1.00 | 88.00  | 7.00  | -1.00  |
| ST. CLOUD         | E11            | Granite              | 88.50  | 7.00   | -1.00 | -1.00 | -1.00 | 278.33 | 9.00  | 19.00  |
| ST. CLOUD, CLEAR  | E12            | Granite              | 62.29  | 9.00   | -1.00 | 35.50 | -1.00 | 272.86 | 9.50  | 14.86  |
| limestone         | G 3/8 BA<br>LS | Limestone            | 20.60  | -1.00  | -1.00 | 30.00 | -1.00 | -1.00  | 7.00  | -1.00  |
| taconite tailings | MT             | Taconite<br>Tailings | -1.00  | 118.50 | -1.00 | -1.00 | -1.00 | -1.00  | 17.50 | 17.50  |
| taconite tailings | AM             | Taconite<br>Tailings | -1.00  | 16.00  | -1.00 | -1.00 | -1.00 | -1.00  | -1.00 | 26.50  |

If the average contained a majority of <LOD in the results, then the average was recorded as "-1"

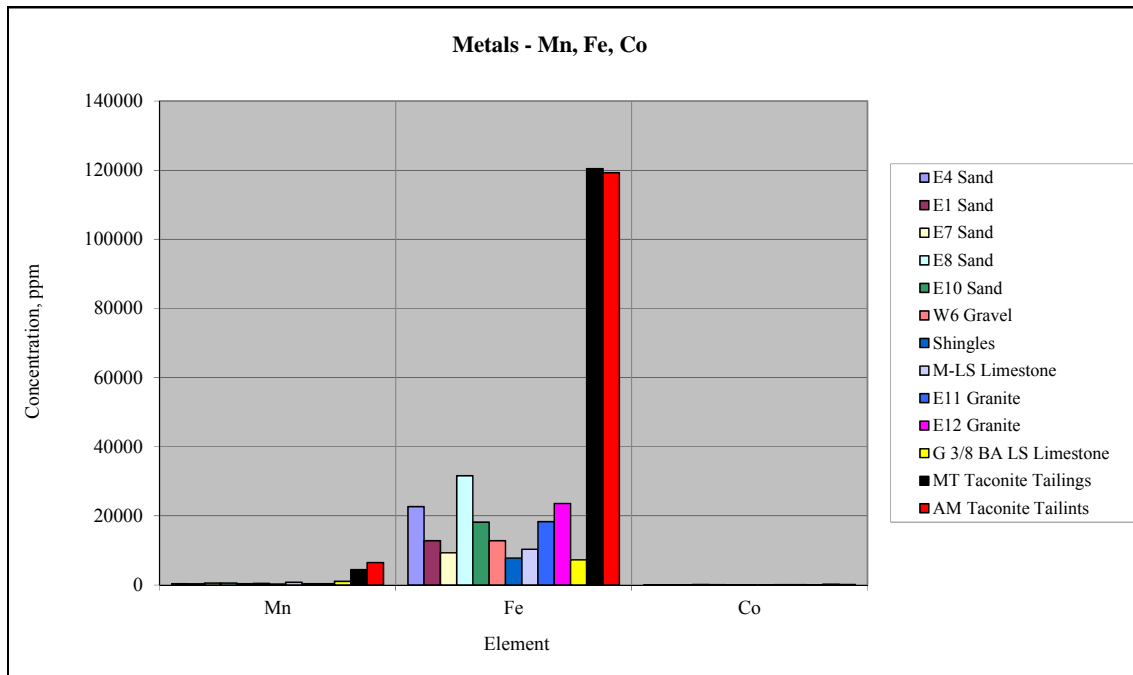


**Figure 6. RCRA metals by XRF.**

Unsurprisingly, Mn, Fe, and Co levels are highest in the two taconite fine aggregate (coarse tailings) samples (Table 14 and Fig. 7).

**Table 14. XRF results: Mn, Fe, and Co.**

| Description                      | Sample #    | Material          | Mn   | Fe     | Co  |
|----------------------------------|-------------|-------------------|------|--------|-----|
| martin-marietta                  | E4          | Sand              | 353  | 22635  | 62  |
| sand                             | E1          | Sand              | 329  | 12839  | 52  |
| sand, ba vonco                   | E7          | Sand              | 549  | 9299   | 29  |
| sand, loken                      | E8          | Sand              | 544  | 31603  | 129 |
| sand-fine-barton                 | E10         | Sand              | 358  | 18196  | 76  |
| gravel, class 3                  | W6          | Gravel            | 420  | 12844  | 56  |
| shingles                         | Shingles    | Shingles          | 269  | 7767   | 37  |
| limestone                        | M-LS        | Limestone         | 756  | 10354  | 32  |
| ST. CLOUD                        | E11         | Granite           | 404  | 18289  | 67  |
| ST. CLOUD, CLEAR                 | E12         | Granite           | 361  | 23555  | 70  |
| limestone                        | G 3/8 BA LS | Limestone         | 1071 | 7289   | 22  |
| taconite tailings, Minntac       | MT          | Taconite Tailings | 4441 | 120455 | 195 |
| taconite tailings, ArcelorMittal | AM          | Taconite Tailings | 6512 | 119245 | 176 |



**Figure 7. Mn, Fe, and Co by XRF.**

## Water Standards

Water Standards used for this study include: EPA Drinking Water Standards and EPA Recommended Water Quality (RWQ) criteria, Minnesota Drinking Water Standards, and Minnesota Surface Water Standards. The EPA standards are used as a common reference for the study's leachate evaluation. The Minnesota standards are presented as a more stringent reference.

Descriptions of the standards are presented below. All water quality/leachate values are reported or shown in µg/L (parts per billion, or ppb), unless otherwise noted.

*EPA Water Quality Standards*

EPA’s Water Quality Standards provide guideline (minimum) water quality standards that individual states can use as a basis for developing their own standards. EPA’s Water Quality Standards are mandated by the Clean Water Act. The Act defines the goals for the standards based on the use – and setting – of a water body to protect it from pollutants that would degrade it for other uses.

The EPA also sets the standards for ground water and surface water to protect aquatic and human health. These standards are based on analytical data associated with scientific study. National Primary Drinking Water Standards (NPDWS) protect the public drinking water systems by limiting the levels of pollutants and are legally enforceable; while the National Recommended Water Quality (NRWQ) criteria apply to surface waters. There are approximately 150 pollutants on the EPA’s list, and this list is used as guidance for the state agencies. NRWQ values are subdivided into acute and chronic. Acute applies to short term exposure and chronic to long term.

**Minnesota Drinking Water Standards**

Drinking water is designated as Class 1B in the Minnesota Pollution Control Agency (MPCA) Surface Water Standards, as reported in Tables 9 and 11 of the MPCA publication, “Working Draft Surface Water Pathway Evaluation User’s Guide, MPCA Remediation Programs,” by Gnabasik and White (2006). The standards include values that are: Maximum Contaminant Levels (MCLs), Health Risk Limits (HRLs), or Health Based Values. For this report, the drinking water standard for each analyte is used as a comparison for all of the sample results.

*Minnesota Surface Water Standards*

There are seven classes of surface waters in Minnesota with separate standards that are based on the type of water use. The classes as defined by Gnabasik and White (2006) are:

|   |   |
|---|---|
| 1 – drinking                              | 5 – navigation and aesthetics               |
| 2 – aquatic life, recreation, and habitat | 7 – limited use                             |
| 3 – industry                              | 6 – other uses not listed in items 1 thru 5 |
| 4 - agriculture                           | and 7                                       |

Class 2 waters that are defined as used for “*Aquatic Life and Recreation: Waters which do or may support fish, other aquatic life, bathing, boating, or other recreational purposes, and where quality control is or may be necessary to protect aquatic or terrestrial life or their habitats, or the public health, safety, or welfare.*” There are 5 subdivisions (A-E) of the Class 2 waters that are determined by the following criteria: 1) is the water a protected drinking water source; 2) requirements for specific fish, other aquatic life, and their associated habitat; and 3) protected aquatic recreations uses (see Table 3, p. 20, of Gnabasik and White (2006)). Class 2A and 2Bd are protective of drinking water. For this study, results are compared to Class 2A, 2B, 2C, and 2D standards.

Values are calculated for Chronic Standards, Maximum Standards, and Final Acute Values. The Chronic Standard (CS) is the level of a contaminant that an aquatic organism can tolerate without chronic toxic effects. Maximum Standard (MS) is the highest quantity of a pollutant that an organism can be exposed to for a brief time with “zero to slight mortality.” Final Acute Values are defined as the concentration of a pollutant high enough at a discharge point to cause death in less than 96 hours. Surface water 2A and 2B CS and MS for certain metals are dependent on the water hardness and must be calculated for specific sites.

Another common class of waters in road side ditches, depending on their location, is Class 7 waters. This class may be more relevant with respect to the types of materials used in road construction.

#### *Great Lakes Initiative Wildlife Values for the Lake Superior Watershed (GLI)*

Waters that can contribute to the water quality of the Lake Superior basin are included in the GLI. These standards are included with the Minnesota Surface Water Quality Standards (MSWQS) and include mercury, PCBs, 2, 3, 7, 8-tetrachlorodibenzo-p-dioxin, and DDT, because these are highly bio-accumulative contaminants (Gnabasik and White, 2006).

#### **pH, TCLP and SPLP testing**

Samples of the aggregate materials collected for this study were submitted to Northeast Technical Service (NTS; now Pace Analytical) for chemical analyses including pH, Toxicity Characteristics Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP) by NRRI. Chemical analysis parameters include: arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver (RCRA metals) as well as cobalt.

#### *pH*

Each aggregate sample was analyzed for its natural pH by NTS. Results of the analyses show that for the geologic materials the pH ranged from 6.62 to 8.6. Table 15 provides the pH for each of the aggregate samples.



**Table 15. pH results for aggregate samples.**

| Description | Sample ID      | pH   |
|-------------|----------------|------|
| River Sand  | E1             | 8.2  |
| Granite     | E4             | 8.3  |
| Sand        | E7             | 8.4  |
| Dirty Sand  | E8             | 8.1  |
| Fine Sand   | E10            | 8.4  |
| Granite     | E11            | 8.5  |
| Granite     | E12            | 8.1  |
| Gravel      | W6             | 8.6  |
| Shingles    | Shingles       | 7.8  |
| Limestone   | M-LS           | 8.5  |
| Limestone   | G 3/8 BA LS    | 8.1  |
| 2010        | Arcelor-Mittal | 6.62 |
| 2010        | Minntac        | 7.51 |

*TCLP results*

The TCLP test method was designed to provide worst case results when evaluating the potential leaching of metals and non-metals in a mixed media landfill setting, and is used to identify hazardous wastes under 40CFR Part 261. TCLP uses a buffered and mildly acidic solution with a pH of 4.9.

Water standard reference values and TCLP analytical results are presented below in Tables 16 and 17, respectively. For comparison, results from chemical analyses of tailings samples completed in 2008 are included. Minnesota drinking water standards are listed across the top of Table 16, while EPA water standards are highlighted in gray in the last three rows.

Barium, cobalt, and lead were detected in the samples above the reporting limits for the TCLP analyses. Lead was detected only once, in granite sample E-11, which produced a result of 53.1 µg/L. For lead, EPA’s drinking water standard requires action at a level of 15 µg/L, and the Recommended Water Quality-Chronic Standard (RWQ-C) is 2.5 µg/L (Table 16). Leachate produced by the TCLP method for the granite sample exceeds both of these standards.

Results for barium and cobalt are presented in the graphs below (Figs. 8 and 9). The Minnesota drinking water standard (1B) and EPA Recommended Water Quality Chronic Standard (RWQ-C) are included on each graph for ease of reference. None of the aggregate samples produce levels of barium above the published standards. A drinking water level for cobalt has not been established, and the EPA also does not have a RWQ-C for cobalt. When cobalt results are compared to Minnesota’s 2A chronic surface water standard of 2.8 µg/L, taconite tailings exceed this value, as does E4 (granite) and E8 (dirty sand).

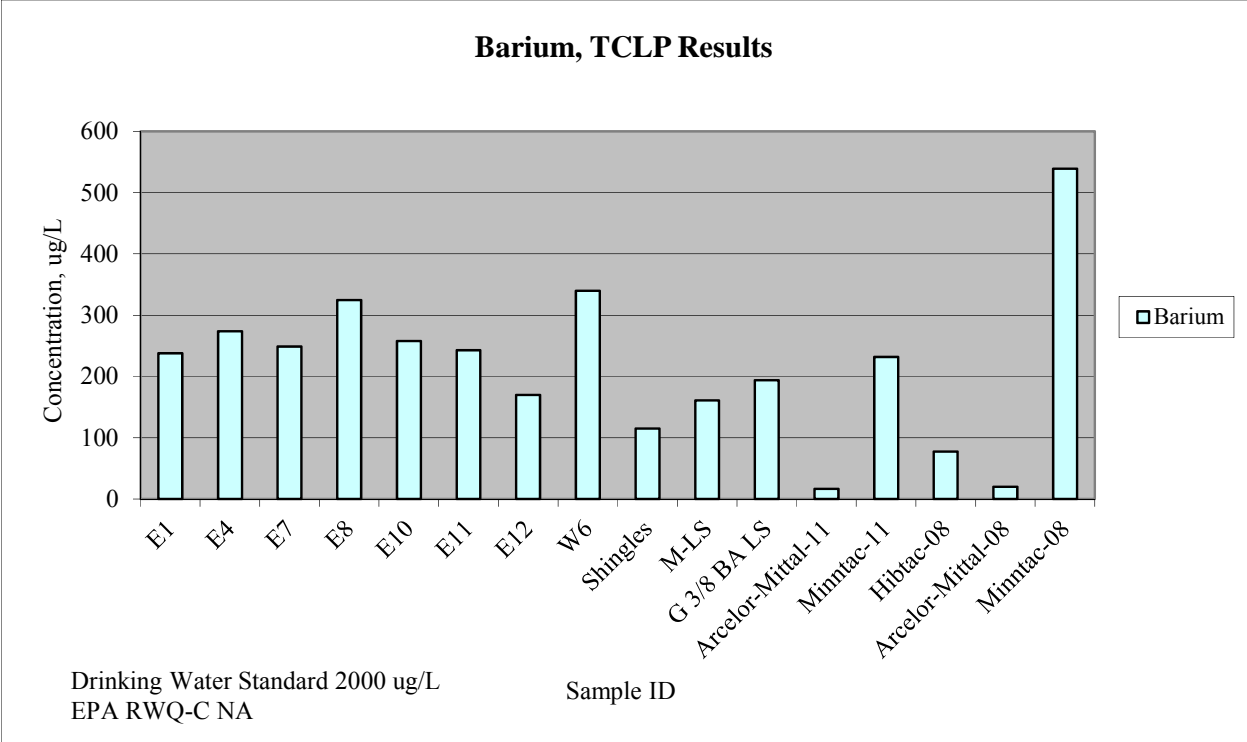
**Table 16. Water standard references for trace metals.**

| INORGANICS -- TRACE METALS               |                 | Metal            | Arsenic | Barium | Cadmium                              | Chromium, total | Cobalt | Lead                                 | Mercury (total) | Selenium | Silver                               |
|--|-----------------|------------------|---------|--------|--------------------------------------|-----------------|--------|--------------------------------------|-----------------|----------|--------------------------------------|
| Water Class:                             |                 | Units            | ug/L    | ug/L   | ug/L                                 | ug/L            | ug/L   | ug/L                                 | ug/L            | ug/L     | ug/L                                 |
| <b>1B</b>                                | <b>DRINKING</b> | <b>WATER (b)</b> | 50 (i)  | 2000   | 5.00                                 | 100             |        |                                      | 2               | 50       | 100(s)                               |
| <b>Minnesota Surface Water Standards</b> |                 |                  |         |        |                                      |                 |        |                                      |                 |          |                                      |
| <b>2A</b>                                | <b>CHRONIC</b>  | <b>(CS)</b>      | 2.0     |        | Hardness Dependent. See worksheet 1. |                 | 2.8    | Hardness Dependent. See worksheet 1. | 0.0069          | 5.0      | 0.12                                 |
| <b>2A</b>                                | <b>MAXIMUM</b>  | <b>(MS)</b>      | 360     |        |                                      |                 | 436    |                                      | 2.4*            | 20       | Hardness Dependent. See worksheet 1. |
| <b>2B, C &amp; D</b>                     | <b>CHRONIC</b>  | <b>(CS)</b>      | 53      |        |                                      |                 | 5      | Hardness Dependent. See worksheet 1. | 0.0069          | 5.0      | 1                                    |
| <b>2B, C &amp; D</b>                     | <b>MAXIMUM</b>  | <b>(MS)</b>      | 360     |        |                                      |                 | 436    |                                      | 2.4*            | 20       | Hardness Dependent. See worksheet 1. |
| <b>GLI</b>                               | <b>WILDLIFE</b> | <b>VALUE (e)</b> |         |        |                                      |                 |        |                                      | 0.0013          |          |                                      |
| <b>TIER II SECONDARY</b>                 | <b>CHRONIC</b>  | <b>VALUE (g)</b> |         | 4      |                                      |                 |        |                                      |                 |          |                                      |
| <b>NPDWS - EPA</b>                       |                 |                  | 10      | 2000   | 5                                    | 100             |        | 15 (action level)                    | 2               | 50       |                                      |
| <b>NRWQ - EPA</b>                        | <b>Acute</b>    |                  | 340     | na     | 2                                    | 16 (Cr VI)      |        | 65                                   | 1.4             |          | 3.2                                  |
| <b>NRWQ - EPA</b>                        | <b>Chronic</b>  |                  | 150     | na     | 0.25                                 | 11 (Cr VI)      |        | 2.5                                  | 0.77            | 5        | 1.9                                  |

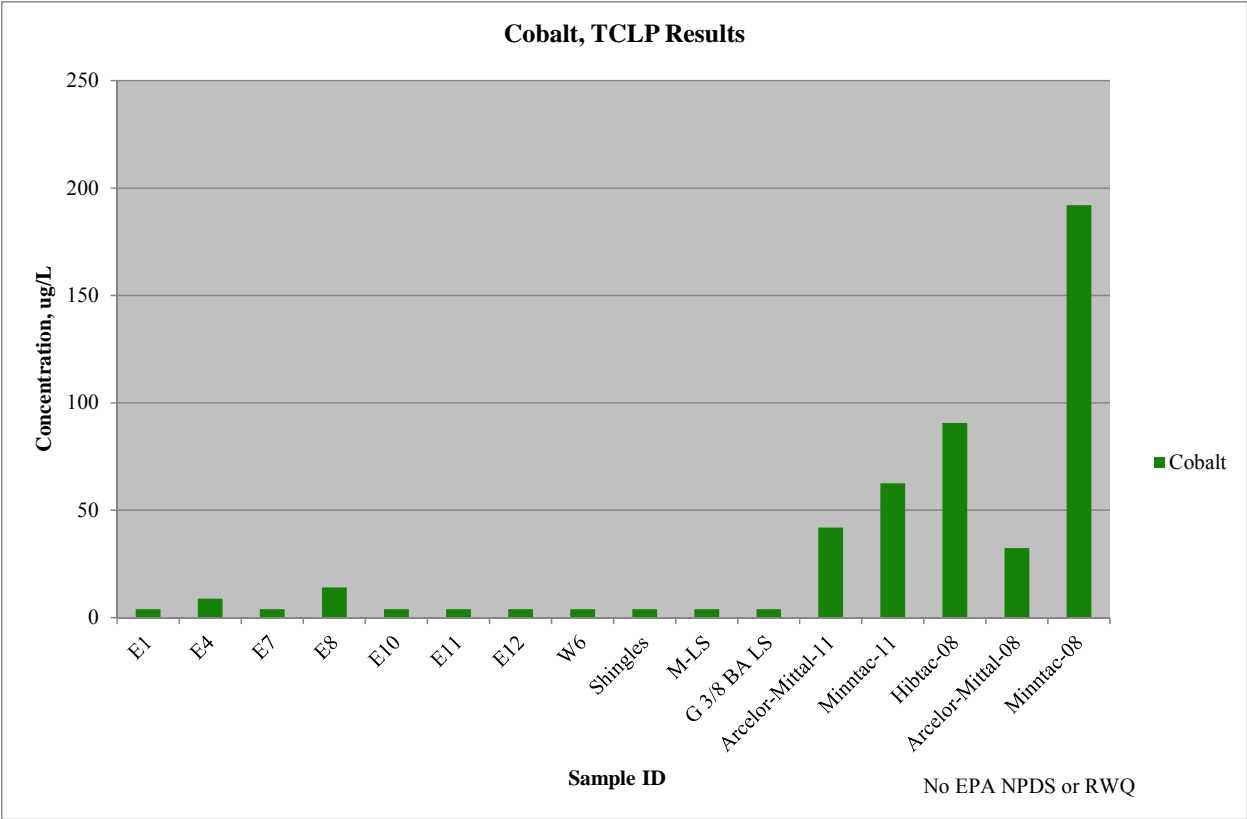
Table 17. TCLP results.

| DESCRIPTION | SAMPLE ID      | TEST | Arsenic | Barium      | Cadmium | Chromium, total | Cobalt      | Lead        | Mercury (total) | Selenium | Silver |
|-------------|----------------|------|---------|-------------|---------|-----------------|-------------|-------------|-----------------|----------|--------|
| River Sand  | E1             | TCLP | <10     | <b>238</b>  | <20     | <8              | <8          | <40         | <0.2            | <10      | <20    |
| Granite     | E4             | TCLP | <10     | <b>274</b>  | <20     | <8              | <b>8.89</b> | <40         | <0.2            | <10      | <20    |
| Sand        | E7             | TCLP | <10     | <b>249</b>  | <20     | <8              | <8          | <40         | <0.2            | <10      | <20    |
| Dirty Sand  | E8             | TCLP | <10     | <b>325</b>  | <20     | <8              | <b>14</b>   | <40         | <0.2            | <10      | <20    |
| Fine Sand   | E10            | TCLP | <10     | <b>258</b>  | <20     | <8              | <8          | <40         | <0.2            | <10      | <20    |
| Granite     | E11            | TCLP | <10     | <b>243</b>  | <20     | <8              | <8          | <b>53.1</b> | <0.2            | <10      | <20    |
| Granite     | E12            | TCLP | <10     | <b>170</b>  | <20     | <8              | <8          | <40         | <0.2            | <10      | <20    |
| Gravel      | W6             | TCLP | <10     | <b>340</b>  | <20     | <8              | <8          | <40         | <0.2            | <10      | <20    |
| Shingles    | Shingles       | TCLP | <10     | <b>115</b>  | <20     | <8              | <8          | <40         | <0.2            | <10      | <20    |
| Limestone   | M-LS           | TCLP | <10     | <b>161</b>  | <20     | <8              | <8          | <40         | <0.2            | <10      | <20    |
| Limestone   | G 3/8 BA LS    | TCLP | <10     | <b>194</b>  | <20     | <8              | <8          | <40         | <0.2            | <10      | <20    |
| 2011        | Arcelor-Mittal | TCLP | <5      | <b>16.4</b> | <1      | <8              | <b>41.9</b> | <2.5        | <0.2            | <5       | <1     |
| 2011        | Minntac        | TCLP | <5      | <b>232</b>  | <1      | <8              | <b>62.5</b> | <2.5        | <0.2            | <5       | <1     |
| 2008        | Hibtac         | TCLP | <10     | <b>77.6</b> | <20     | <8              | <b>90.6</b> | <40         | <0.2            | <10      | <20    |
| 2008        | Arcelor-Mittal | TCLP | <10     | <40         | <20     | <8              | <b>32.4</b> | <40         | <0.2            | <10      | <20    |
| 2008        | Minntac        | TCLP | <10     | <b>539</b>  | <20     | <8              | <b>192</b>  | <40         | <0.2            | <10      | <20    |
|             |                |      |         |             |         |                 |             |             |                 |          |        |

Values above the reporting limit are presented in bold; all values are reported as µg/L (ppb).



**Figure 8. TCLP barium results.**



**Figure 9. TCLP cobalt results.**

### *SPLP results*

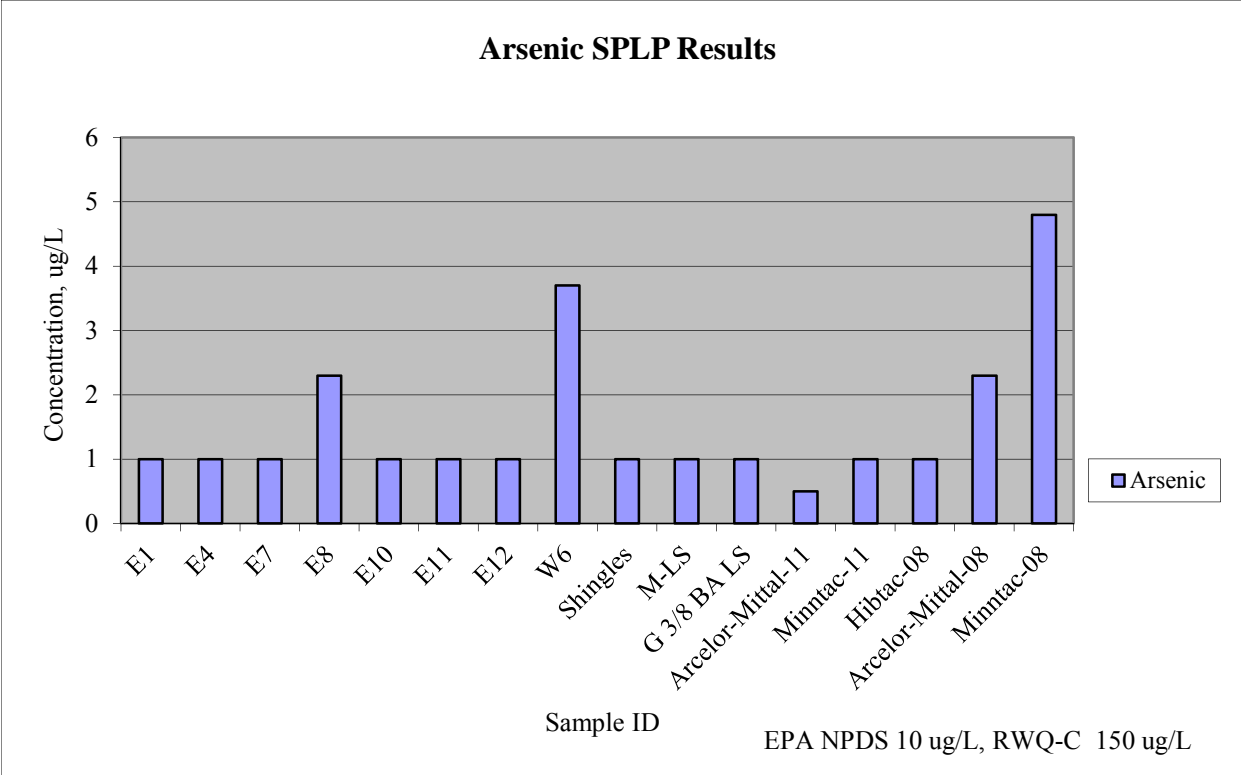
SPLP test methods were designed to simulate and evaluate the leaching potential for metals into groundwater and surface water by infiltration of rain water. The analysis uses geographic location (east or west of the Mississippi River) to determine the specific pH for the extraction. The SPLP solution is not buffered and is more acidic than in the TCLP test. For locations west of the Mississippi, a pH 5.0 extraction fluid is used; for locations east of the Mississippi and for mine wastes, the SPLP procedure mandates that the more acidic pH 4.2 extraction fluid be used (Hageman, et al., 2000). For this study, SPLP analyses were conducted at pH 4.2 and at a liquid/solid (L/S) ratio of 20:1.

Arsenic, barium, chromium, cobalt, and lead were detected by SPLP analysis (Table 18). Again, Table 16 can be referenced for trace metal water quality standards. Arsenic was detected in samples E8, W6, and the 2008 tailings samples from Arcelor-Mittal and Minntac. None of the results exceed the EPA water standards (Fig. 10); however, they do exceed the Minnesota Surface Water standard of 2 µg/L for class 2A-CS waters. Samples E8, Minntac 11, and Minntac-08 reported barium concentrations between 5.92 µg/L and 84 µg/L, well below the drinking water standard of 2000 µg/L. Total chromium detections were 3.71 µg/L in shingles, and 6.43 µg/L and 13.3µg/L in samples W6, and E8, respectively, with E8 (Dirty Sand) exceeding the acute RWQS for chromium VI (Fig. 11). Seven leachate samples detected cobalt above the reporting limit, with values ranging between 0.28 µg/L and 5.94 µg/L. Minnesota has surface water standards for cobalt, but no published standards are available from the EPA (Fig. 12); two samples, E8 and W6, exceeded the 2A, 2B, 2C, and 2D chronic standard. Lead was present above the reporting limits in six samples, E1, E4, E8, E11, W6, and shingles; none exceeded the EPA National Primary Drinking Standard (NPDS) action level of 15 µg/L (Fig. 13).

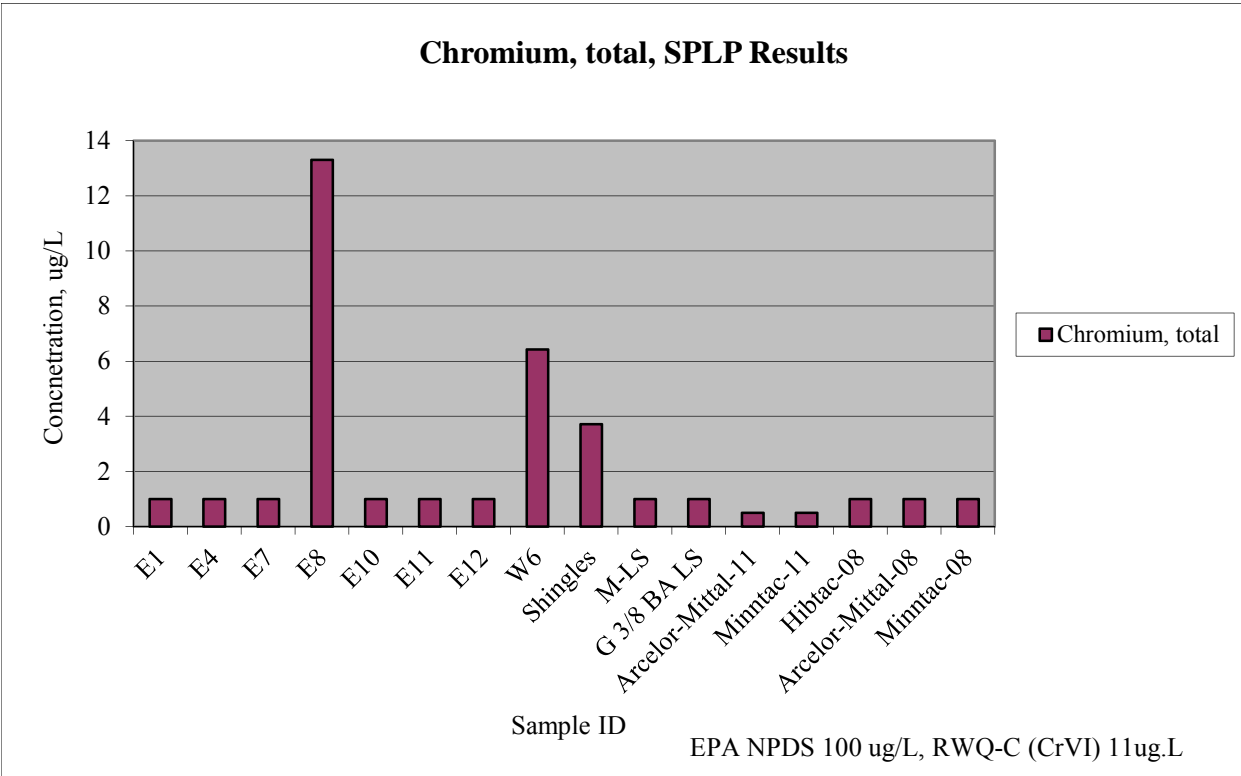
Table 18. SPLP results.

| DESCRIPTION | SAMPLE ID      | TEST  | Arsenic    | Barium      | Cadmium | Chromium, total | Cobalt                      | Lead        | Mercury (total) | Selenium | Silver |
|-------------|----------------|-------|------------|-------------|---------|-----------------|-----------------------------|-------------|-----------------|----------|--------|
|             |                | Units | µg/L       | µg/L        | µg/L    | µg/L            | µg/L                        | µg/L        | µg/L            | µg/L     | µg/L   |
| River Sand  | E1             | SPLP  | <2         | <70         | <0.2    | <2              | <b>0.9</b>                  | <b>0.69</b> | <0.2            | <1       | <0.2   |
| Granite     | E4             | SPLP  | <2         | <70         | <0.2    | <2              | <b>0.36</b>                 | <b>2</b>    | <0.2            | <1       | <0.2   |
| Sand        | E7             | SPLP  | <2         | <70         | <0.2    | <2              | <b>0.28</b>                 | <0.5        | <0.2            | <1       | <0.2   |
| Dirty Sand  | E8             | SPLP  | <b>2.3</b> | <b>84</b>   | <0.2    | <b>13.3</b>     | <b>4.3</b><br><b>(5.94)</b> | <b>3.8</b>  | <0.2            | <1       | <0.2   |
| Fine Sand   | E10            | SPLP  | <2         | <70         | <0.2    | <2              | <b>0.4</b>                  | <0.5        | <0.2            | <1       | <0.2   |
| Granite     | E11            | SPLP  | <2         | <70         | <0.2    | <2              | <0.2                        | <b>0.72</b> | <0.2            | <1       | <0.2   |
| Granite     | E12            | SPLP  | <2         | <70         | <0.2    | <2              | <0.2                        | <0.5        | <0.2            | <1       | <0.2   |
| Gravel      | W6             | SPLP  | <b>3.7</b> | <70         | <0.2    | <b>6.43</b>     | <b>2.2</b><br><b>(3.17)</b> | <b>2.5</b>  | <0.2            | <1       | <0.2   |
| Shingles    | Shingles       | SPLP  | <2         | <70         | <0.2    | <b>3.71</b>     | <b>0.51</b>                 | <b>1.4</b>  | <0.2            | <1       | <0.2   |
| Limestone   | M-LS           | SPLP  | <2         | <70         | <0.2    | <2              | <0.2                        | <0.5        | <0.2            | <1       | <0.2   |
| Limestone   | G 3/8 BA LS    | SPLP  | <2         | <70         | <0.2    | <2              | <0.2                        | <0.5        | <0.2            | <1       | <0.2   |
| 2011        | Arcelor-Mittal | SPLP  | <1         | <1          | <0.2    | <1              | <2                          | <0.5        | <0.2            | <1       | <0.2   |
| 2011        | Minntac        | SPLP  | <2         | <b>5.92</b> | <0.2    | <1              | <2                          | <0.5        | <0.2            | <1       | <0.2   |
| 2008        | Hibtac         | SPLP  | <2         | <10         | <0.2    | <2              | <2                          | <1          | <0.2            | <2       | <1     |
| 2008        | Arcelor-Mittal | SPLP  | <b>2.3</b> | <10         | <0.2    | <2              | <2                          | <1          | <0.2            | <2       | <1     |
| 2008        | Minntac        | SPLP  | <b>4.8</b> | <b>10.8</b> | <0.2    | <2              | <2                          | <1          | <0.2            | <2       | <1     |

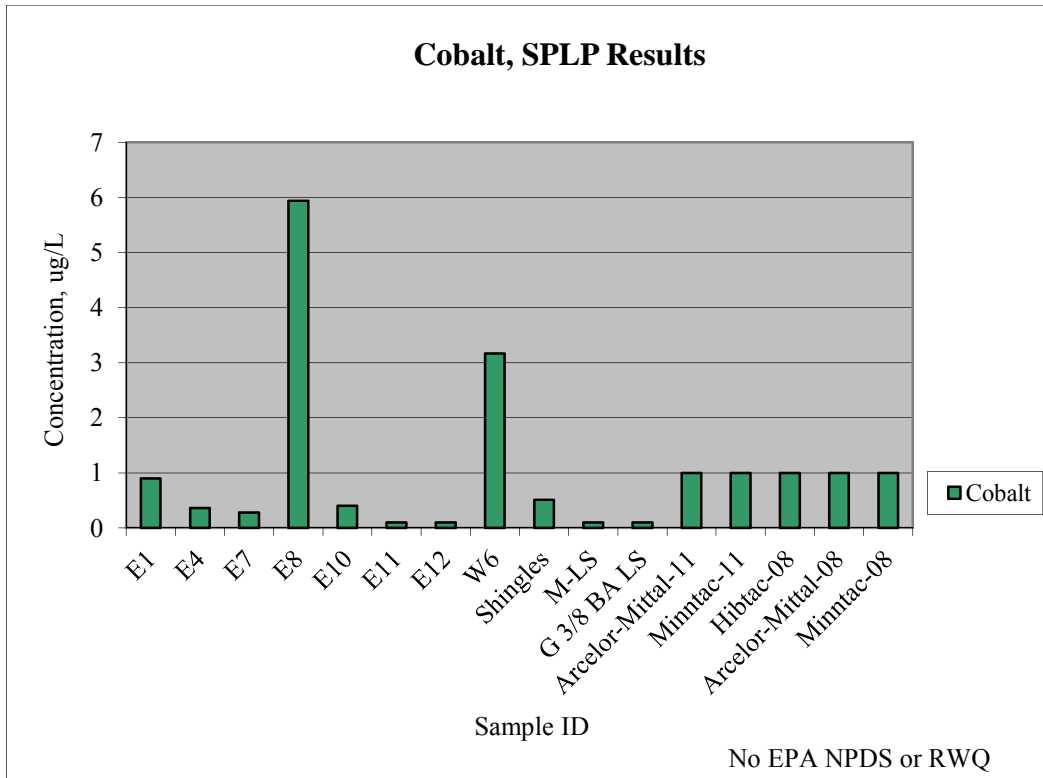
Values above the reporting (detection) limit are presented in bold; all values are reported as µg/L (ppb)



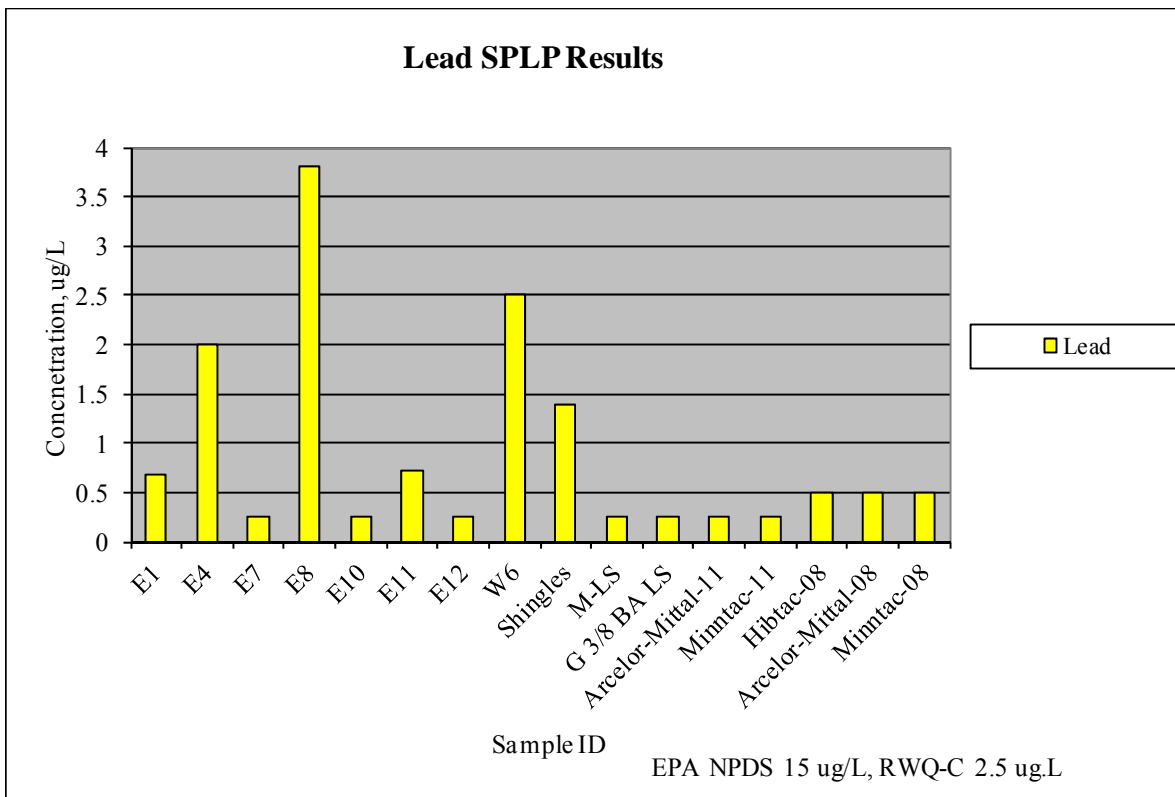
**Figure 10. SPLP arsenic results.**



**Figure 11. SPLP chromium results.**



**Figure 12. SPLP cobalt results.**



**Figure 13. SPLP lead results.**



## **Liquid/Solid (L/S) ratio and variable pH leachate testing**

The purpose of this phase of testing was to produce leachate for each sample at different Liquid/Solid (L/S) ratios and variable pH. Five tests were conducted to determine what types of metals leach out of specific types of aggregates. Three of the tests utilized different L/S ratios (0.5:1, 2:1, and 10:1) to simulate different moisture conditions. For the other two tests, leachate was produced by varying the pH (to pH's of 3.5 and 9.0) of the liquid added to the aggregate to simulate extreme environmental conditions, and holding the L/S ratio constant at 10:1.

Six aggregate samples, including two taconite tailings samples, were selected for leachate testing using Kosson's variable pH (A.2.SR002.1, Solubility and Release as a function of pH) and variable Liquid/Soil ratio (SR003.1, Solubility and Release as a Function of L/S Ratio) testing (Kosson, 2002). Reasoning used to select the non-taconite tailings samples included: highest values by Toxicity Characteristics Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP), and XRF data obtained during testing of chemical parameters. The following six samples were used for leachate testing:

- W6 – Gravel;
- E8 – Dirty Sand;
- E12 – Granite;
- M-LS Limestone;
- ArcelorMittal (Minorca) taconite tailings; and
- U.S. Steel Minntac taconite tailings.

### *Procedures*

To produce the 400mL of liquid needed for analysis, aggregate samples were first screened on a No. 10 (2mm) sieve and then processed through a sample splitter to make more manageable laboratory-sized samples, with the <2mm fraction used for experimentation. Each prepared sample was weighed to within +/- 0.005g accuracy on an analytical balance to correspond to the target liquid-to-solid (L/S) ratio and placed in multiple 55mL vials, to which the appropriate volume of distilled/deionized water was added (Fig. 14). Prior testing showed that the moisture content of the aggregates was not significant enough to factor into determining the amount of liquid to add to each sample. Instead, the amount of distilled/deionized water added was based upon a volumetric scale to achieve the desired L/S ratio. The sample-containing vials were then rotated end over end for 48 hours at 28rpm +/-, as described by Kosson et al. (2002), using the tumbler apparatus shown in Figure 15.

For the variable pH tests, the pH was changed by measuring out 4.00 grams of aggregate and adding 30.0 mL of deionized water. The sample was then tested for its natural pH by rotating it end over end for 20 minutes, allowing it to settle for 5 minutes, and then measuring the pH. Alterations were then made by adding a 1N solution of nitric acid (HNO<sub>3</sub>), one drop at a time, and repeating the agitation cycle. The pH was again tested. This was repeated until the desired pH of 3.5 was achieved or reasonably approached. The remaining 10.0 ml or DI water was added, the sample was sent through another agitation cycle, and the pH was again measured. Even with the addition of water, the sample pH did not significantly change (+/- 0.1 pH). A similar procedure was used for creating the higher pH (9.0) solution by using 1N and 0.25N potassium hydroxide (KOH); the lower strength solution was used on higher pH aggregates.

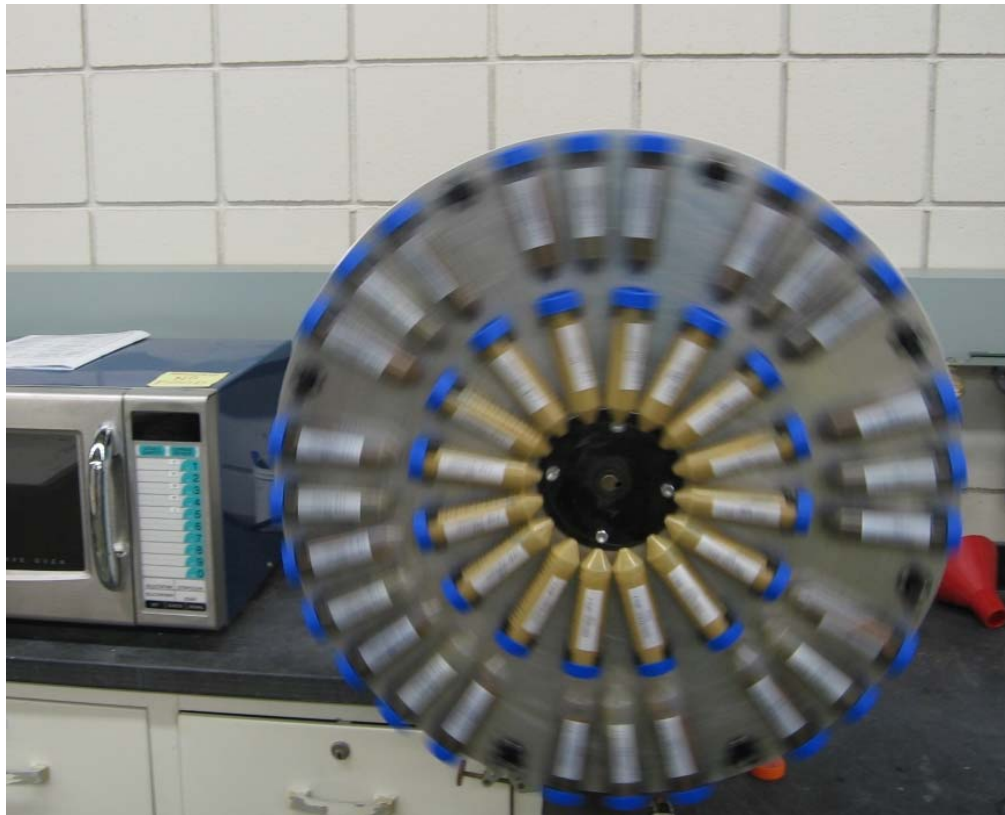


**Figure 14. 55mL vials prepared for leachate tests.**



**Figure 15. Tumbler apparatus used for 48-hour leachate production.**

Up to 14 sample vials can be fitted to the tumbler shown in Figure 15. Based on the number of project samples and the total liquid volume needed for chemical analysis, it was estimated that it would take an unacceptable length of time and effort (several weeks) to generate the required leachate. The NRRI investigators then worked with the NRRI's machine shop to fabricate a much larger tumbler apparatus (Fig. 16). The new tumbler could accommodate up to 48 vials on each side (96, total). This configuration allowed NRRI to produce leachate much more efficiently.



**Figure 16. Re-designed large capacity tumbler apparatus.**

For both the L/S ratio and variable pH tests, the liquids and solids were separated by gravity filtration and centrifugation at NRRI. Once prepared in this fashion, leachate samples were refrigerated at 34°F until a representative from Pace Analytical retrieved them. Because only preliminary filtration was performed, preservatives were not added to the samples. Instead, the samples were simply put into a cooler with a bag full of ice. Final filtration, through a P5 (5 micrometer) Fisher brand filter, and preservation were provided by Pace Analytical. Resource Conservation and Recovery Act (RCRA) Metals analyses, plus iron and manganese, were then completed on the 19 prepared samples submitted to Pace.

Table 20 (Variable L/S ratio) and Table 21 (Variable pH) summarize the results of the chemical analyses performed on the aggregate leachate, with Minnesota water standards located across the top of each table (highlighted in yellow) and EPA (national) standards highlighted in gray. Sample IDs and corresponding analytical laboratory codes (Lab#) are listed for each test condition. Analytical results presented in **bold** indicate detection above the laboratory's reporting limit. Numbers in **bold italics** indicate that the sample leachate exceeded at least one of the water quality standards.

**Table 19. Variable liquid/solid (L/S) ratio results.**

| INORGANICS -- TRACE METALS - Variable Liquid Solid Ratio Testing |                |             |           | Metal     | Arsenic     | Barium      | Cadmium | Chromium, total | Cobalt      | Lead              | Mercury (total) | Selenium    | Silver | Iron         | Manganese   |
|--|----------------|-------------|-----------|-----------|-------------|-------------|---------|-----------------|-------------|-------------------|-----------------|-------------|--------|--------------|-------------|
| Water Class:   |                |             |           | Units     | ug/L        | ug/L        | ug/L    | ug/L            | ug/L        | ug/L              | ug/L            | ug/L        | ug/L   | ug/L         | ug/L        |
| 1B   | DRINKING       |             |           | WATER (b) | 50 (i)      | 2000        | 5.00    | 100             |             |                   | 2               | 50          | 100(s) | 300 (s)      | 50 (s)      |
| 2A   | CHRONIC        |             |           | (CS)      | 2.0         |             |         |                 | 2.8         |                   | 0.0069          | 5.0         | 0.12   |              |             |
| 2A   | MAXIMUM        |             |           | (MS)      | 360         |             |         |                 | 436         |                   | 2.4*            | 20          |        |              |             |
| 2B, C & D  | CHRONIC        |             |           | (CS)      | 53          |             |         |                 | 5           |                   | 0.0069          | 5.0         | 1      |              |             |
| 2B, C & D  | MAXIMUM        |             |           | (MS)      | 360         |             |         |                 | 436         |                   | 2.4*            | 20          |        |              |             |
| GLI  | WILDLIFE       |             |           | VALUE(e)  |             |             |         |                 |             |                   | 0.0013          |             |        |              |             |
| SECONDARY  | CHRONIC        |             |           | VALUE(g)  |             | 4           |         |                 |             |                   |                 |             |        |              |             |
| National Primary Drinking Standards                              |                |             |           |           | 10          | 2000        | 5       | 100             |             | 15 (action level) | 2               | 50          |        | na           | na          |
| Recommended Water Quality Standards                              | Acute          |             |           |           | 340         | na          | 2       | 16 (Cr VI)      |             | 65                | 1.4             |             | 3.2    | na           | na          |
| Recommended Water Quality Standards                              | Chronic        |             |           |           | 150         | na          | 0.25    | 11 (Cr VI)      |             | 2.5               | 0.77            | 5           | 1.9    | na           | na          |
| DESCRIPTION  | SAMPLE ID      | Lab #       | L/S ratio | TEST      | Arsenic     | Barium      | Cadmium | Chromium, total | Cobalt      | Lead              | Mercury (total) | Selenium    | Silver | Iron         | Manganese   |
|  |                |             |           | Units     | ug/L        | ug/L        | ug/L    | ug/L            | ug/L        | ug/L              | ug/L            | ug/L        | ug/L   | ug/L         | ug/L        |
| Dirty Sand   | E8             | WDD05       | 10:1      | metals    | <b>3.84</b> | <b>79.4</b> | <0.2    | <b>14.8</b>     | <b>8.28</b> | <b>5.88</b>       | <0.2            | <1          | <0.2   | <b>14300</b> | <b>318</b>  |
| Granite  | E12            | WDD04       | 10:1      | metals    | <b>1.18</b> | <10         | <0.2    | <2              | <2          | <b>2.03</b>       | <0.2            | <1          | <0.2   | <b>695</b>   | <b>28.3</b> |
| Gravel   | W6             | WDD02       | 10:1      | metals    | <b>3.97</b> | <b>32.4</b> | <0.2    | <b>4.01</b>     | <b>2.06</b> | <b>2.45</b>       | <0.2            | <1          | <0.2   | <b>4320</b>  | <b>138</b>  |
| Gravel   | W6             | WDD02.2     | 10:1      | metals    | <0.5        | <10         | <0.2    | <2              | <2          | <0.5              | <0.2            | <1          | <0.2   | <50          | <10         |
| Limestone  | M-LS           | WDD03       | 10:1      | metals    | <2          | <10         | <0.2    | <2              | <2          | <2                | <0.2            | <1          | <0.2   | <50          | <10         |
| Tailings   | Arcelor-Mittal | WDD08       | 10:1      | metals    | <b>2.62</b> | <10         | <0.2    | <2              | <2          | <0.5              | <0.2            | <1          | <0.2   | <50          | <b>28.1</b> |
| Tailings   | MINNTAC        | WDD09       | 10:1      | metals    | <b>1.42</b> | <10         | <0.2    | <2              | <2          | <0.5              | <0.2            | <1          | <0.2   | <50          | <b>39.8</b> |
| Gravel   | W6             | WDD06       | 2:1       | metals    | <b>6.4</b>  | <b>32</b>   | <0.2    | <b>4.74</b>     | <2          | <b>2.03</b>       | <0.2            | <1          | <0.2   | <b>3620</b>  | <b>148</b>  |
| Limestone  | M-LS           | WDD07       | 2:1       | metals    | <b>0.53</b> | <b>271</b>  | <0.2    | <b>11.7</b>     | <2          | <b>0.72</b>       | <b>0.33</b>     | <1          | <0.2   | <b>68.3</b>  | <10         |
| Dirty Sand   | E8             | WDD10       | 2:1       | metals    | <b>1.02</b> | <b>11.6</b> | <0.2    | <2              | <2          | <0.5              | <0.2            | <1          | <0.2   | <b>349</b>   | <b>11.5</b> |
| Tailings   | Arcelor-Mittal | WDD11       | 2:1       | metals    | <b>3.63</b> | <10         | <0.2    | <2              | <2          | <0.5              | <0.2            | <1          | <0.2   | <b>80.1</b>  | <b>79.2</b> |
| Tailings   | MINNTAC        | WDD12 MNNTC | 2:1       | metals    | <b>1.99</b> | <b>13.9</b> | <0.2    | <2              | <2          | <0.5              | <0.2            | <b>1.46</b> | <0.2   | <50          | <b>30.8</b> |
| Granite  | E12            | WDD13E12    | 2:1       | metals    | <b>1.57</b> | <10         | <0.2    | <2              | <2          | <0.5              | <0.2            | <1          | <0.2   | <50          | <b>10.5</b> |
| Granite  | E12            | WDD14E12    | 0.5:1     | metals    | <b>1.19</b> | <b>16.1</b> | <0.2    | <2              | <2          | <b>1.16</b>       | <0.2            | <b>1.13</b> | <0.2   | <50          | <b>27.2</b> |
| Dirty Sand   | E8             | WDD15       | 0.5:1     | metals    | <b>1.07</b> | <b>26.4</b> | <0.2    | <2              | <2          | <0.5              | <0.2            | <1          | <0.2   | <50          | <10         |
| Gravel   | W6             | WDD16W6     | 0.5:1     | metals    | <b>4.05</b> | <b>17.8</b> | <0.2    | <b>4.41</b>     | <2          | <0.5              | <0.2            | <1          | <0.2   | <b>153</b>   | <10         |
| Limestone  | MLS            | WDD17MLS    | 0.5:1     | metals    | <b>1.61</b> | <b>15.8</b> | <0.2    | <b>4.48</b>     | <2          | <0.5              | <0.2            | <b>4.49</b> | <0.2   | <50          | <b>14.7</b> |
| Tailings   | Arcelor-Mittal | WDD18ARCR   | 0.5:1     | metals    | <b>4.74</b> | <10         | <0.2    | <2              | <2          | <0.5              | <0.2            | <b>1.38</b> | <0.2   | <50          | <b>64.4</b> |
| Tailings   | MINNTAC        | WDD19MINNTC | 0.5:1     | metals    | <b>2.78</b> | <b>31.1</b> | <0.2    | <2              | <2          | <0.5              | <0.2            | <b>7.46</b> | <0.2   | <50          | <b>208</b>  |

Bold = detected above reporting limit

**Table 20. Variable pH results.**

| INORGANICS -- TRACE METALS- Variable pH Testing |                |                 |     | Metal     | Arsenic     | Barium      | Cadmium     | Chromium, total | Cobalt      | Lead              | Mercury (total) | Selenium    | Silver | Iron         | Manganese    |      |
|---|----------------|-----------------|-----|-----------|-------------|-------------|-------------|-----------------|-------------|-------------------|-----------------|-------------|--------|--------------|--------------|------|
| Water Class:                                    |                |                 |     | Units     | ug/L        | ug/L        | ug/L        | ug/L            | ug/L        | ug/L              | ug/L            | ug/L        | ug/L   | ug/L         | ug/L         | ug/L |
| 1B  | DRINKING       |                 |     | WATER (b) | 50 (i)      | 2000        | 5.00        | 100             |             |                   | 2               | 50          | 100(s) | 300 (s)      | 50 (s)       |      |
| 2A  | CHRONIC        |                 |     | (CS)      | 2.0         |             |             |                 | 2.8         |                   | 0.0069          | 5.0         | 0.12   |              |              |      |
| 2A  | MAXIMUM        |                 |     | (MS)      | 360         |             |             |                 | 436         |                   | 2.4*            | 20          |        |              |              |      |
| 2B, C & D                                       | CHRONIC        |                 |     | (CS)      | 53          |             |             |                 | 5           |                   | 0.0069          | 5.0         | 1      |              |              |      |
| 2B, C & D                                       | MAXIMUM        |                 |     | (MS)      | 360         |             |             |                 | 436         |                   | 2.4*            | 20          |        |              |              |      |
| GLI   | WILDLIFE       |                 |     | VALUE(e)  |             |             |             |                 |             |                   | 0.0013          |             |        |              |              |      |
| National Primary Drinking Standards             |                |                 |     |           | 10          | 2000        | 5           | 100             |             | 15 (action level) | 2               | 50          |        | na           | na           |      |
| Recommended Water Quality Standards             | Acute          |                 |     |           | 340         | na          | 2           | 16 (Cr VI)      |             | 65                | 1.4             |             | 3.2    | na           | na           |      |
| Recommended Water Quality Standards             | Chronic        |                 |     |           | 150         | na          | 0.25        | 11 (Cr VI)      |             | 2.5               | 0.77            | 5           | 1.9    | na           | na           |      |
| DESCRIPTION                                     | SAMPLE ID      | Lab #           | pH  | TEST      | Arsenic     | Barium      | Cadmium     | Chromium, total | Cobalt      | Lead              | Mercury (total) | Selenium    | Silver | Iron         | Manganese    | pH   |
|   |                |                 |     | Units     | ug/L        | ug/L        | ug/L        | ug/L            | ug/L        | ug/L              | ug/L            | ug/L        | ug/L   | ug/L         | ug/L         | ug/L |
| Granite   | E12            | WDD20-E12-3.5   | 3.5 | metals    | <1          | <b>224</b>  | <b>1.23</b> | <2              | <b>18</b>   | <0.5              | <0.2            | <1          | <0.2   | <b>19000</b> | <b>3580</b>  | 4.7  |
| Dirty Sand                                      | E8             | WDD21-E8-3.5    | 3.5 | metals    | <1          | <b>246</b>  | <b>0.58</b> | <2              | <b>24.1</b> | <0.5              | <0.2            | <1          | <0.2   | <50          | <b>2680</b>  | 5.5  |
| Gravel  | W6             | WDD22-W6-3.5    | 3.5 | metals    | <1          | <b>1490</b> | <b>2.31</b> | <b>25.4</b>     | <b>60.5</b> | <b>10.8</b>       | <0.2            | <b>5.05</b> | <0.2   | <b>7840</b>  | <b>7120</b>  | 2.1  |
| Limestone                                       | MLS            | WDD23-MLS-3.5   | 3.5 | metals    | <10         | <b>997</b>  | <b>42.4</b> | <100            | <100        | <10               | <0.2            | <20         | <4     | <2500        | <b>48200</b> | 6.5  |
| blank   |                | WDD24-BLNK      | 3.5 | metals    | <1          | <10         | <0.2        | <2              | <2          | <0.5              | <0.2            | <1          | <0.2   | <50          | <10          | 5.7  |
| Tailings  | Arcelor-Mittal | WDD25-ARCIR-3.5 | 3.5 | metals    | <1          | <10         | <0.2        | <2              | <b>16.8</b> | <0.5              | <0.2            | <1          | <0.2   | <50          | <b>7180</b>  | 7.2  |
| Tailings  | MINNTAC        | WDD26-MNNTC-3.5 | 3.5 | metals    | <1          | <b>64.8</b> | <0.2        | <2              | <b>6.41</b> | <0.5              | <0.2            | <1          | <0.2   | <50          | <b>3260</b>  | 7.7  |
| Tailings  | MINNTAC        | WDD27-MNNTC-9   | 9   | metals    | 2.27        | <b>11.5</b> | <0.2        | <2              | <2          | <0.5              | <0.2            | <1          | <0.2   | <50          | <b>28.2</b>  | 7.9  |
| Granite   | E12            | WDD28-E12-9     | 9   | metals    | <b>1.84</b> | <10         | <0.2        | <2              | <2          | <b>0.63</b>       | <0.2            | <1          | <0.2   | <b>176</b>   | <10          | 7.3  |
| Dirty Sand                                      | E8             | WDD29-E8-9      | 9   | metals    | 2.7         | <b>40.2</b> | <0.2        | <b>6.43</b>     | <b>2.88</b> | <b>2.66</b>       | <0.2            | <1          | <0.2   | <b>5580</b>  | <b>150</b>   | 6.4  |
| Gravel  | W6             | WDD30W6-9.0     | 9   | metals    | 3.52        | <b>38.3</b> | <0.2        | <b>3.57</b>     | <2          | 0.74              | 0.21            | <1          | <0.2   | <b>1300</b>  | <b>57</b>    | 6.6  |
| Limestone                                       | MLS            | WDD31MLS-9.0    | 9   | metals    | <1          | <10         | <0.2        | <2              | <2          | <0.5              | <0.2            | <1          | <0.2   | <50          | <10          | 9.3  |
| Tailings  | Arcelor-Mittal | WDD32ARCIR-9.0  | 9   | metals    | <b>9.14</b> | <10         | <0.2        | <2              | <2          | <b>0.56</b>       | <0.2            | <1          | <0.2   | <b>547</b>   | <b>75.6</b>  | 7.5  |
| Bold = detected above reporting limit           |                |                 |     |           |             |             |             |                 |             |                   |                 |             |        |              |              |      |

## Aggregate leachate results compared to Water Quality Standards

To better assess the various leachate test results for each of the aggregate types analyzed, maximum and minimum results are compiled in Table 21 and compared to the national (gray columns) and Minnesota 2A (green column) standards. A review of these data indicates that the granite aggregate (E12) produced leachate with the least number of RCRA metals detected above water quality standards, while gravel (W6) produced the most. Arsenic, cobalt, and selenium were detected in the two taconite tailings samples, and some analyses exceeded Minnesota Surface Water 2A chronic Standards. However, when only national standards are considered (Table 22), samples of limestone (M-LS), gravel (W6), granite (E12), and dirty sand (E8) exceed the most restrictive (chronic) standard for the metals detected. In comparison, just one taconite tailings sample (Minntac) exceeded the RWQS-chronic standard, and only for selenium.

**Table 21. Maximum and minimum metal values for each tested aggregate, compared to national and Minnesota water standards.**

| Analyte         | Sample      | Minntac           | Arcelor-Mittal    | M-LS      | W6      | E12     | E8         | National Primary Drinking Standards | Recommended Water Quality Standards | Recommended Water Quality Standards | MN Surface Water 2A Standard |
|-----------------|-------------|-------------------|-------------------|-----------|---------|---------|------------|-------------------------------------|-------------------------------------|-------------------------------------|------------------------------|
|                 | Description | Taconite Tailings | Taconite Tailings | Limestone | Gravel  | Granite | Dirty Sand |                                     | Acute                               | Chronic                             | Chronic                      |
| Arsenic         | Min         | 1.42              | 2.62              | 0.53      | 3.52    | 1.18    | 1.02       | 10                                  | 340                                 | 150                                 | 2.0                          |
|                 | Max         | 2.78              | 9.14              | 1.61      | 6.40    | 1.84    | 2.70       |                                     |                                     |                                     |                              |
| Barium          | Min         | 5.92              | 16.4              | 15.80     | 17.80   | 16.10   | 11.60      | 2000                                | na                                  | na                                  |                              |
|                 | Max         | 232               | 16.4              | 997.00    | 1490.00 | 224.00  | 325.00     |                                     |                                     |                                     |                              |
| Cadmium         | Min         | brl               | brl               | 42.40     | 2.31    | 1.23    | 0.58       | 5                                   | 2                                   | 0.25                                |                              |
|                 | Max         | brl               | brl               | 42.40     | 2.31    | 1.23    | 0.58       |                                     |                                     |                                     |                              |
| Chromium, total | Min         | brl               | brl               | 4.48      | 3.57    | brl     | 6.43       | 100                                 | 16 (Cr VI)                          | 11 (Cr VI)                          |                              |
|                 | Max         | brl               | brl               | 11.70     | 25.40   | brl     | 13.30      |                                     |                                     |                                     |                              |
| Cobalt          | Min         | 6.41              | 16.8              | brl       | 2.06    | 18.00   | 2.88       |                                     |                                     |                                     | 2.8                          |
|                 | Max         | 6.41              | 41.9              | brl       | 60.50   | 18.00   | 14.00      |                                     |                                     |                                     |                              |
| Lead            | Min         | brl               | 0.26              | 0.72      | 0.74    | 0.63    | 2.66       | 15 (action level)                   | 65                                  | 2.5                                 |                              |
|                 | Max         | brl               | 0.56              | 0.72      | 10.80   | 2.03    | 3.80       |                                     |                                     |                                     |                              |
| Mercury (total) | Min         | brl               | brl               | brl       | 0.21    | brl     | brl        | 2                                   | 1.4                                 | 0.77                                | 0.0069                       |
|                 | Max         | brl               | brl               | brl       | 0.21    | brl     | brl        |                                     |                                     |                                     |                              |
| Selenium        | Min         | 1.46              | brl               | brl       | 5.05    | 1.13    | brl        | 50                                  |                                     | 5                                   | 5.0                          |
|                 | Max         | 7.46              | brl               | brl       | 5.05    | 1.13    | brl        |                                     |                                     |                                     |                              |
| Silver          | Min         | brl               | brl               | brl       | brl     | brl     | brl        |                                     | 3.2                                 | 1.9                                 | 0.12                         |
|                 | Max         | brl               | brl               | brl       | brl     | brl     | brl        |                                     |                                     |                                     |                              |

brl - below reporting limit

**Table 22. Maximum and minimum metal values for each tested aggregate, compared to national water standards only.**

| Analyte         | Sample      | Minntac           | Arcelor-Mittal    | M-LS      | W6      | E12     | E8         | National Primary Drinking Standards | Recommended Water Quality Standards | Recommended Water Quality Standards |
|-----------------|-------------|-------------------|-------------------|-----------|---------|---------|------------|-------------------------------------|-------------------------------------|-------------------------------------|
|                 | Description | Taconite Tailings | Taconite Tailings | Limestone | Gravel  | Granite | Dirty Sand |                                     | Acute                               | Chronic                             |
| Arsenic         | Min         | 1.42              | 2.62              | 0.53      | 3.52    | 1.18    | 1.02       | 10                                  | 340                                 | 150                                 |
|                 | Max         | 2.78              | 9.14              | 1.61      | 6.40    | 1.84    | 2.7        |                                     |                                     |                                     |
| Barium          | Min         | 5.92              | 16.4              | 15.80     | 17.80   | 16.10   | 11.6       | 2000                                | na                                  | na                                  |
|                 | Max         | 232               | 16.4              | 997.00    | 1490.00 | 224.00  | 325        |                                     |                                     |                                     |
| Cadmium         | Min         | brl               | brl               | 42.40     | 2.31    | 1.23    | 0.58       | 5                                   | 2                                   | 0.25                                |
|                 | Max         | brl               | brl               | 42.40     | 2.31    | 1.23    | 0.58       |                                     |                                     |                                     |
| Chromium, total | Min         | brl               | brl               | 4.48      | 3.57    | brl     | 6.43       | 100                                 | 16 (Cr VI)                          | 11 (Cr VI)                          |
|                 | Max         | brl               | brl               | 11.70     | 25.40   | brl     | 13.30      |                                     |                                     |                                     |
| Cobalt          | Min         | 6.41              | 16.8              | brl       | 2.06    | 18.00   | 2.88       | na                                  | na                                  | na                                  |
|                 | Max         | 6.41              | 41.9              | brl       | 60.50   | 18.00   | 14         |                                     |                                     |                                     |
| Lead            | Min         | brl               | 0.26              | 0.72      | 0.74    | 0.63    | 2.66       | 15 (action level)                   | 65                                  | 2.5                                 |
|                 | Max         | brl               | 0.56              | 0.72      | 10.80   | 2.03    | 3.8        |                                     |                                     |                                     |
| Mercury (total) | Min         | brl               | brl               | brl       | 0.21    | brl     | brl        | 2                                   | 1.4                                 | 0.77                                |
|                 | Max         | brl               | brl               | 0.33      | 0.21    | brl     | brl        |                                     |                                     |                                     |
| Selenium        | Min         | 1.46              | brl               | brl       | 5.05    | 1.13    | brl        | 50                                  |                                     | 5                                   |
|                 | Max         | 7.46              | brl               | brl       | 5.05    | 1.13    | brl        |                                     |                                     |                                     |
| Silver          | Min         | brl               | brl               | brl       | brl     | brl     | brl        |                                     | 3.2                                 | 1.9                                 |
|                 | Max         | brl               | brl               | brl       | brl     | brl     | brl        |                                     |                                     |                                     |

brl - below reporting limit

## Change in leachate chemistry due to variable pH and L/S ratios

The ensuing discussion summarizes the impact of variable pH and different L/S ratios on leachate chemistry results, by aggregate type, for selected RCRA metals. X-ray fluorescence (XRF) results for corresponding aggregates and RCRA metals (presented previously in Table 13) are presented again in Table 23 for convenience. Variable pH was run at two different L/S ratios due to methodology, i.e., traditional leachate testing and Kosson method testing. For example, TCLP and SPLP samples were each run at a 20:1 L/S ratio, and at pH 4.9 (TCLP) and pH 4.2 (SPLP); Kosson variable pH (pH 3.5 and 9) samples were run at a liquid/solid ratio of 10:1 (Kosson, 2002).

NOTE: For samples that returned leachate chemistry values below the analytical reporting limit, one half of that reporting limit value was used for graphing purposes in the following presentation of results.

**Table 23. XRF results: RCRA metals.**

| Description       | Sample #       | Material             | Cr     | As     | Se    | Ag    | Cd    | Ba     | Hg    | Pb     |
|-------------------|----------------|----------------------|--------|--------|-------|-------|-------|--------|-------|--------|
| martin-marietta   | E4             | Sand                 | 41.80  | -1.00  | -1.00 | 43.00 | -1.00 | 196.60 | 8.50  | 24.00  |
| sand              | E1             | Sand                 | 38.80  | -1.00  | -1.00 | -1.00 | -1.00 | 118.00 | -1.00 | 8.50   |
| sand, ba vonco    | E7             | Sand                 | 27.00  | -1.00  | -1.00 | 22.00 | -1.00 | 86.00  | 8.00  | 8.00   |
| sand, loken       | E8             | Sand                 | 68.25  | 8.00   | -1.00 | -1.00 | -1.00 | 193.75 | 9.00  | 15.00  |
| sand-fine-barton  | E10            | Sand                 | 58.83  | -1.00  | -1.00 | 29.00 | -1.00 | 154.50 | 8.00  | 10.00  |
| gravel, class 3   | W6             | Gravel               | 42.25  | 6.00   | -1.00 | -1.00 | -1.00 | 124.25 | 9.00  | 17.00  |
| shingles          | Shingles       | Shingles             | 57.40  | 11.00  | -1.00 | -1.00 | -1.00 | 90.60  | -1.00 | 153.00 |
| limestone         | M-LS           | Limestone            | 112.00 | 7.50   | -1.00 | 25.50 | -1.00 | 88.00  | 7.00  | -1.00  |
| ST. CLOUD         | E11            | Granite              | 88.50  | 7.00   | -1.00 | -1.00 | -1.00 | 278.33 | 9.00  | 19.00  |
| ST. CLOUD, CLEAR  | E12            | Granite              | 62.29  | 9.00   | -1.00 | 35.50 | -1.00 | 272.86 | 9.50  | 14.86  |
| limestone         | G 3/8 BA<br>LS | Limestone            | 20.60  | -1.00  | -1.00 | 30.00 | -1.00 | -1.00  | 7.00  | -1.00  |
| taconite tailings | MT             | Taconite<br>Tailings | -1.00  | 118.50 | -1.00 | -1.00 | -1.00 | -1.00  | 17.50 | 17.50  |
| taconite tailings | AM             | Taconite<br>Tailings | -1.00  | 16.00  | -1.00 | -1.00 | -1.00 | -1.00  | -1.00 | 26.50  |

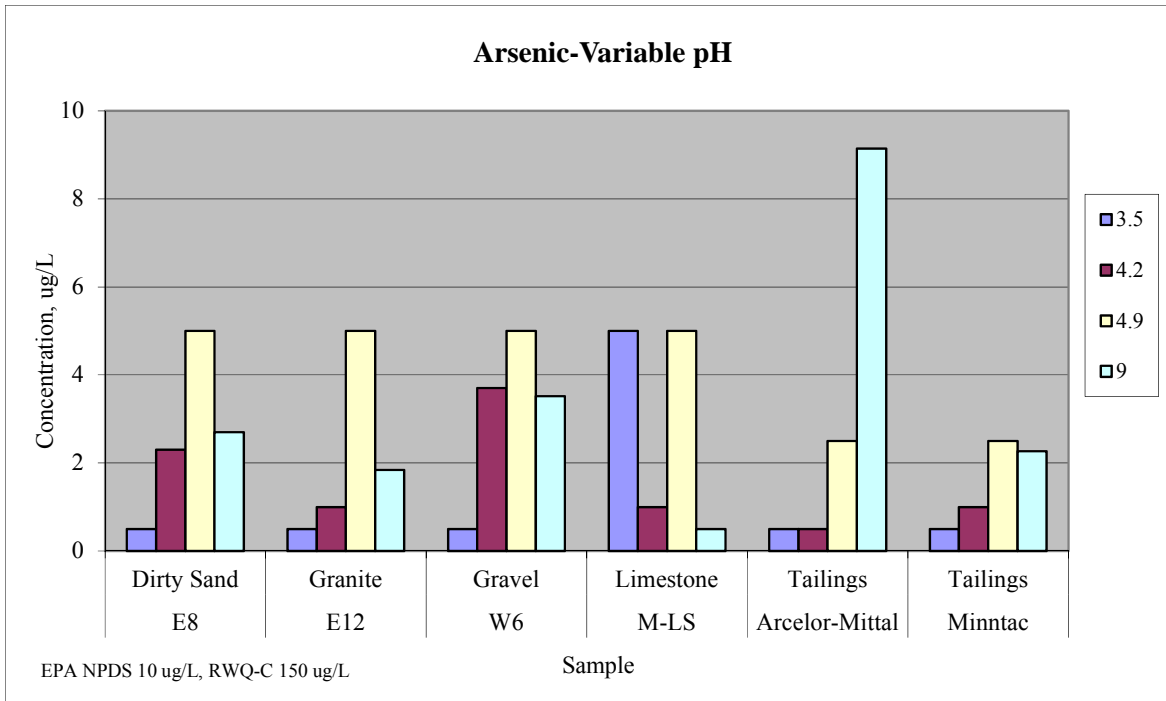
If the average contained a majority of <LOD in the results, then the average was recorded as "-1"

### *Arsenic relative to variable pH and L/S ratios*

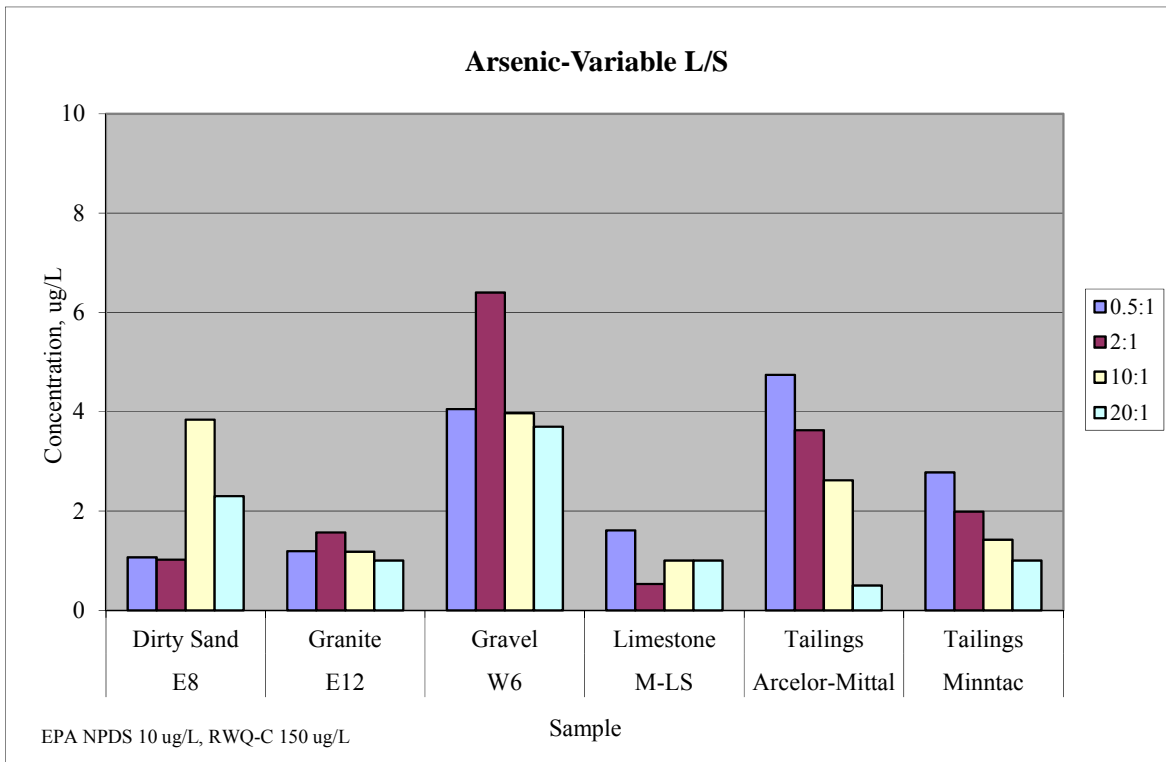
Arsenic was detected in each of the samples by XRF and ranged from 6 ppm (W6, Gravel) to 118.5 ppm in the Minntac tailings. The XRF results for the ArcelorMittal sample returned 16 ppm arsenic.

As Figure 17 shows, the concentration of arsenic tends to increase with an increase in alkalinity, with the exception of M-LS (limestone). The highest arsenic result was produced by taconite tailings from Arcelor-Mittal at a pH of 9. However, none of the leachate results exceeded the National Primary Drinking Standard (NPDS) for arsenic of 10 µg/L.

The concentration of arsenic in the variable L/S ratio samples decreased in the taconite tailings as the liquid content increased (Fig. 18). Granite and gravel also initially increased, but decreased in the 10:1 and 20:1 samples. The highest arsenic content was derived from the gravel sample at a L/S ratio of 2:1. Variable L/S ratio results do not exceed national standards for arsenic.



**Figure 17. Arsenic results for variable pH levels, by aggregate type.**



**Figure 18. Arsenic results for variable L/S ratios, by aggregate type.**

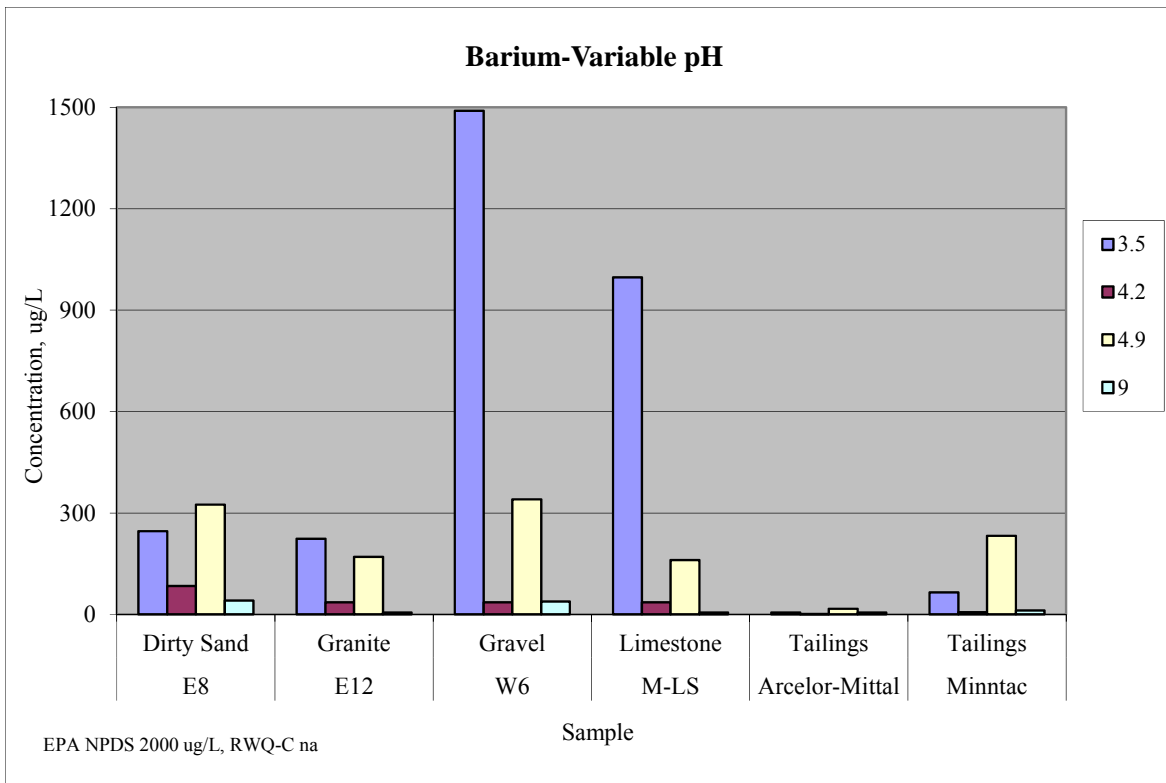


*Barium relative to variable pH and L/S ratios*

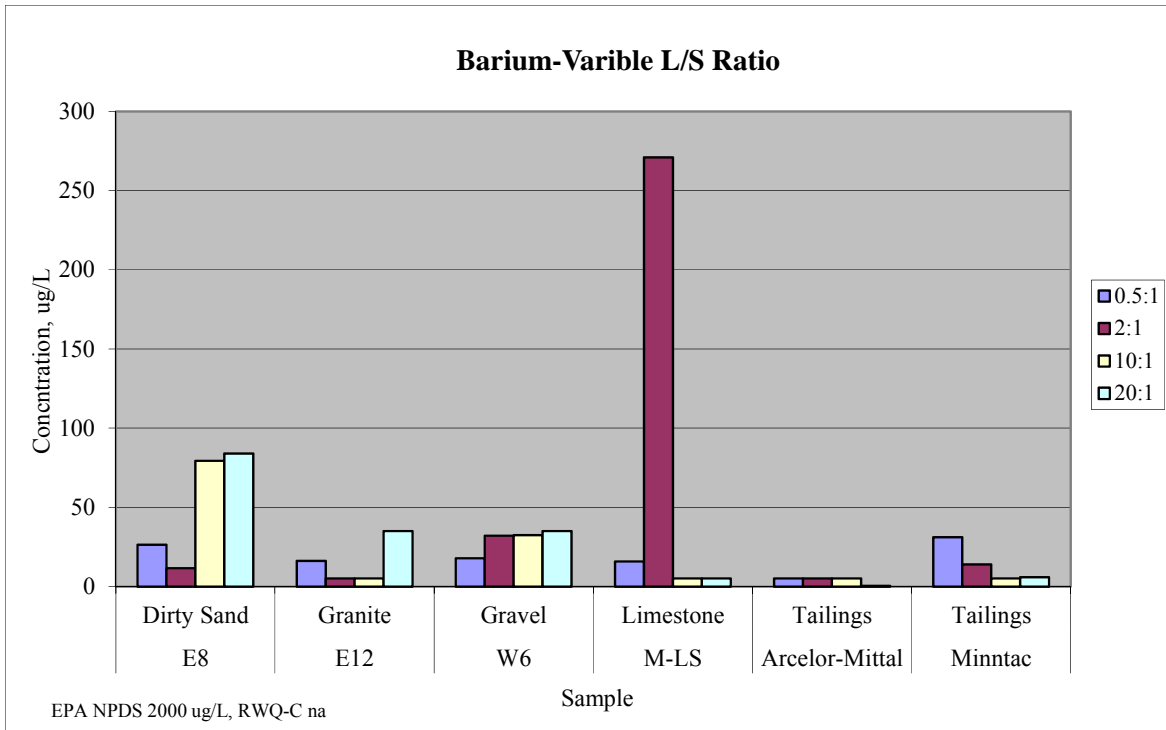
Detection of barium by XRF ranged from 88 ppm to 273 ppm for the six variable pH and L/S ratio samples.

Release of barium was higher in acidic conditions, with highest results for the gravel (W6) and limestone (M-LS) samples (Fig. 19). However, none of the results exceed the NPDS of 2000 µg/L.

For variable L/S ratios, release of barium from the tailings samples was less than 50 µg/L and well below drinking water standards. Limestone produced a result of 271 µg/L (the highest result) for the samples run at an L/S ratio of 2:1 (Fig. 20). However, this value appears to be an anomaly.



**Figure 19. Barium results for variable pH levels, by aggregate type.**



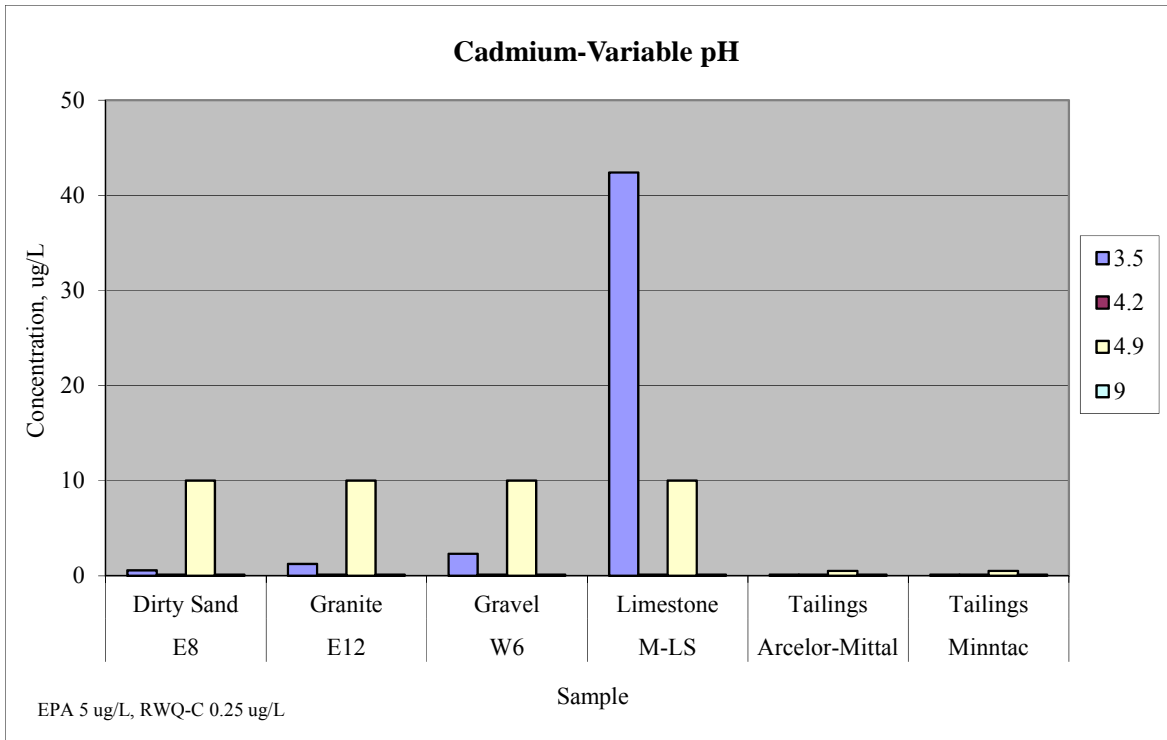
**Figure 20. Barium results for variable L/S ratios, by aggregate type.**

*Cadmium relative to variable pH and L/S ratios*

XRF analysis results did not record cadmium concentrations above the limit of detection for any of the samples (see Table 23).

Limestone (M-LS) tested at a pH of 3.5 produced the highest leachate concentration of cadmium, relative to the other aggregates. The detection of 10 µg/L in four of the pH 4.9 samples is a reflection of the detection limit (Fig. 21).

Cadmium was not detected above the reporting detection limit in the L/S ratio samples, and is therefore not graphed.

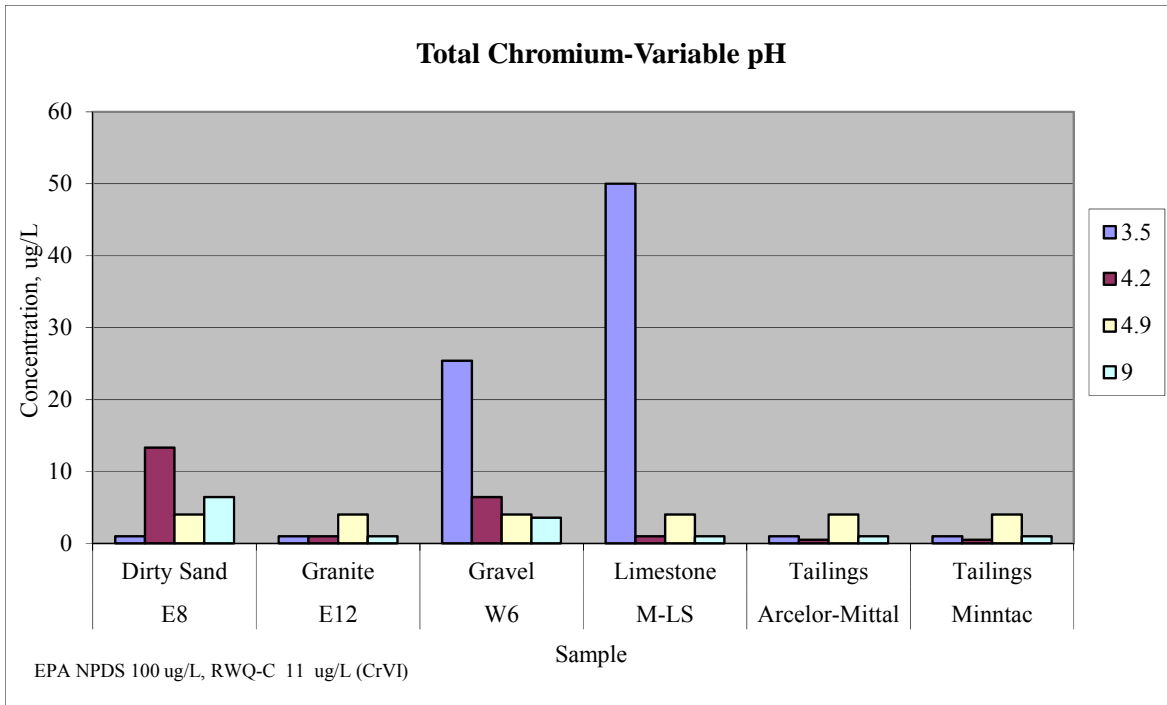


**Figure 21. Cadmium results for variable pH levels, by aggregate type.**

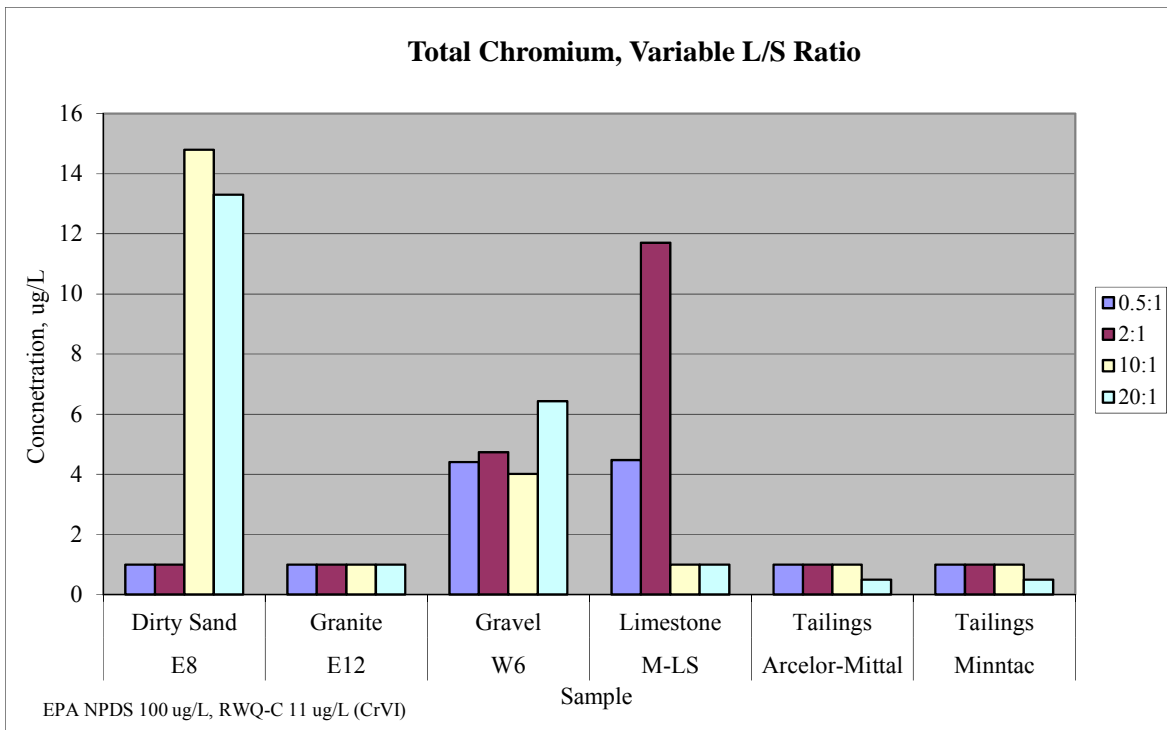
*Chromium relative to variable pH and L/S ratios*

Chromium values ranged between 42.25 ppm (W6, gravel) to 112 ppm (M-LS, limestone) by XRF analysis.

In the leachate produced by variable pH experiments on W6 and M-LS, chromium levels appear to decrease as the pH increases (Fig. 22). Highest concentrations of total chromium are also recorded in these samples. Varying the L/S ratio produced a spike in the total chromium detected in the limestone sample at 2:1 and then a drop to the reporting limit at higher ratios (Fig. 23). Dirty sand (E8) had the highest total chromium concentration in the 10:1 and 20:1 L/S ratio samples.



**Figure 22. Total chromium results for variable pH levels, by aggregate type.**



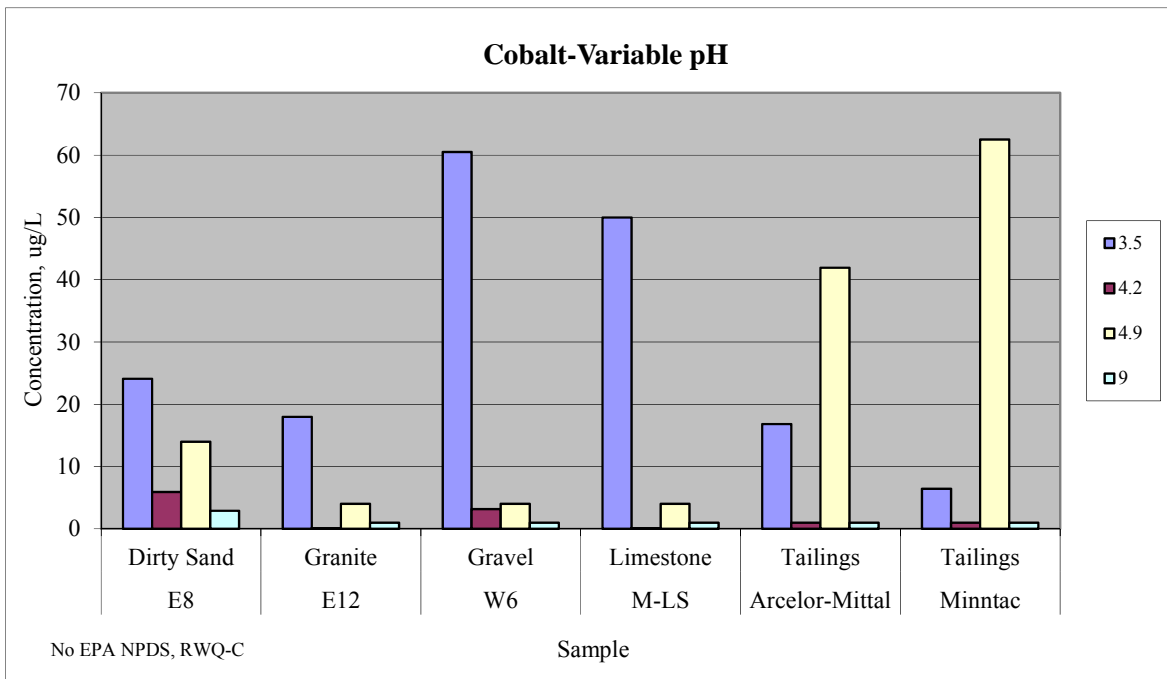
**Figure 23. Total chromium results for variable L/S ratios, by aggregate type.**

*Cobalt relative to variable pH and L/S ratios*

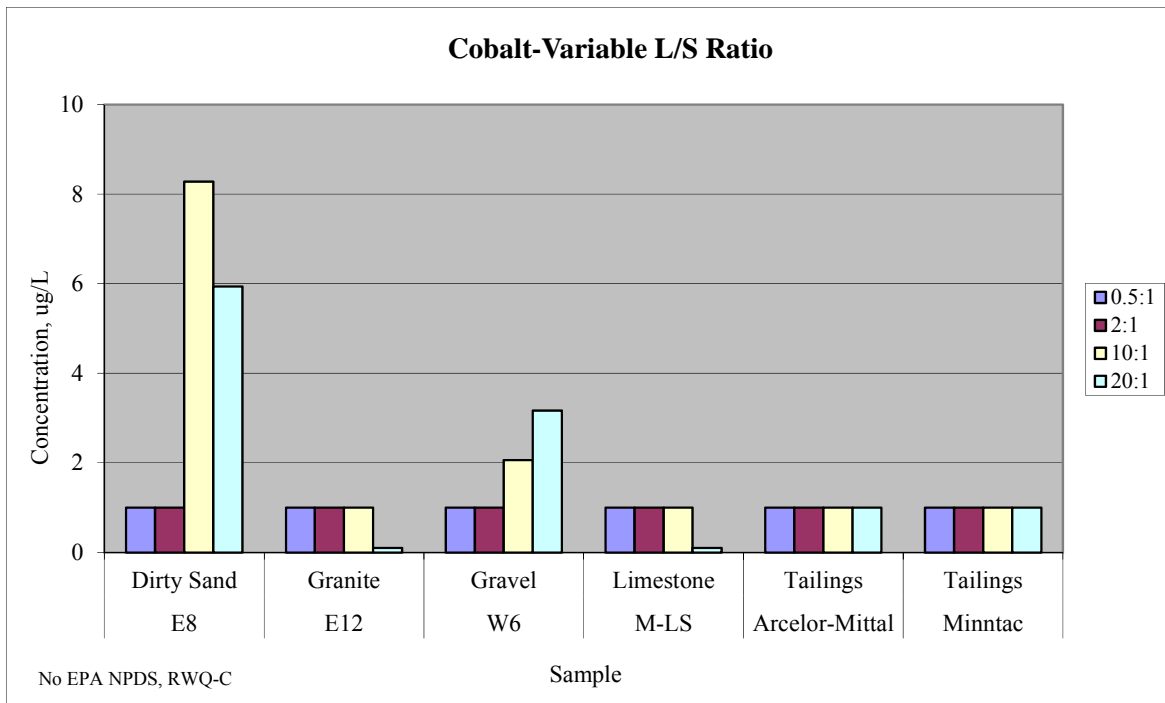
XRF cobalt results ranged from 31.50 to 129.00 ppm.

Variable pH total cobalt levels ranged from below the reporting limit to 62.5 µg/L. Release of cobalt increased at a pH of 4.9 for the tailings samples (Fig. 24). The remaining non-taconite aggregate samples had the highest cobalt results at a pH of 3.5.

The majority of the variable L/S ratio cobalt results were below the reporting limit (Fig. 25). Leachate analysis results for Dirty Sand (E8) and Gravel (W6) samples were above the reporting limit for L/S ratios of 10:1 and 20:1.



**Figure 24. Cobalt results for variable pH levels, by aggregate type.**



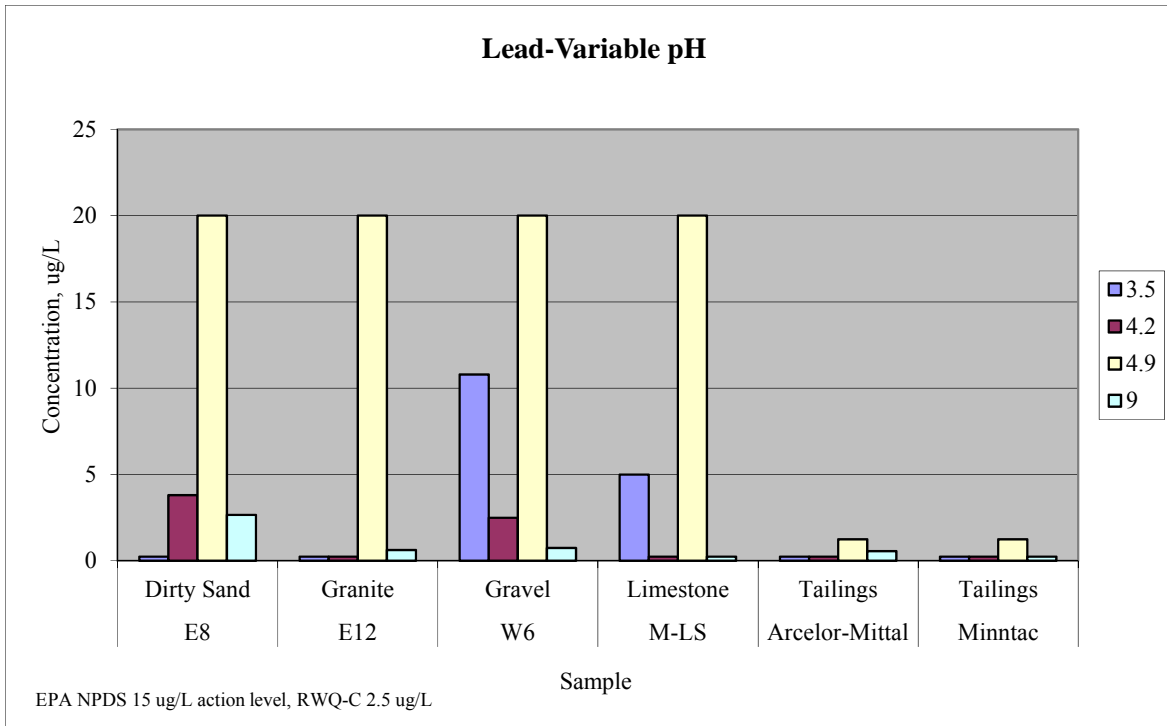
**Figure 25. Cobalt results for variable L/S ratios, by aggregate type.**

*Lead relative to variable pH and L/S ratios*

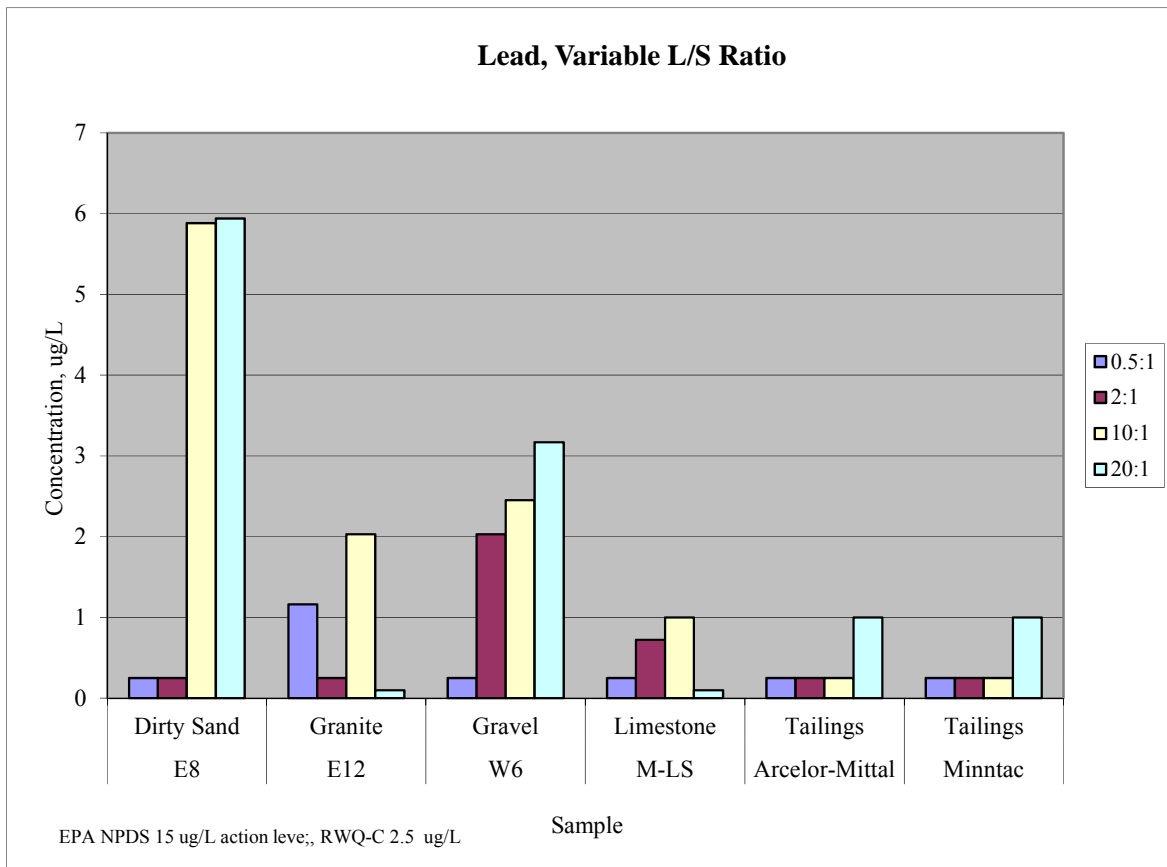
XRF results for lead ranged from below limit of detection to 17.00 ppm.

Variable pH analyses produced lead results above the reporting limit, between 0.56 µg/L and 10.8 µg/L (Fig. 26). The 20 µg/L values reported for pH 4.9 analyses are a consequence of a high laboratory reporting limit for SPLP, and should be viewed accordingly.

Variable L/S ratios returned lead values from 0.72 µg/L to 5.88 µg/L (Fig. 27).



**Figure 26. Lead results for variable pH levels, by aggregate type.**



**Figure 27. Lead results for variable L/S ratios, by aggregate type.**

**Evaluation of HMA Mix Design Sample Leachate**

Three mix design samples created by MnDOT were selected for SPLP analysis. The intent was to compare potential metal release of aggregates coated with asphalt binder to that of aggregates in the unbound state.

The tested samples were unmolded and still pliable. Approximately 400 g of samples TM11-001, TM11-002, and TM11-005 were submitted to Pace Analytical for SPLP RCRA metals analysis. The non-asphalt components of the three mix design samples are shown in Table 24.

**Table 24. Composition of SPLP-tested asphalt mix designs.**

| <u>4.75 Design</u>                  | <u>Mix ID</u> | <u>Fine RAP %</u> | <u>Mineral Filler %</u> | <u>Sand %</u> | <u>Taconite %</u> | <u>Granite %</u> |
|-------------------------------------|---------------|-------------------|-------------------------|---------------|-------------------|------------------|
| Dense Graded Virgin Control Granite | TM11-001      |                   |                         |               |                   | 100              |
| 4.75 SMA                            | TM11-002      |                   | 12                      | 17            | 71                |                  |
| Dense Graded taconite & sand        | TM11-005      |                   |                         | 45            | 55                |                  |

Table provided by MnDOT.

A description of the mix designs follows:

“Mixtures were designed, and specimens were produced, using a Flint Hills PG 64-34 asphalt binder for mixtures with virgin materials, and an unmodified Flint Hills PG 49-34 for mixtures containing RAP. Aggregate materials included:

- non-taconite manufactured sand bituminous aggregate
- minus 3/8-in. unwashed granite
- coarse taconite tailings
- washed coarse taconite tailings
- Mesabi Select 9/16-in. chip
- CC-70 limestone mineral filler.”

NOTE: The tested samples did not contain RAP.

*SPLP results for asphalt mix design samples*

The only metal detected by SPLP methods in the asphalt mix was arsenic. Results ranged from <0.5 µg/L to 0.84 µg/L. EPA and Minnesota water quality standards were not exceeded by these samples. Leachate from control sample TM11-001 (granite mix) did not contain arsenic above the reporting limit. The arsenic results for samples TM11-002 and TM11-005, which contained the common materials of taconite tailings and sand, were just above the laboratory’s reporting limit of 0.5 µg/L.



These results are compared with the previously reported SPLP results for aggregates alone (unbound/uncoated) in Table 25; laboratory reporting limits for these previous analyses ranged from <1 µg/L to <2 µg/L. Only one sample, E8 (Dirty Sand), exceeded the arsenic reporting limit at 2.3 µg/L. But due to the laboratory's higher reporting limit for the earlier SPLP analysis (i.e., <1 µg/L to <2 µg/L), it is difficult to say specifically what aggregate type contributed to the SPLP results for the asphalt mixes. In neither case, however, did the results exceed either EPA or Minnesota water quality standards.

**Table 25. SPLP results for asphalt-coated mix design samples.**

| INORGANICS -- Asphalt Mixes            |                |          | Metal     | Arsenic     | Barium      | Cadmium | Chromium, total | Lead             | Mercury (total) | Selenium | Silver |
|--|----------------|----------|-----------|-------------|-------------|---------|-----------------|------------------|-----------------|----------|--------|
| Water Class:                           |                |          | Units     | ug/L        | ug/L        | ug/L    | ug/L            | ug/L             | ug/L            | ug/L     | ug/L   |
| 1B                                     | DRINKING       |          | WATER (b) | 50 (i)      | 2000        | 5.00    | 100             |                  | 2               | 50       | 100(s) |
| 2A                                     | CHRONIC        |          | (CS)      | 2.0         |             |         |                 |                  | 0.0069          | 5.0      | 0.12   |
| 2A                                     | MAXIMUM        |          | (MS)      | 360         |             |         |                 |                  | 2.4*            | 20       |        |
| 2B, C & D                              | CHRONIC        |          | (CS)      | 53          |             |         |                 |                  | 0.0069          | 5.0      | 1      |
| 2B, C & D                              | MAXIMUM        |          | (MS)      | 360         |             |         |                 |                  | 2.4*            | 20       |        |
| GLI                                    | WILDLIFE       |          | VALUE (e) |             |             |         |                 |                  | 0.0013          |          |        |
| SECONDARY                              | CHRONIC        |          | VALUE (g) |             | 4           |         |                 |                  |                 |          |        |
| National Primary Drinking Standards    |                |          |           | 10          | 2000        | 5       | 100             | 5 (action level) | 2               | 50       |        |
| Recommended Water Quality Standards    | Acute          |          |           | 340         | na          | 2       | 16 (Cr VI)      | 65               | 1.4             |          | 3.2    |
| Recommended Water Quality Standards    | Chronic        |          |           | 150         | na          | 0.25    | 11 (Cr VI)      | 2.5              | 0.77            | 5        | 1.9    |
| DESCRIPTION                            | SAMPLE ID      | Lab #    | TEST      | Arsenic     | Barium      | Cadmium | Chromium, total | Lead             | Mercury (total) | Selenium | Silver |
| Granite                                | TM11-001       | TM11-001 | SPLP      | <0.5        | <5          | <0.2    | <1              | <0.5             | <0.2            | <1       | <0.2   |
| 71% Taconite, 17% Sand, 12% Filler(LS) | TM11-002       | TM11-002 | SPLP      | <b>0.62</b> | <5          | <0.2    | <1              | <0.5             | <0.2            | <1       | <0.2   |
| 55% Taconite, 45% Sand                 | TM11-005       | TM11-005 | SPLP      | <b>0.84</b> | <5          | <0.2    | <1              | <0.5             | <0.2            | <1       | <0.2   |
| SPLP Aggregate Results                 |                |          |           | Arsenic     | Barium      | Cadmium | Chromium, total | Lead             | Mercury (total) | Selenium | Silver |
| Granite                                | E4             |          | SPLP      | <2          | <70         | <0.2    | <2              | <b>2</b>         | <0.2            | <1       | <0.2   |
| Granite                                | E11            |          | SPLP      | <2          | <70         | <0.2    | <2              | <b>0.72</b>      | <0.2            | <1       | <0.2   |
| Granite                                | E12            |          | SPLP      | <2          | <70         | <0.2    | <2              | <0.5             | <0.2            | <1       | <0.2   |
| 2011                                   | Arcelor-Mittal |          | SPLP      | <1          | <1          | <0.2    | <1              | <0.5             | <0.2            | <1       | <0.2   |
| 2011                                   | Minntac        |          | SPLP      | <2          | <b>5.92</b> | <0.2    | <1              | <0.5             | <0.2            | <1       | <0.2   |
| River Sand                             | E1             |          | SPLP      | <2          | <70         | <0.2    | <2              | <b>0.69</b>      | <0.2            | <1       | <0.2   |
| Sand                                   | E7             |          | SPLP      | <2          | <70         | <0.2    | <2              | <0.5             | <0.2            | <1       | <0.2   |
| Dirty Sand                             | E8             |          | SPLP      | <b>2.3</b>  | <b>84</b>   | <0.2    | <b>13.3</b>     | <b>3.8</b>       | <0.2            | <1       | <0.2   |
| Fine Sand                              | E10            |          | SPLP      | <2          | <70         | <0.2    | <2              | <0.5             | <0.2            | <1       | <0.2   |
| Limestone                              | M-LS           |          | SPLP      | <2          | <70         | <0.2    | <2              | <0.5             | <0.2            | <1       | <0.2   |
| Limestone                              | G 3/8 BA LS    |          | SPLP      | <2          | <70         | <0.2    | <2              | <0.5             | <0.2            | <1       | <0.2   |

### Study Area 3 Summary

Maximum and minimum detections of RCRA metals above the reporting limits are summarized again in Table 26. When compared to the national water standards, unbound taconite tailings from Minntac exceed the selenium chronic RWQS of 5.0 µg/L by 2.46 µg/L. The 7.46 µg/L result is from the 0.5:1 L/S ratio sample. All other metals are within national guidelines. Granite exceeds the cadmium chronic RWQS. The limestone, gravel, and dirty sand samples exceed standards for at least two RCRA metals. Gravel exceeds standards for cadmium, total chromium, lead, and selenium.

NOTE: In Table 26, brl = below reporting level; results in **bold** exceed at least one of the standards in the rightmost (gray) columns.

**Table 26. Summary of Maximum and Minimum results for samples.**

| Analyte         | Sample                      | Minntac           | Arcelor-Mittal    | M-LS      | W6      | EI2     | E8         | National Primary Drinking Standards | Recommended Water Quality Standards | Recommended Water Quality Standards |  |
|-----------------|-----------------------------|-------------------|-------------------|-----------|---------|---------|------------|-------------------------------------|-------------------------------------|-------------------------------------|--|
|                 | Description                 | Taconite Tailings | Taconite Tailings | Limestone | Gravel  | Granite | Dirty Sand |                                     | Acute                               | Chronic                             |  |
| Arsenic         | Min                         | 1.42              | 2.62              | 0.53      | 3.52    | 1.18    | 1.02       | 10                                  | 340                                 | 150                                 |  |
|                 | Max                         | 2.78              | 9.14              | 1.61      | 6.40    | 1.84    | 2.7        |                                     |                                     |                                     |  |
| Barium          | Min                         | 5.92              | 16.4              | 15.80     | 17.80   | 16.10   | 11.6       | 2000                                | na                                  | na                                  |  |
|                 | Max                         | 232               | 16.4              | 997.00    | 1490.00 | 224.00  | 325        |                                     |                                     |                                     |  |
| Cadmium         | Min                         | brl               | brl               | 42.40     | 2.31    | 1.23    | 0.58       | 5                                   | 2                                   | 0.25                                |  |
|                 | Max                         | brl               | brl               | 42.40     | 2.31    | 1.23    | 0.58       |                                     |                                     |                                     |  |
| Chromium, total | Min                         | brl               | brl               | 4.48      | 3.57    | brl     | 6.43       | 100                                 | 16 (Cr VI)                          | 11 (Cr VI)                          |  |
|                 | Max                         | brl               | brl               | 11.70     | 25.40   | brl     | 13.30      |                                     |                                     |                                     |  |
| Cobalt          | Min                         | 6.41              | 16.8              | brl       | 2.06    | 18.00   | 2.88       | na                                  | na                                  | na                                  |  |
|                 | Max                         | 6.41              | 41.9              | brl       | 60.50   | 18.00   | 14         |                                     |                                     |                                     |  |
| Lead            | Min                         | brl               | 0.26              | 0.72      | 0.74    | 0.63    | 2.66       | 15 (action level)                   | 65                                  | 2.5                                 |  |
|                 | Max                         | brl               | 0.56              | 0.72      | 10.80   | 2.03    | 3.8        |                                     |                                     |                                     |  |
| Mercury (total) | Min                         | brl               | brl               | brl       | 0.21    | brl     | brl        | 2                                   | 1.4                                 | 0.77                                |  |
|                 | Max                         | brl               | brl               | 0.33      | 0.21    | brl     | brl        |                                     |                                     |                                     |  |
| Selenium        | Min                         | 1.46              | brl               | brl       | 5.05    | 1.13    | brl        | 50                                  |                                     | 5                                   |  |
|                 | Max                         | 7.46              | brl               | brl       | 5.05    | 1.13    | brl        |                                     |                                     |                                     |  |
| Silver          | Min                         | brl               | brl               | brl       | brl     | brl     | brl        |                                     | 3.2                                 | 1.9                                 |  |
|                 | Max                         | brl               | brl               | brl       | brl     | brl     | brl        |                                     |                                     |                                     |  |
|                 | brl - below reporting limit |                   |                   |           |         |         |            |                                     |                                     |                                     |  |

Table 27 identifies: a) the samples for which at least one of the national water standards was exceeded; and b) the test method that produced these results. These results suggest that environments in which higher levels of a metal can be produced in leachate can be dependent on the liquid-to-solid (L/S) ratio. For example, placing unbound tailings in a location with a L/S ratio of 0.5:1 could produce higher levels of selenium that could exceed RWQS in surface waters.

**Table 27. Summary of samples that exceeded national water standards.**

| DESCRIPTION        | SAMPLE ID | L/S ratio | pH, required | TEST   | Cadmium | Chromium, total | Lead | Mercury (total) | Selenium |
|--------------------|-----------|-----------|--------------|--------|---------|-----------------|------|-----------------|----------|
| Tailings           | MINNTAC   | 0.5:1     |              | metals | <0.2    | <2              | <0.5 | <0.2            | 7.46     |
| Limestone          | M-LS      | 2:1       |              | metals | <0.2    | 11.7            | 0.72 | 0.33            | <1       |
| Limestone          | M-LS      | 10:1      | 3.5          | metals | 42.4    | <100            | <10  | <0.2            | <20      |
| Gravel             | W6        | 10:1      | 3.5          | metals | 2.31    | 25.4            | 10.8 | <0.2            | 5.05     |
| Granite            | EI2       | 10:1      | 3.5          | metals | 1.23    | <2              | <0.5 | <0.2            | <1       |
| Dirty Sand (Loken) | E8        | 10:1      | 3.5          | metals | 0.58    | <2              | <0.5 | <0.2            | <1       |
| Dirty Sand (Loken) | E8        | 10:1      | 9            | metals | <0.2    | 6.43            | 2.66 | <0.2            | <1       |
| Dirty Sand (Loken) | E8        | 1:20      | 4.2          | SPLP   | <0.2    | 13.3            | 3.8  | <0.2            | <1       |

The limestone, gravel, and dirty sand samples exceeded water quality standards for more than one metal and in more than one L/S ratio test condition, suggesting that these aggregates are more likely to release metals in a larger variety of environmental conditions than taconite tailings. The results also indicate that more acidic conditions will tend to release higher concentrations of metals from unbound aggregate materials.

These testing results suggest that leachate derived from the taconite fine aggregates used in this project, in both an as-is (unbound) state and in an asphalt-bound state, meets EPA standards for all RCRA metals, with the exception of selenium (Se) for the unbound Minntac tailings sample. This single result – 7.46 µg/L (ppb) – is 2.46 µg/L (ppb) above the EPA recommended water quality standard (RWQS-C) of 5.0 µg/L. The testing condition for this unbound sample was a liquid/solid ratio of 0.5:1, which indicates limited potential release of this metal, depending on construction site environmental conditions.

In comparison, leachate derived from unbound samples of more typical aggregate sources (limestone, gravel, and dirty sand) exceeded water quality standards for more than one metal and for more than one liquid-to-solid (L/S) ratio test condition, suggesting that these aggregates – when used in an unbound state – are more likely to release metals in a larger variety of environmental conditions than would taconite tailings.

Lastly, none of the leachate derived from the three tested HMA mix design samples (two of which contained taconite materials) exceeded EPA or Minnesota water quality standards.

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## APPENDIX A

### Study Area 1 and 2 contacts

During the current FHWA project, contacts regarding taconite aggregate product usage and physical property data for 2006 to the present were re-initiated with the following entities and individuals:

1. MnDOT District 1 – Duluth
  - a) Pat Houston, Resident Engineer (phone: 725-2775)
  - b) Rod Garver, Materials Engineer (phone: 725-2733; referred by P. Houston)
  - c) Denise Anderson, Materials Laboratory (phone: 725-2738; assigned task by R. Garver)
2. MnDOT District 1 – Virginia Construction Office
  - a) Kevin Adolfs, Resident Engineer (cell phone: 218-750-0000)
3. Northland Constructors
  - a) Jim Oswald (phone: 218-722-8170)
  - b) Donovan Gustafson (phone: 218-722-5020; cell: 218-349-8368; referred by J. Oswald)
4. Ulland Brothers, Inc.
  - a) Keith Mancina (phone: 218-262-3406, cell phone: 218-966-6786)
  - b) Troy Plaster (phone: 218-384-4266, cell phone: 218-428-7564; referred by K. Mancina)
5. St. Louis County Public Works, Duluth
  - a) Jim Foldesi, Public Works Director/HWY Engineer (phone: 218-625-3830; cell: 218-343-1441)
  - b) Wayne Wilmot, Principal Engineering Tech (phone: 218-625-3843; cell: 218-343-4821; referred by J. Foldesi)
  - c) Audrey McCusker, Lab (phone: 218-625-3825)
6. St. Louis County Engineering Department, Virginia Office
  - a) Earl Wilkins, Resident Engineer, Virginia (phone: 218-742-9820)

Some of the information provided during this contact process has already been presented in this progress report. Examples of contact responses, and references to data and data files compiled as a result of this process, are presented below.

#### MnDOT District 1 – Duluth

Per P. Houston: There has been no use of taconite tailings for base or sub-grade out of the Duluth office since the Piedmont Ave. project in 2003. Subsequent use has occurred only in bituminous. (I believe this can be traced to Northland Bituminous' loss of use of the Canadian National (CN) rail yard for stockpiling and the inability to get timely rail delivery by CN. – J. Oreskovich).

Physical property data was sent via email and U.S. mail by D. Anderson. A total of 25 Aggregates Test Reports were received, covering the years 2002, 2003, and 2006-2009. All reports contained gradations, with many containing other laboratory test results such as absorptions and bulk specific gravities. The data has been entered into the Gradation\_Data\_FHWA\_012010-Transpose\_JAO\_043010 spreadsheet page "All" under the heading D1\_D. Anderson.

The data are plant-specific, rather than project-specific, for the bituminous trial mixes. Once MnDOT approves the mix, a contractor can use it on any project where it meets spec (D. Anderson, pers. comm., April, 2010). LAR and Mag Sulfate are no longer run by the District 1

laboratory on taconite aggregate as the material had consistently exceeded the specs, per Ms. Anderson. This was confirmed by Rod Garver, MnDOT District 1 Materials Engineer (pers. comm., June, 2010). According to Mr. Garver, the quality of the taconite aggregate and MnDOT's familiarity with the product make it such that they don't waste their time running these two tests. Regarding Mag Sulfate, he stated that the aggregate cannot absorb enough moisture to be affected.

#### MnDOT District 1 – Virginia

K. Adolfs emailed an Excel spreadsheet containing Bituminous Projects With Taconite Tailings, Virginia Construction 2006-2009. Individual projects are listed by year and indicate whether tailings were used in the bituminous mix or not. Where used, the mix type, percentage of tailings, and tonnage of tails are provided, as are the project number, project location start/stop points (for GIS use) and contractor.

**File:** BIT PROJECTS 2006-2009 (3).xlsx

#### Northland Constructors

Only coarse taconite tailings (no taconite crushed rock products) have been used by Northland Constructors in the requested years: 2006-present (J. Oswald, pers. comm., April, 2010). Northland Constructors has used the coarse tailings solely in bituminous mixes during these years, although Mr. Oswald is aware of others that have purchased tailings for mounds and septic systems. Oswald referred Donovan Gustafson as the contact for physical property data.

D. Gustafson agreed to pull together taconite aggregate data and provided info on a couple projects where taconite aggregate was used in bituminous (pers. comm., April, 2010). He stated that St. Louis County used 80% mine rock on a 1"-2" reclaim and re-surface job on Haines Road in Duluth in 2003, going down the hill to West Duluth. He commented that the road went five years without developing a crack and has extended the road life well beyond its initial intent. A second project he mentioned was Jean Duluth Road (Project SAP 69-637-13), where Minntac coarse tailings and 1/2" fine aggregate were used in the bituminous mix in late Fall of 2004.

D. Gustafson (pers. comm., April, 2010) provided the following aggregate make-up of the bituminous used on Haines Road (Project MP91-1250, 2003):

- 15% -3/4" rock CA (taconite)
- 5% -1/2" rock CA (taconite)
- 40% -1/2" fine FA (taconite)
- 20% coarse tailings (taconite)
- 20% RAP

The taconite came from Minntac. He noted that the -3/4" rock, -1/2" rock, and coarse tailings were all "clean," but said that the -1/2" fine FA was pretty dirty: 7.5% -200 after crushing. (Notes from the EDA project (April, 2006) report that D. Gustafson said this same mix was used for an overlay on TH 61 north from the Lester River to Homestead Road (2005?) and on County 102 in Mountain Iron (2005, by St. Louis County).

Other comments made by Gustafson (pers. comm., April, 2010):

- MnDOT changed the spec in the last few years regarding AFT (asphalt film thickness).

- Northland was penalized greatly for being too heavy (using the taconite aggregate). Adjustments were made to the mix to get closer to the #s/ft<sup>3</sup> in the spec, essentially making the mix “worse” to meet the spec.
- MnDOT switched to square yard-inch for payment. He felt that payment should be by tons.

Gustafson faxed the Field Gradation Test Report for Minorca taconite tailings on June 7, 2010. This contains just one gradation and one specific gravity per year for the years 2007-2010. He explained that the contractor must submit one gradation and specific gravity per year to the state on the material. The state plugs the contractor’s gradation results into their computer but must run a separate specific gravity. The state and the contractor have to be within 0.030 on the specific gravity for acceptance.

Gustafson stated that Northland Constructors has gotten tailings only from ArcelorMittal for approximately 4 years. This is due to a trucking issue. A trucking company hauls coal from Superior to ArcelorMittal and brings the tailings to Northland on a back haul. He would like to be able to get the tailings by rail again.

**File:** Minorca Tailings \_Northland Bit.xlsx

Ulland Bros.

K. Mancina reported that taconite aggregate was used in recent years on Cty 7 (75% Mittal coarse tailings in the bituminous?) and Cty 16 (mix of coarse tailings and waste rock used?) (pers. comm., April, 2010). This info needs to be checked for correct interpretation of notes. Mancina referred Troy Plaster as the data contact.

T. Plaster emailed an Excel table containing data on ArcelorMittal taconite aggregate products for 2006, 2008 and 2009. The products include Coarse Tailings, ¾” Minus, ½” Minus, and ¾” Rock. When queried as to whether these were yearly averages, he replied that they weren’t; “they are averages based on quantities that we used for different projects. Specific gravities are based on usually being run once per material per year. Gradations may be based on a few samples prior to mix design.” (email correspondence, April 22, 2010.)

**File:** Mittal Aggregate Properties.xls

St. Louis County Public Works, Duluth

Jim Foldesi was contacted per Donovan Gustafson of Northland Constructors regarding the 2003 Haines Road reclaim and re-surfacing project in 2003. He referred Wayne Wilmot for details. Both J. Foldesi and W. Wilmot echoed D. Gustafson’s remarks about the quality of the pavement emplaced in 2003 (pers. comm., June, 2010). Notes from D. Gustafson in April, 2006, give the job location as Haines Road from the railroad tracks to Morris Thomas Road. Per W. Wilmot (pers. comm., June, 2006), the county put down a 1” skim coat that was to buy an additional 5 years’ time for this high traffic volume road. The road is now scheduled for a rebuild in 2012 or 2013, nearly doubling the expected life of the temporary fix. Only \$3.5M of the \$10M cost has been acquired thus far. Wilmot said that the surface has held up very well, although there are problems with the underlying foundation. Culverts are moving up now. He agreed with Gustafson’s assessment of no cracks showing up in the pavement for approximately five years. Wilmot stated that coarse taconite tailings are the only taconite product used in the Duluth



district now, and only in bituminous. Another recent project that used tailings in bituminous was the Mall Project. TH 53 was MnDOT's responsibility, while St. Louis County was responsible for Maple Grove Road.

W. Wilmot referred Audrey McCusker as a contact for laboratory data. (Contact in progress.)

McCusker said that the aggregate samples she receives are the full blended aggregates used in the designed mix and that the gradations she does would be a reflection of the entire suite (pers. comm., June, 2007). She runs gradations, specific gravities, and % crushed. Data for the individual aggregate types should be obtained from the contractor.

#### St. Louis County Engineering Department, Virginia Office

E. Wilkins provided info on several county projects done out of the Virginia office (pers. comm., March, 2010):

- Hoover Road, Virginia (2007-2008): "Mill Feed" (Railroad Ballast Rejects) were used as Class 5 (6,854 yds<sup>3</sup>), \*Select Granular Embankment Mod 7% (8,000 yds<sup>3</sup>), and \*Granular Embankment Mod 7% (2,550 yds<sup>3</sup>) in the road base. Two feet of mill feed was topped with six inches of Class 5, yielding a 2 ½-ft under-pavement that is very strong. Ulland's did the hot mix pavement out of the Minorca/Laurentian pit. (\*100% passing 3" sieve & not more than 7% passing #200 sieve.)
- CSAH 63, Hibbing to Kelly Lake (2007): 803 yds<sup>3</sup> of Cobb Rock (mill rejects from Hibbing Taconite) were used 1000' from Hibbing by Hoover as a drainable subgrade material (Coarse Filter Aggregate Mod). The material was emplaced as a "giant burrito": a fabric was laid down, sloped up at the end; 1' of mill rejects was laid down and topped with another fabric. Perforated 6" pipe was used in conjunction with this. This was done for the width of the road (60') along a 300' stretch.
- Cook: ~500 yds of mill feed was used in the swamp on Ballpark Road (project connected Ballpark Road to 3<sup>rd</sup> Ave. in town).
- CSAH 16: ~2 mile stretch was paved with bituminous out of the Laurentian Pit.

**File:** Earl Wilson\_Taconite Mine Byproducts email.pdf

#### MnDOT: MnROAD 2" HMA Taconite Overlay

MnDOT has conducted work at its MnROAD facility in Albertville, MN, via a Partnership Agreement with NRRI and in collaboration with NRRI's U.S. Department of Commerce/Economic Development Administration-supported taconite aggregate research program. In a December 2009 report to NRRI, titled: *Use of Taconite Aggregates in Pavement Applications*, MnDOT reported the following about MnROAD's Mainline Cell 6 test section, in which coarse taconite tailings were used in the Cell's overlay mix design and construction in late 2008:

"Cell 6 was constructed on the Mainline to investigate the performance of a thin layer of fine aggregate asphalt mixture. It consisted of a 2" HMA overlay of a new 5" concrete pavement. This cell also supported a pooled fund study of

composite pavements, and it was split into subcells 106 and 206 (dowelled and undowelled concrete, respectively). The HMA was a 4.75 mm Superpave mixture comprised of two sources of fine taconite tailings along with a local granite manufactured sand. MnDOT designed the mixture for a laboratory study, and this was a chance to validate that design with a field test section. Fine mixtures such as this are attractive for their potential for surface course and thin lift applications. The taconite HMA overlay will be evaluated for its surface characteristics (noise, ride, texture, and friction) as well as durability and resistance to reflective cracking.

Hardrives paved the 4.75 mm asphalt mixture in a single lift in late October 2008. The HMA was only able to achieve about 90% to 91% density in the field, which was typical of other 4.75 mm mixtures observed by the National Center for Asphalt Technology (NCAT) (4). The mixture proved tough and durable, resisting damage by turning truck movements while paving the shoulders later the same day. Researchers from NCAT were on hand to observe the paving and perform early testing, and many samples of HMA were taken for several research groups to conduct laboratory performance testing.”

**CHAPTER 2: UM-CE**  
Mihai Marasteanu, Ki Hoon Moon, and Mugur Turos  
University of Minnesota

**STUDY AREA 4 – MIX DESIGN TESTING FOR THIN LAYER ASPHALT MADE WITH TACONITE TAILINGS**

**Objective**

The objective of this project is to investigate the advantages and benefits of applying taconite materials in asphalt mixture design as well as to facilitate technical information gathering of taconite and marketing of taconite uses and properties.

This report summarizes the experimental work performed in the Department of Civil Engineering at University of Minnesota as part of this investigation

**Materials used in experimental work**

Six different sets of gyratory specimens, compacted at 7% air void, were prepared at MnDOT materials laboratory and delivered to University of Minnesota. Two different types of binder from Flint Hills, PG 64-34 and PG 49-34, were used to prepare the mixtures. The PG 64-34 asphalt binder was used for the mixtures that do not contain RAP, and the unmodified PG 49-34 asphalt binder was used for the mixtures with RAP.

The following types of aggregate were used in the mix design:

- Non-taconite manufactured sand bituminous aggregate;
- Minus 3/8 inch unwashed granite;
- Coarse taconite tailings;
- Washed coarse taconite tailings;
- Mesabi Select 9/16inch chip;
- CC-70 limestone mineral filler; and
- Minus 3/8 inch RAP.

More details about the mixtures used in this study can be found in Tables 28 and 29.

**Table 28. Asphalt Mixtures Used in Experimental Work.**

| Mix ID   | Description             | Binder PG |
|----------|-------------------------|-----------|
| TM11-001 | 100% Granite            | 64-34     |
| TM11-002 | 4.75mm SMA              | 64-34     |
| TM11-003 | 4.75mm SMA w/ RAP       | 49-34     |
| TM11-004 | Granite w/RAP           | 49-34     |
| TM11-005 | Taconite w/Sand         | 64-34     |
| TM11-006 | Taconite w/Sand and RAP | 49-34     |

**Table 29. Aggregate Composition of Asphalt Mixtures.**

| Mix ID   | RAP % | Filler % | Sand % | Taconite % | Granite % |
|----------|-------|----------|--------|------------|-----------|
| TM11-001 |       |          |        |            | 100       |
| TM11-002 |       | 12       | 17     | 71         |           |
| TM11-003 | 17    | 10       | 12     | 61         |           |
| TM11-004 | 20    |          |        |            | 80        |
| TM11-005 |       |          | 45     | 55         |           |
| TM11-006 | 20    |          | 30     | 50         |           |

### Experimental work

Three different test methods, Indirect Tensile test (IDT) creep, IDT strength and Semi-Circular Bend (SCB) fracture tests, were performed to investigate the low temperature properties of asphalt mixture. Detailed information about these test methods are described elsewhere (Marasteanu et al., 2009). Three different temperatures, based on the low PG limit of the binder, were used in testing: -12°C (PG+10+12°C), -24°C (PG+10) and -36°C (PG+10-12°C). Three replicates were tested at each temperature, for a total of nine specimens per mix: 3 for IDT creep, 3 for IDT strength, and 3 for SCB fracture test.

### Data Analysis

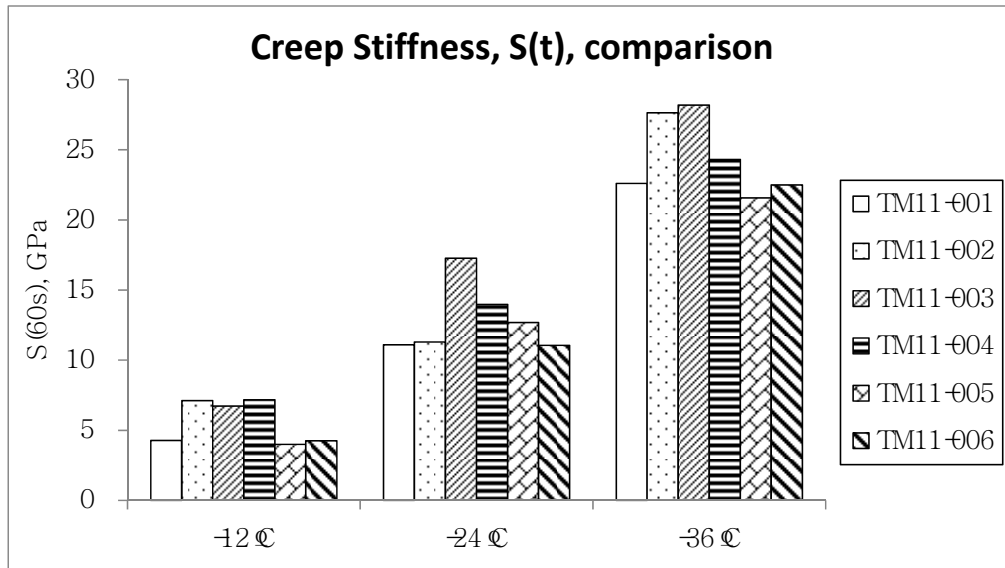
#### *IDT creep test*

IDT creep tests were performed for 1000s loading time. The inverse of creep compliance, creep stiffness  $S(t)$ , was calculated at 60 seconds and 500 seconds loading times and the values were used in the data analysis. Table 30 summarizes the  $S(60s)$  and  $S(500s)$  values for all mixtures tested.

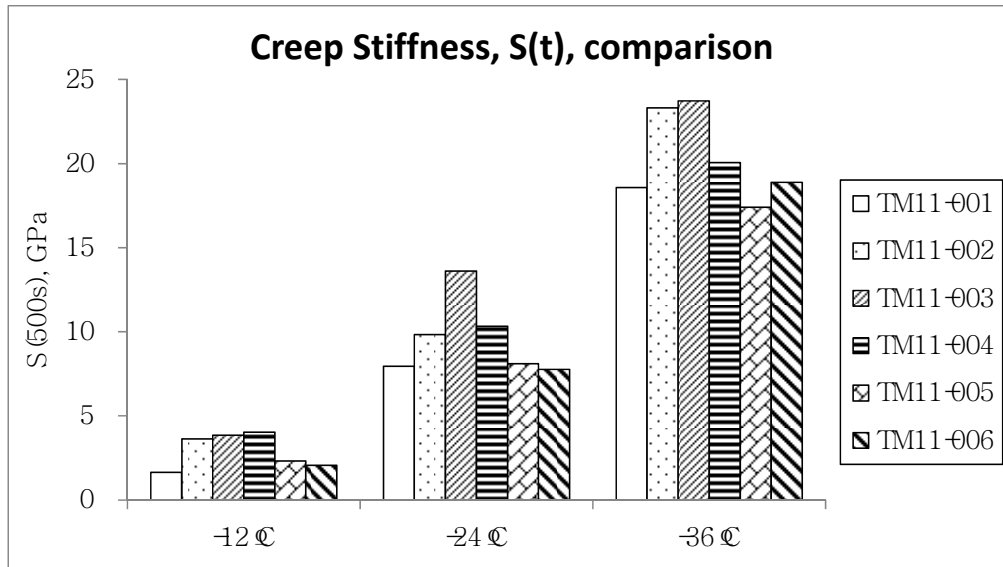
**Table 30. Summary of IDT creep test.**

| Temp, °C | Mix ID   | 60 seconds  |         | 500 seconds  |         |
|----------|----------|-------------|---------|--------------|---------|
|          |          | S(60s), GPa | C.V., % | S(500s), GPa | C.V., % |
| -12°C    | TM11-001 | 4.274       | 12.7    | 1.642        | 18.3    |
|          | TM11-002 | 7.096       | 16.0    | 3.639        | 14.1    |
|          | TM11-003 | 6.716       | 6.4     | 3.847        | 1.9     |
|          | TM11-004 | 7.146       | 15.2    | 4.033        | 19.1    |
|          | TM11-005 | 3.978       | 7.9     | 2.318        | 9.8     |
|          | TM11-006 | 4.232       | 19.1    | 2.060        | 29.0    |
| -24°C    | TM11-001 | 11.098      | 11.8    | 7.957        | 7.9     |
|          | TM11-002 | 11.270      | 7.8     | 9.837        | 7.0     |
|          | TM11-003 | 17.248      | 28.2    | 13.601       | 12.1    |
|          | TM11-004 | 13.970      | 21.2    | 10.320       | 20.8    |
|          | TM11-005 | 12.657      | 11.5    | 8.104        | 8.5     |
|          | TM11-006 | 11.039      | 11.8    | 7.771        | 12.3    |
| -36°C    | TM11-001 | 22.605      | 17.8    | 18.580       | 10.0    |
|          | TM11-002 | 27.639      | 23.0    | 23.299       | 15.2    |
|          | TM11-003 | 28.192      | 8.7     | 23.723       | 8.8     |
|          | TM11-004 | 24.309      | 5.8     | 20.055       | 12.7    |
|          | TM11-005 | 21.557      | 7.8     | 17.397       | 4.7     |
|          | TM11-006 | 22.491      | 7.8     | 18.884       | 4.8     |

The average values are also plotted in Figures 28 and 29.



**Figure 28. Comparison of creep stiffness at 60 seconds, S(60s).**



**Figure 29. Comparison of creep stiffness at 500 seconds, S(500s).**

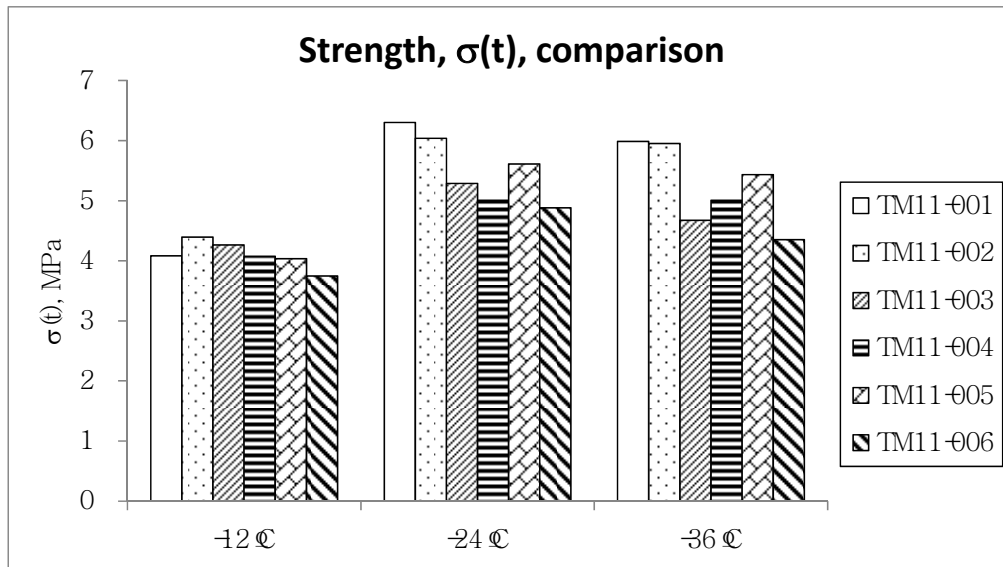
It can be observed that mixtures 3 and 2, corresponding to 4.75mm SMA with and without RAP, that also have the highest taconite content, have the higher stiffness values at all three temperatures. Mixture 4, made with granite and RAP has comparable stiffness values at the highest two temperatures.

#### *IDT strength test*

Similar to IDT creep test, strength properties of asphalt mixtures were investigated at three test temperatures: -12°C, -24°C and -36°C. A summary of IDT strength values is given in Table 31, and the average values for each tested mixture are plotted in Figure 30.

**Table 31. Summary of IDT strength results.**

| Temp, °C | Mix ID   | IDT Strength      |         |
|----------|----------|-------------------|---------|
|          |          | $\sigma(t)$ , MPa | C.V., % |
| -12°C    | TM11-001 | 4.082             | 3.3     |
|          | TM11-002 | 4.392             | 3.7     |
|          | TM11-003 | 4.265             | 3.3     |
|          | TM11-004 | 4.072             | 3.4     |
|          | TM11-005 | 4.035             | 1.3     |
|          | TM11-006 | 3.745             | 1.7     |
| -24°C    | TM11-001 | 6.301             | 1.6     |
|          | TM11-002 | 6.307             | 1.4     |
|          | TM11-003 | 5.287             | 2.0     |
|          | TM11-004 | 5.005             | 10.8    |
|          | TM11-005 | 5.610             | 4.4     |
|          | TM11-006 | 4.882             | 4.0     |
| -36°C    | TM11-001 | 5.986             | 4.6     |
|          | TM11-002 | 5.952             | 5.0     |
|          | TM11-003 | 4.672             | 3.1     |
|          | TM11-004 | 5.004             | 3.2     |
|          | TM11-005 | 5.431             | 8.7     |
|          | TM11-006 | 4.348             | 4.8     |

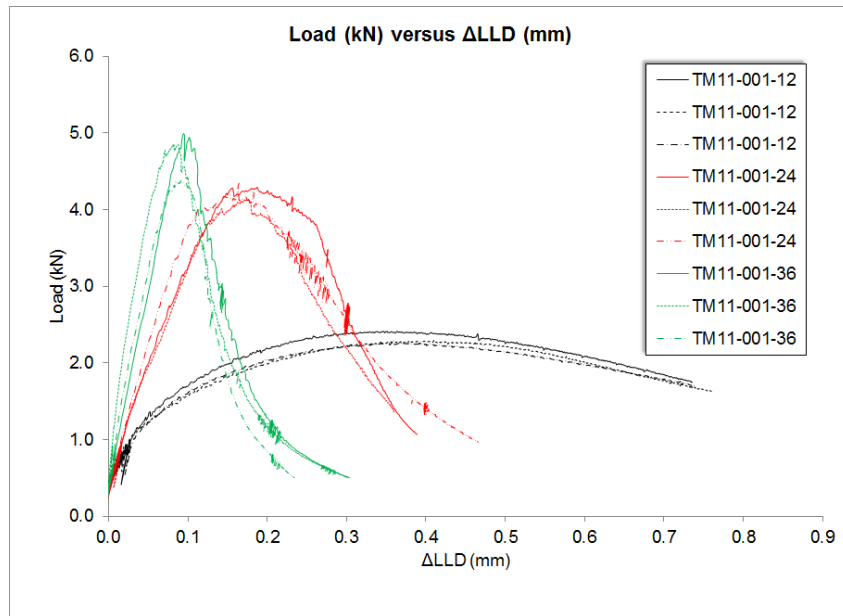


**Figure 30. IDT strength results.**

It can be observed that at -12°C, the mixtures have similar strength values. At the lower two temperatures, mixtures 1 and 2, corresponding to 100% Granite and 4.75mm SMA, respectively, have the higher strength values. It can also be observed that the mixtures containing RAP (3, 4, and 6) have the lower strength values at -24°C and -36°C.

*SCB fracture test*

Two fracture properties, fracture toughness,  $K_{IC}$  (MPa\*m<sup>0.5</sup>), and fracture energy,  $G_f$  (KJ/m<sup>2</sup>), were calculated and compared. The fracture energy,  $G_f$ , is calculated from the load versus load line displacement  $P-u$  plot. An example is shown in Figure 31. Detailed information about the calculation process can be found elsewhere (Li, 2005; Li and Marasteanu, 2009).



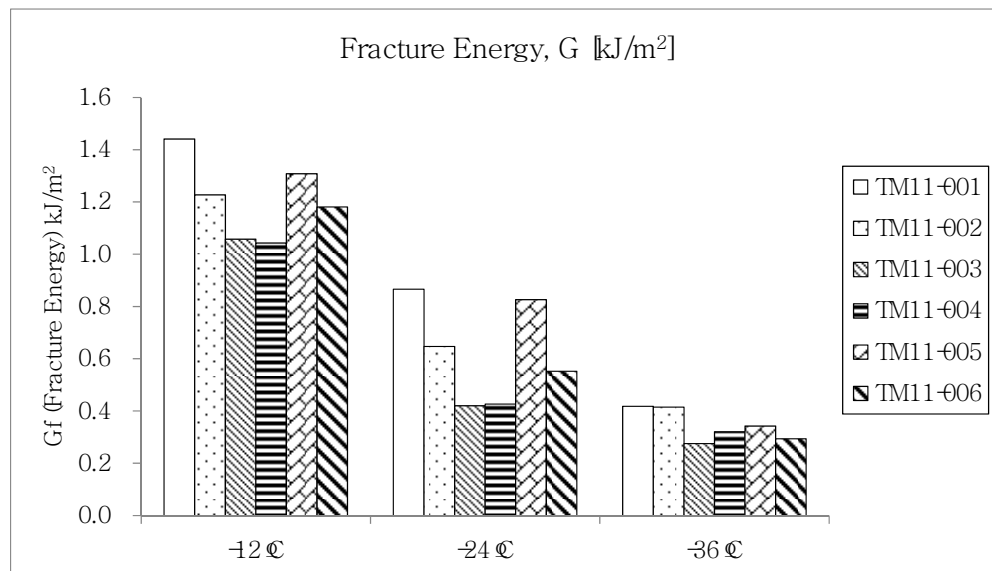
**Figure 31. P-u plot (Mixture TM11-001, -12°C, -24°C and -36°C).**

Summary data table and plots of calculated  $K_{IC}$  and  $G_f$  are shown in Table 32, and in Figures 32 and 33, respectively.

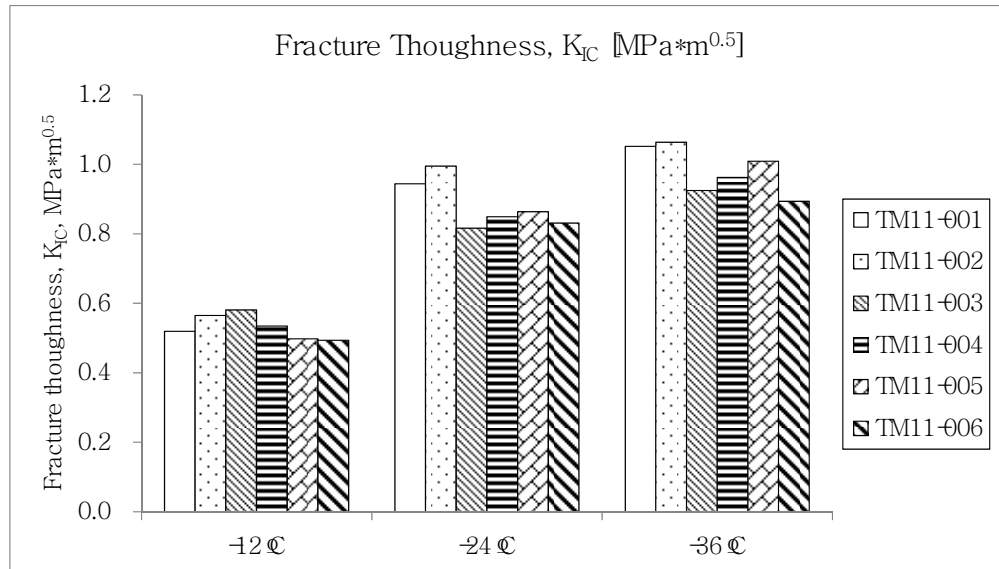


**Table 32. Summary of mixture SCB fracture toughness and energy results.**

| Temp, °C | Mix ID   | Fracture Toughness              |         | Fracture Energy           |         |
|----------|----------|---------------------------------|---------|---------------------------|---------|
|          |          | $K_{IC}$ , MPa*m <sup>0.5</sup> | C.V., % | $G_f$ , KJ/m <sup>2</sup> | C.V., % |
| -12°C    | TM11-001 | 0.519                           | 2.8     | 1.441                     | 2.2     |
|          | TM11-002 | 0.565                           | 3.2     | 1.227                     | 8.0     |
|          | TM11-003 | 0.581                           | 1.6     | 1.058                     | 5.3     |
|          | TM11-004 | 0.534                           | 3.2     | 1.043                     | 6.8     |
|          | TM11-005 | 0.498                           | 2.8     | 1.309                     | 11.1    |
|          | TM11-006 | 0.494                           | 2.4     | 1.181                     | 11.5    |
| -24°C    | TM11-001 | 0.944                           | 2.6     | 0.866                     | 5.8     |
|          | TM11-002 | 0.995                           | 3.0     | 0.647                     | 1.9     |
|          | TM11-003 | 0.816                           | 6.5     | 0.420                     | 5.4     |
|          | TM11-004 | 0.849                           | 6.1     | 0.427                     | 3.3     |
|          | TM11-005 | 0.864                           | 9.6     | 0.826                     | 19.4    |
|          | TM11-006 | 0.831                           | 2.7     | 0.553                     | 8.2     |
| -36°C    | TM11-001 | 1.051                           | 6.7     | 0.417                     | 9.7     |
|          | TM11-002 | 1.063                           | 4.0     | 0.415                     | 11.1    |
|          | TM11-003 | 0.925                           | 3.8     | 0.276                     | 4.1     |
|          | TM11-004 | 0.962                           | 1.3     | 0.321                     | 20.7    |
|          | TM11-005 | 1.009                           | 4.6     | 0.342                     | 7.8     |
|          | TM11-006 | 0.894                           | 2.3     | 0.294                     | 16.1    |



**Figure 32. SCB fracture energy values.**



**Figure 33. SCB fracture toughness values.**

It can be observed that mixtures 1 and 5, corresponding to 100% granite and taconite with sand, respectively, have the highest fracture energy at -12°C, and -24°C. At -36°C, mixtures 1 and 2 have the highest energy followed by mixture 5. For fracture toughness, mixtures 1 and 2 are the toughest at -24°C and -36°C. At -12°C, the six mixtures have similar values.

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## **CHAPTER 3: MnDOT**

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### **STUDY AREA 5 – DEVELOP MIX DESIGN FOR THIN LAYER ASPHALT MADE WITH TACONITE TAILINGS**

#### **Project Background**

This study area examined the mixture performance and viability of designing 4.75-mm-type mixtures using several Minnesota aggregate products. Seven (7) asphalt mixtures were designed and evaluated as part of this task. A total of 99 laboratory specimens were produced. This report summarizes the results of the laboratory mixture evaluation.

The objective of this cooperative project is to advance the knowledge of the beneficial uses of taconite mining tailings as well as to facilitate technical information gathering and marketing of such uses and properties.

#### **Research Objectives**

The primary objective of this study area was to design several taconite-bearing asphalt mixtures and produce laboratory test specimens. The design outcomes and test results were then compared to similar mixtures that were primarily composed of granite. Comparisons focused on several points, including:

- Recycled asphalt mixture content;
- Virgin asphalt binder content;
- Compaction effort;
- Asphalt demand;
- Tensile strength;
- Laboratory permeability; and
- Performance testing (dynamic modulus, asphalt pavement analyzer).

#### **Design Criteria**

During the past decade several research efforts have focused on 4.75-mm mixtures. Researchers have made recommendations regarding aggregate gradation, volumetric design, traffic level, and material type. The outcomes from several studies by the Minnesota Department of Transportation (MnDOT) and the National Center for Asphalt Technology (NCAT), and the current and proposed AASHTO design recommendations, were most useful to this study.

Although standards and recommendations exist there are still some questions as to which set of available criteria would be most appropriate for Minnesota. In this study the asphalt technologists approached the design process of every mixture with an eye toward which set of recommendations would produce a satisfactory design.

### *Summary of Design Recommendations for Dense-Graded 4.75-mm Mixtures*

Cooley et al. (2002) performed research on the design of 4.75-mm mixtures and proposed a set of criteria for design for mixtures designed at 75 gyrations:

- Gradation control:
  - 30 to 54% passing the #16 (1.18-mm) sieve
  - 6 to 12% passing the #200 (0.075-mm) sieve;
- Design air voids of 4%;
- VMA of 16 (for all traffic levels) to 18% (reduces excessive optimum binder content);
- VFA from 75 to 78%; and
- A dust to effective binder ratio (P#200/Pbe) from 0.9 to 2.2.

### *Summary of Design Recommendations for 4.75-mm SMA Mixtures*

Xui et al. (2003) performed research on the design of 4.75-mm SMA mixtures and made the following conclusions:

- Non-modified asphalt is not recommended due to high APA rutting for all the mixtures tested;
- Material type, aggregate shape, angularity and texture influence SMA mixture volumetric criteria;
- Design the aggregate blend with 12 to 15% passing the #200 (0.075 mm) sieve; and
- Use #8 (2.36 mm) wire mesh size for drain down basket when testing 4.75 mm NMA SMA and standard 1/4-in. (6.3 mm) mesh when testing other mixtures.

Table 33 compares the treatment of selected mixture properties relative to AASHTO, NCAT, and MnDOT recommendations. Table 34 presents the aggregate gradation standards that were used in the design process.

**Table 33. Selected 4.75-mm Mixture Properties.**

| Mixture Property    | 4.75 Design Criteria |                |         |             |
|---------------------|----------------------|----------------|---------|-------------|
|                     | Proposed AASHTO      | Current AASHTO | NCAT    | MnDOT UTBWC |
| VCA;<br>mix and dry |                      |                | x       |             |
| Ndes                | 75                   | 75             | 75      |             |
| min FAA             | 45                   | 40             | 47      | 40          |
| Gsb                 |                      |                |         |             |
| Gse                 |                      |                |         |             |
| %AC                 |                      |                | Min 5.8 | 4.8-6.0     |
| Vbe                 | 12.0-15.0            | na             |         |             |
| %Gmm at initial     | <90.5                | <90.5          |         |             |
| F/E                 | 1.0-2.0              | 0.9-2.0        |         |             |
| Gmm                 |                      |                |         |             |
| Gmb                 |                      |                |         |             |
| Voids               | 4.0-6.0              | 4.0            | 4.0     |             |
| VMA                 | na                   | 16.0           | 17.0    |             |
| VFA                 | na                   | 65-78          |         |             |
| Pbe                 |                      |                |         |             |
| adj SA              |                      |                |         |             |
| adj AFT             |                      |                |         | Min 10.5    |
| %newAC              |                      |                |         | 100         |
| %RAP                |                      |                |         |             |
| min TSR             | 80                   | 80             | 80      | 80          |
| APA                 |                      |                |         |             |
| CAA 1face           |                      |                |         | 95          |
| CAA 2face           |                      |                |         | 85          |
| Flat-Elongated      |                      |                |         | Max 25      |
| LAR                 |                      |                |         | Max 40      |

**Table 34. Aggregate Gradation Standards.**

| Sieve,<br>in | Sieve,<br>mm | Current<br>AASHTO | Proposed<br>AASHTO | NCAT SMA    | MnDOT<br>UTBWC |
|--------------|--------------|-------------------|--------------------|-------------|----------------|
|              |              | Percent Passing   |                    |             |                |
| 1/2"         | 12.5         | 100               | 100                | 100         | 100            |
| 3/8"         | 9.5          | 95 - 100          | 95 - 100           | 100         | 85 - 100       |
| #4           | 4.75         | 90 - 100          | 90 - 100           | 90 - 100    | 28 - 42        |
| #8           | 2.36         |                   |                    | 28 - 65     | 22 - 36        |
| #16          | 1.18         | 30 - 60           | 30 - 55            | 22 - 36     | 15 - 23        |
| #30          | 0.6          |                   |                    | 18 - 28     | 10 - 18        |
| #50          | 0.3          |                   |                    | 15 - 22     | 8 - 13         |
| #100         | 0.15         |                   |                    |             | 6 - 10         |
| #200         | 0.075        | 6.0 - 12.0        | 6.0 - 13.0         | 12.0 - 15.0 | 4.0 - 5.5      |

## MATERIAL DESCRIPTIONS

### Aggregates

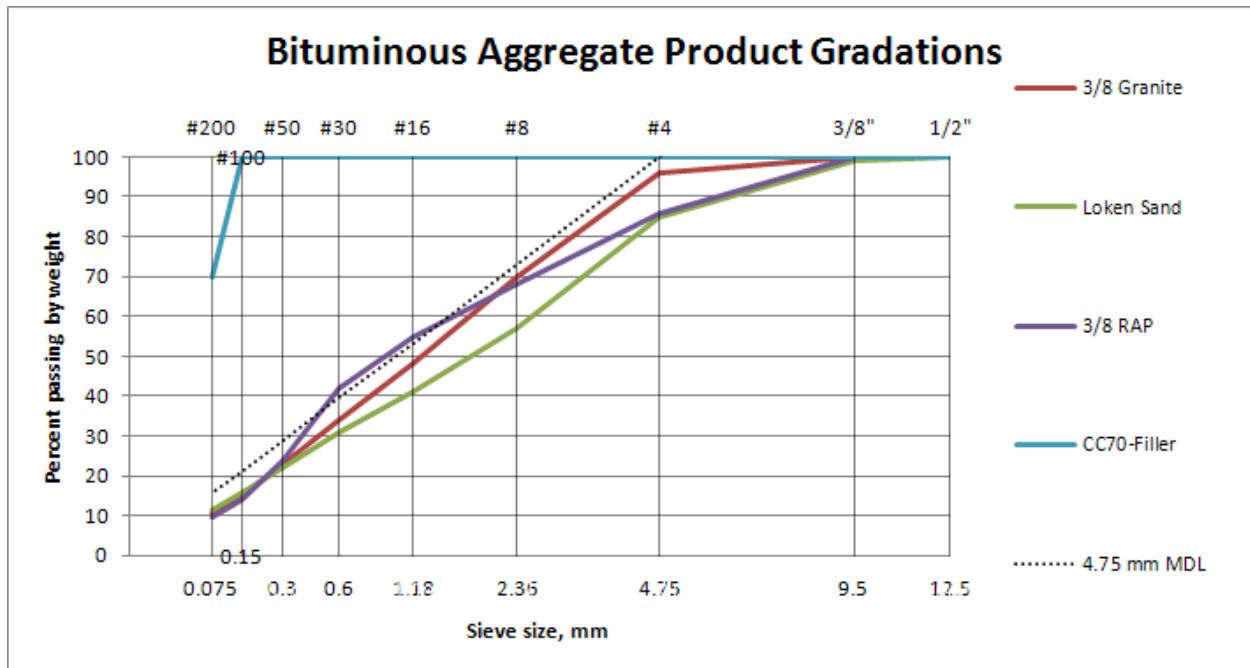
Description of aggregates used in the study:

- Select Mesabi-Range taconite tailings were obtained from NRRI stockpiles:
  - Minntac coarse tailings 3/8-in. (9.5 mm) minus material was uniformly graded from coarse to fine;
  - ArcelorMittal coarse tailings 3/8-in. (9.5 mm) minus material was uniformly graded from coarse to fine;
  - Martin Marietta granite unwashed 3/8-in. (9.5 mm) minus material was uniformly graded from coarse to fine;
  - Mittal coarse tailings (2<sup>nd</sup> wash), gap graded passing the #8 (2.36 mm) sieve;
  - Mittal coarse tailings (2<sup>nd</sup> wash), gap graded on the minus 1 1/4-in. (4.75 mm) to #8 (2.36 mm) sieves;
  - Mesabi Select 9/16-in. coarse aggregate; and
  - Washed Manufactured Sand was a coarsely graded fine aggregate.
- Loken 3/8-in. minus manufactured sand material was uniformly graded from coarse to fine;
- Screened RAP, 100 percent passing the 3/8-in. (9.5 mm) sieve; and
- Superior Materials CC70 calcium carbonate mineral filler.

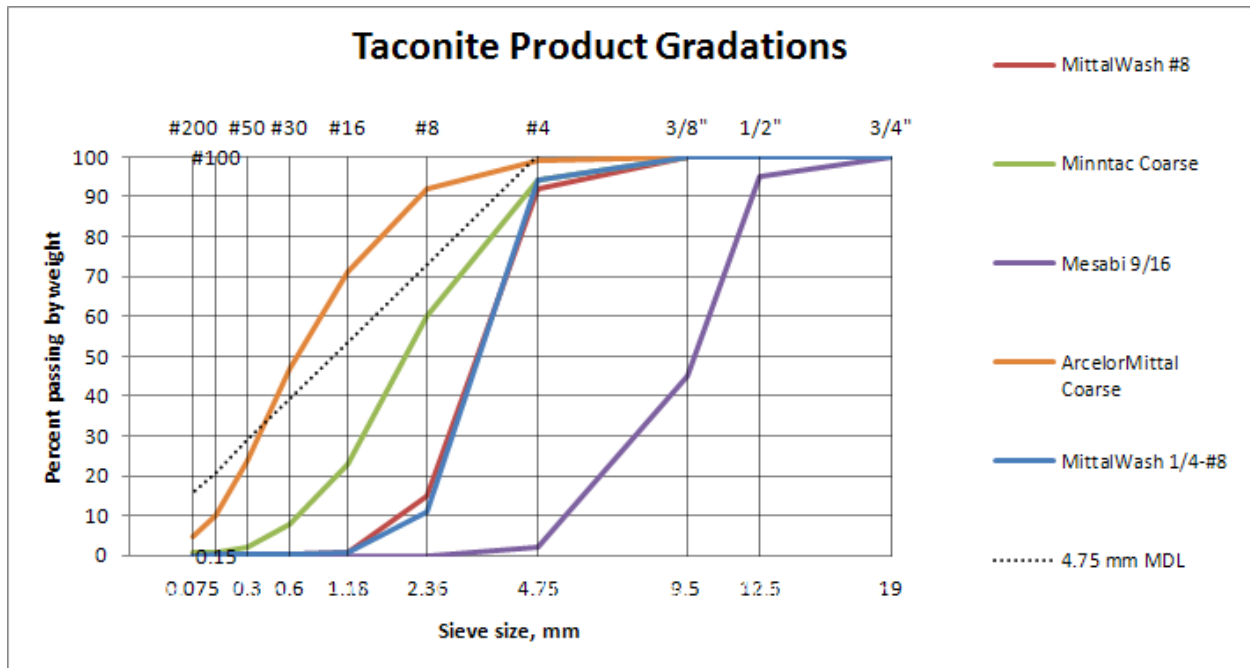
Aggregate gradation and bulk specific gravity was performed on all of the aggregate materials. Fine aggregate angularity and percent asphalt was also performed on the RAP material. Fine aggregate angularity was performed on the final aggregate blends as part of the mixture design testing. Table 35 lists the bulk specific gravity of aggregate materials and gradations of are plotted in Figure 34 and Figure 35.

**Table 35. Bulk Specific Gravity of Aggregates.**

| Aggregate                     | Gsb   |
|-------------------------------|-------|
| Loken Man Sand                | 2.688 |
| 3/8 Unwashed Granite          | 2.679 |
| Minntac Coarse Tailings       | 2.886 |
| Mittal Washed Products        | 2.826 |
| ArcelorMittal Coarse Tailings | 2.900 |
| CC-70 Mineral Filler          | 2.722 |
| 3/8 RAP (FAA = 49, AC =5.3)   | 2.631 |
| Mesabi 9/16 Chip              | 2.929 |



**Figure 34. Gradation of non-taconite aggregates in 4.75 mm Mixture Study.**



**Figure 35. Gradation of taconite aggregates in 4.75 mm Mixture Study.**

The aggregate materials were split on the #4 and #8 (4.75 and 2.36 mm) sieves and stored as sized fractions to control segregation within storage containers. The RAP was fractionated by screening on the 3/8 (9.5 mm) sieve, then handled in a manner similar to the other aggregates.

### Asphalt Binders

Two binder grades were used during the mixture design and specimen production phases. PG 64-34 was selected as the primary grade, and was used in the mixtures that contained only virgin aggregates. PG 64-34 was chosen because this grade would be best for potential high-traffic wear-course applications, and so that comparisons could be made to the MnROAD Cell 6 mixture, which was constructed at MnROAD's high-volume test facility in 2008. The second binder grade was PG 49-34, which was used for mixtures containing RAP. Both binders were produced by Flint Hills Resources Pine Bend refinery.

### Dynamic Shear Rheometer (DSR) Testing

Dynamic Shear Rheometer (DSR) testing was performed on the two asphalt binders used in the mixture development study area. DSR testing utilizes frequency sweeps at multiple temperatures, and measures the binder response in terms of shear modulus ( $G^*$ ) and response phase angle ( $\delta$ ). The test temperatures range is somewhat dependent upon material behavior. High temperature limits depend on the ability to maintain geometric standards, and low temperature depends on the ability to collect meaningful data. The unmodified PG 49-34 binder was tested at 40, 46, 52, and 58 °C and the PG 64-34 binder was tested at 52, 58, 64, 70, and 76°C.

DSR results are presented in Table 36, Figure 36, and Figure 37. The effect of binder modification is shown in Figure 38, where the binder materials are compared for stiffness versus frequency at 52°C and 58°C. Note that the effect of polymer modification is maintained stiffness



as test frequencies move from high to low. This effect corresponds to the expected behavior as temperatures move from low to high.

**Table 36. DSR Results.**

|                    |                         | <b>PG 49-34</b> |                      |               | <b>PG 64-34</b> |                      |               |
|--------------------|-------------------------|-----------------|----------------------|---------------|-----------------|----------------------|---------------|
| <b>Temperature</b> | <b>Frequency, rad/s</b> | <b>Time, s</b>  | <b>Delta degrees</b> | <b>G*, Pa</b> | <b>Time, s</b>  | <b>Delta degrees</b> | <b>G*, Pa</b> |
| 40 °C              | 1                       | 241.75          | 84.98                | 1414          |                 |                      |               |
|                    | 1.586                   | 320.03          | 84                   | 2170          |                 |                      |               |
|                    | 2.512                   | 419.91          | 82.94                | 3276          |                 |                      |               |
|                    | 3.98                    | 484.56          | 81.99                | 4976          |                 |                      |               |
|                    | 6.309                   | 527.34          | 81.04                | 7582          |                 |                      |               |
|                    | 10                      | 557.59          | 79.99                | 11410         |                 |                      |               |
|                    | 15.84                   | 579.27          | 78.98                | 17200         |                 |                      |               |
|                    | 25.12                   | 593.08          | 78.04                | 25720         |                 |                      |               |
|                    | 39.81                   | 604.03          | 77.12                | 38350         |                 |                      |               |
|                    | 63.09                   | 611.42          | 76.23                | 56900         |                 |                      |               |
| 100                | 617.61                  | 75.25           | 83840                |               |                 |                      |               |
| 46 °C              | 1                       | 241.75          | 87.07                | 569.1         |                 |                      |               |
|                    | 1.586                   | 319.95          | 86.23                | 843.6         |                 |                      |               |
|                    | 2.512                   | 468.8           | 85.33                | 1314          |                 |                      |               |
|                    | 3.98                    | 597.05          | 84.41                | 2048          |                 |                      |               |
|                    | 6.309                   | 639.66          | 83.45                | 3077          |                 |                      |               |
|                    | 10                      | 671.02          | 82.51                | 4771          |                 |                      |               |
|                    | 15.84                   | 692.05          | 81.55                | 7260          |                 |                      |               |
|                    | 25.12                   | 705.91          | 80.59                | 10950         |                 |                      |               |
|                    | 39.81                   | 717.02          | 79.65                | 16470         |                 |                      |               |
|                    | 63.09                   | 724.63          | 78.7                 | 24650         |                 |                      |               |
| 100                | 730.92                  | 77.73           | 36710                |               |                 |                      |               |
| 52 °C              | 1                       | 241.67          | 88.53                | 227.5         | 120.63          | 55.95                | 2031          |
|                    | 1.586                   | 319.98          | 873.9                | 359.6         | 198.95          | 56.13                | 2726          |
|                    | 2.512                   | 370.78          | 87.18                | 550           | 249.36          | 56.42                | 3620          |
|                    | 3.98                    | 467.31          | 86.4                 | 885.2         | 282.5           | 56.78                | 4778          |
|                    | 6.309                   | 510.11          | 85.51                | 1331          | 304.48          | 57.23                | 6312          |
|                    | 10                      | 568.69          | 84.7                 | 2171          | 319.88          | 57.84                | 8410          |
|                    | 15.84                   | 610.83          | 83.71                | 3148          | 331.42          | 58.42                | 11270         |
|                    | 25.12                   | 624.78          | 82.79                | 4844          | 338.59          | 59.19                | 15210         |
|                    | 39.81                   | 635.88          | 81.86                | 7382          | 344.86          | 60.05                | 20630         |
|                    | 63.09                   | 643.45          | 80.88                | 11171         | 349.16          | 60.93                | 27980         |
| 100                | 649.22                  | 79.88           | 16860                | 352.45        | 62.22           | 37150                |               |
| 58 °C              | 1                       | 241.73          | 89.33                | 98.51         | 241.78          | 56.85                | 1191          |
|                    | 1.586                   | 320.42          | 89.01                | 155.7         | 473.73          | 56.88                | 1577          |
|                    | 2.512                   | 371.28          | 88.5                 | 245.7         | 574.02          | 56.95                | 2085          |
|                    | 3.98                    | 404.42          | 87.88                | 381.9         | 638.7           | 57.1                 | 2771          |

|             |                  | PG 49-34 |               |        | PG 64-34 |               |        |
|-------------|------------------|----------|---------------|--------|----------|---------------|--------|
| Temperature | Frequency, rad/s | Time, s  | Delta degrees | G*, Pa | Time, s  | Delta degrees | G*, Pa |
|             | 6.309            | 488.5    | 87.17         | 597.3  | 681.44   | 57.37         | 3699   |
|             | 10               | 532.63   | 86.4          | 932.2  | 711.69   | 57.75         | 4956   |
|             | 15.84            | 574.83   | 85.55         | 1445   | 733.36   | 58.21         | 6648   |
|             | 25.12            | 595.06   | 84.94         | 2308   | 747.23   | 58.79         | 8951   |
|             | 39.81            | 606.05   | 83078         | 3452   | 758.31   | 59.45         | 12090  |
|             | 63.09            | 613.77   | 82.79         | 5285   | 765.94   | 60.19         | 16410  |
|             | 100              | 620.03   | 81.87         | 8099   | 772.27   | 60.95         | 22370  |
| 64 °C       | 1                |          |               |        | 362.64   | 58.49         | 681.5  |
|             | 1.586            |          |               |        | 517.77   | 58.23         | 914.4  |
|             | 2.512            |          |               |        | 617.61   | 58.07         | 1223   |
|             | 3.98             |          |               |        | 682.47   | 58            | 1639   |
|             | 6.309            |          |               |        | 725.13   | 58.06         | 2198   |
|             | 10               |          |               |        | 755.42   | 58.22         | 2955   |
|             | 15.84            |          |               |        | 777.47   | 58.49         | 3974   |
|             | 25.12            |          |               |        | 790.97   | 58.87         | 5355   |
|             | 39.81            |          |               |        | 801.89   | 59.35         | 7233   |
|             | 63.09            |          |               |        | 809.14   | 59.92         | 9801   |
|             | 100              |          |               |        | 815.38   | 60.58         | 13340  |
| 70 °C       | 1                |          |               |        | 362.83   | 61.04         | 395    |
|             | 1.586            |          |               |        | 518.42   | 60.42         | 536.3  |
|             | 2.512            |          |               |        | 618.31   | 59.94         | 728.8  |
|             | 3.98             |          |               |        | 683.06   | 59.57         | 988.8  |
|             | 6.309            |          |               |        | 725.83   | 59.36         | 1340   |
|             | 10               |          |               |        | 756.08   | 59.28         | 1813   |
|             | 15.84            |          |               |        | 778.05   | 59.32         | 2448   |
|             | 25.12            |          |               |        | 791.56   | 59.49         | 3311   |
|             | 39.81            |          |               |        | 802.59   | 59.79         | 4481   |
|             | 63.09            |          |               |        | 810.31   | 60.22         | 6073   |
|             | 100              |          |               |        | 816.59   | 60.8          | 8255   |
| 76 °C       | 1                |          |               |        | 241.61   | 64.67         | 229    |
|             | 1.586            |          |               |        | 397.22   | 63.61         | 316.7  |
|             | 2.512            |          |               |        | 497.14   | 62.7          | 435.9  |
|             | 3.98             |          |               |        | 562.31   | 61.95         | 598.3  |
|             | 6.309            |          |               |        | 605.02   | 61.38         | 817.9  |
|             | 10               |          |               |        | 625.27   | 60.98         | 1114   |
|             | 15.84            |          |               |        | 657.39   | 60.71         | 1516   |
|             | 25.12            |          |               |        | 671.3    | 60.6          | 2061   |
|             | 39.81            |          |               |        | 682.36   | 60.63         | 2802   |
|             | 63.09            |          |               |        | 690.02   | 60.81         | 3810   |
|             | 100              |          |               |        | 696.25   | 61.14         | 5190   |

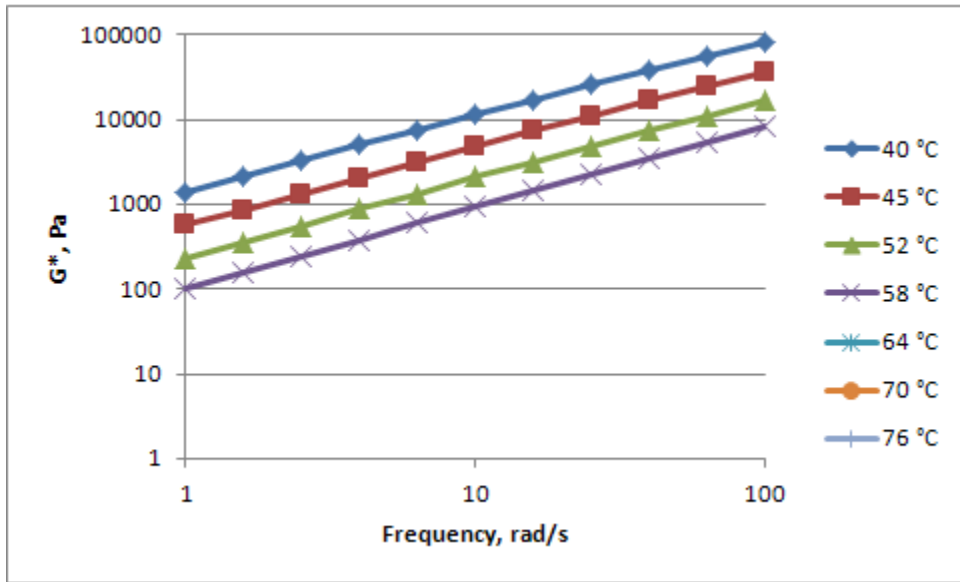


Figure 36. DSR frequency sweep results for PG 49-34 binder.

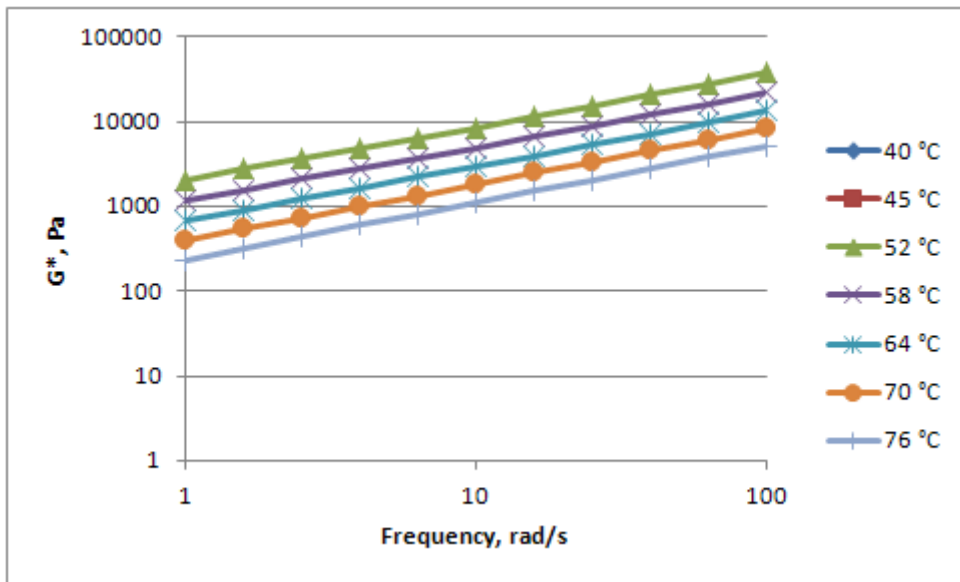
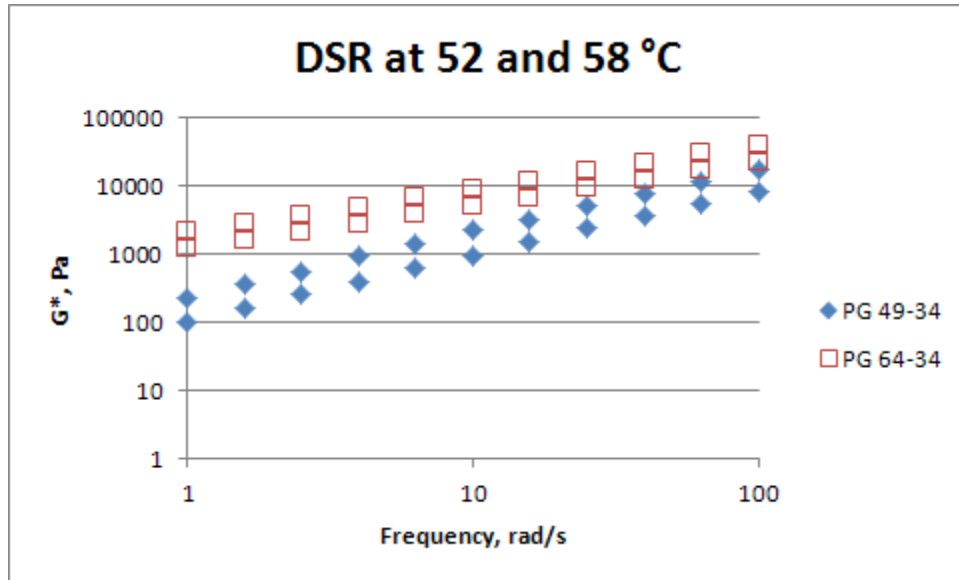


Figure 37. DSR frequency sweep results for PG 64-34 binder.



**Figure 38. Binder DSR comparisons at 52 and 58°C.**

## MIXTURES

### *Mixture Descriptions*

Laboratory designs were performed for seven mixture using taconite tailings or granite as the primary aggregate types.

The mixtures contained between 5.98 and 7.5 percent binder content by weight. The RAP content of the companion designs varied from 17 to 20 percent.

One dense graded control mixture was designed solely from granite, and was designated the primary control mixture. A secondary control mixture was designed using granite and RAP. This document will refer to the following set of 4.75-mm mixtures as listed in Table 37:

1. Dense-graded control mixture (G-001), 100% granite;
2. 4.75-mm SMA (S-002), 71% coarse taconite tailings;
3. 4.75-mm SMA (RS-003), 61% coarse taconite tailings and 17% RAP;
4. Dense-graded recycled mixture (RG-004), 80% granite and 20% RAP;
5. Dense-graded taconite mixture (RG-005), 55% taconite;
6. Dense-graded recycled mixture (RG-004), 50% granite and 20% RAP;
7. Gap-graded taconite mixture (UTBWC), 69% taconite; and
8. Dense-graded taconite mixture (T-2008), 65% taconite designed for MnROAD in 2008.

**Table 37. Mixture Designs by Material Type.**

| <b>4.75 Design Type</b>                          | <b>Mix ID</b> | <b>Fine RAP %</b> | <b>Mineral Filler %</b> | <b>Sand %</b> | <b>Taconite %</b> | <b>Granite %</b> |
|--|---------------|-------------------|-------------------------|---------------|-------------------|------------------|
| 2008's Dense Graded taconite & sand              | T-2008        |                   |                         | 35            | 65                |                  |
| Dense Graded Granite <i>Virgin Control</i>       | G-001         |                   |                         |               |                   | 100              |
| 4.75 SMA   | S-002         |                   | 12                      | 17            | 71                |                  |
| 4.75 SMA w/RAP                                   | RS-003        | 17                | 10                      | 12            | 61                |                  |
| Dense Graded Granite%RAP <i>Recycled Control</i> | RG-004        | 20                |                         |               |                   | 80               |
| Dense Graded taconite & sand                     | T-005         |                   |                         | 45            | 55                |                  |
| Dense Graded taconite & sand w/RAP               | RT-006        | 20                |                         | 30            | 50                |                  |
| Gap Graded taconite & sand                       | UTBWC         |                   |                         | 31            | 69                |                  |

Table 38 presents the aggregate blends by percentage of product for the seven mixture designs.

**Table 38. Mixture Designs by Product Percentage**

| <b>Product</b>   | <b>Percent Product by Mixture ID</b> |              |               |               |              |               |              |
|--|--------------------------------------|--------------|---------------|---------------|--------------|---------------|--------------|
|  | <b>G-001</b>                         | <b>S-002</b> | <b>RS-003</b> | <b>RG-004</b> | <b>T-005</b> | <b>RT-006</b> | <b>UTBWC</b> |
| Mesabi 9/16 chip (taconite overburden)                   |                                      |              |               |               |              |               | 55%          |
| ArcelorMittal coarse taconite tailings                   |                                      | 11%          | 5%            |               | 40%          | 35%           | 14%          |
| Mittal 2 <sup>nd</sup> wash -1/4 to #8 taconite tailings |                                      | 43%          | 56%           |               |              |               |              |
| Mittal 2 <sup>nd</sup> wash #8 taconite tailings         |                                      | 5%           |               |               |              |               |              |
| Minntac coarse taconite tailings                         |                                      | 12%          |               |               | 15%          | 15%           |              |
| Loken sand   |                                      | 17%          | 12%           |               | 45%          | 30%           | 31%          |
| Granite 3/8 unwashed man sand                            | 100%                                 |              |               | 80%           |              |               |              |
| -3/8 RAP   |                                      |              | 17%           | 20%           |              | 20%           |              |
| Mineral filler CC-70                                     |                                      | 12%          | 10%           |               |              |               |              |
| Asphalt binder PG 64-34                                  | 6.9%                                 | 5.8%         |               |               | 7.5%         |               | 6.0%         |
| Asphalt binder PG 49-34                                  |                                      |              |               |               |              |               |              |
| Total AC   |                                      |              | 5.8%          | 6.4%          |              | 7.1%          |              |
| New AC   |                                      |              | 5.0%          | 5.4%          |              | 6.1%          |              |

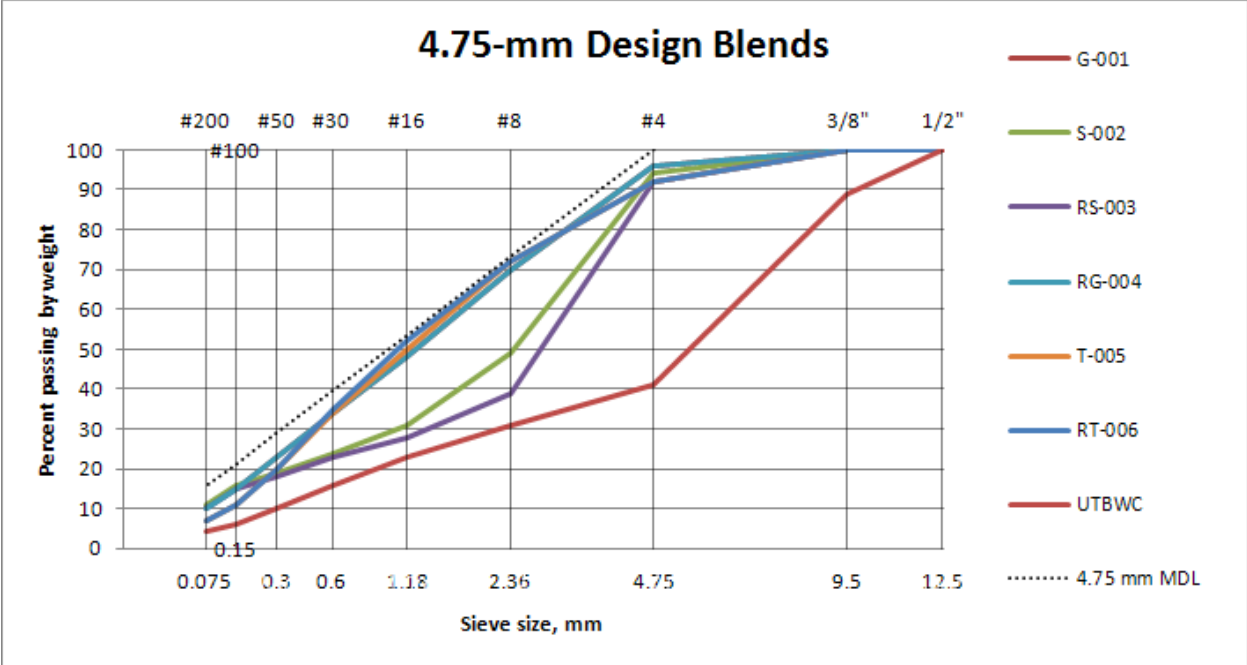
*Design Results for 4.75-mm Mixtures*

As previously discussed, the design process was intended to compare the use of granite, taconite aggregates, and RAP in satisfying recommended design criteria. In particular, properties of the granite control (G-001) and the taconite-sand (T-006) mixtures would be compared to the 2008 taconite mixture that was constructed at MnROAD (T-2008).

Table 39 and Figure 39 show the gradation of the aggregate blends. The dense-graded mixtures (G-001, RG-004, T-005, and RT-006) satisfied the current and proposed AASHTO broadband gradation limits (Table 34). Extrapolating from maximum specific gravity values obtained from the design process, the unit weights of mixtures G-001, T-005, UTBWC, and T-2008 at 96 percent Gmm (4.0 percent air voids) would be 146.1, 152.1, 152.9, and 152.9 lbs/ft<sup>3</sup> (23.0, 23.9, 24.0, and 24.0 kN/m<sup>3</sup>).

**Table 39. Aggregate Design Data for 4.75-mm Mixtures.**

|  |            | Mixture                                |       |        |        |       |        |       |
|--|------------|--|-------|--------|--------|-------|--------|-------|
|  |            | G-001                                  | S-002 | RS-003 | RG-004 | T-005 | RT-006 | UTBWC |
| Sieve  |            | Percent Passing by Weight of Aggregate |       |        |        |       |        |       |
| English  | Metric, mm |  |       |        |        |       |        |       |
| ½  | 12.5       | 100                                    | 100   | 100    | 100    | 100   | 100    | 100   |
| 3/8  | 9.5        | 100                                    | 100   | 100    | 100    | 100   | 100    | 89    |
| #4   | 4.75       | 95                                     | 94    | 92     | 93     | 92    | 92     | 41    |
| #8   | 2.36       | 71                                     | 49    | 39     | 70     | 71    | 72     | 31    |
| #16  | 1.18       | 48                                     | 31    | 28     | 49     | 50    | 52     | 23    |
| #30  | 0.600      | 33                                     | 24    | 23     | 35     | 34    | 35     | 16    |
| #50  | 0.300      | 21                                     | 19    | 18     | 22     | 20    | 20     | 10    |
| #100   | 0.150      | 13                                     | 16    | 15     | 13     | 11    | 11     | 6     |
| #200   | 0.075      | 9.1                                    | 11.0  | 10.3   | 9.2    | 7.1   | 7.1    | 4.2   |
|  |            | Properties of Aggregate Blends         |       |        |        |       |        |       |
| Blend  | Gsb        | 2.679                                  | 2.834 | 2.806  | 2.669  | 2.799 | 2.776  | 2.846 |
| Blend  | FAA        | 50                                     | 45    | 44     | 46.7   | *     | 45     | *     |
| (*) Blend not measured: Blend FAA > 40 by comparison of blends containing similar products |            |  |       |        |        |       |        |       |



**Figure 39. Design Gradations.**

*Design Results for Ultra-Thin Bonded Wear Mixtures*

The design process was intended to evaluate the use of taconite aggregates in satisfying MnDOT’s design criteria. The aggregate design contained 69% taconite tailings and overburden. The only non-taconite component was the angular sand that was used in the other bituminous mixtures.

Trial mixture iterations were performed on the design gradation at asphalt binder percentages between 5.2 and 6.0. Results from drain-down testing were negative, indicating that fibers would not be required for designs using these materials. Asphalt demand was greater than expected, but within the practical range. If necessary, the demand can be decreased by adjusting the aggregate gradation to remove fine material.

**Table 40. Properties of Mixtures.**

| Mixture Property        | Mix ID |              |              |        |       |        |        |       |
|-------------------------|--------|--------------|--------------|--------|-------|--------|--------|-------|
|                         | G-001  | S-002        | RS-003       | RG-004 | T-005 | RT-006 | T-2008 | UTBWC |
| VCA mix dry             |        | 43.3<br>42.6 | 41.4<br>42.0 |        |       |        |        |       |
| Ndes                    | 75     | 75           | 75           | 75     | 75    | 75     | 75     |       |
| FAA                     | 50     | 45           | 44           | 46.7   |       | 45     | 47     |       |
| Gsb                     | 2.679  | 2.834        | 2.806        | 2.669  | 2.799 | 2.776  | 2.848  | 2.846 |
| Gse                     | 2.717  | 2.886        | 2.855        | 2.704  | 2.881 | 2.850  | 2.893  | 2.944 |
| Total %AC               | 6.9    | 5.8          | 5.8          | 6.4    | 7.5   | 7.1    | 7.4    | 6.0   |
| Vbe                     | 14.6   | 12.6         | 12.5         | 13.5   | 15.3  | 14.5   | 16.3   |       |
| %Gmm at initial         | 87.2   | 86.7         | 86.9         | 86.9   |       | 85.5   | 86.8   |       |
| F/E                     | 1.4    | 2.1          | 2.0          | 1.6    | 1.1   | 1.1    | 1.1    | 0.8   |
| Gmm                     | 2.440  | 2.612        | 2.588        | 2.451  | 2.539 | 2.532  | 2.553  | 2.648 |
| Gmb                     | 2.346  | 2.497        | 2.473        | 2.353  | 2.412 | 2.399  | 2.453  |       |
| Voids                   | 3.9    | 4.4          | 4.4          | 4.0    | 5.0   | 5.3    | 4.0    |       |
| VMA                     | 18.5   | 17.0         | 16.9         | 17.5   | 20.3  | 19.7   | 20.3   |       |
| VFA                     | 79.1   | 74.1         | 73.8         | 77.1   | 75.3  | 73.4   | 80.8   |       |
| Pbe                     | 6.4    | 5.2          | 5.2          | 5.9    | 6.5   | 6.2    | 6.7    | 4.9   |
| adj SA                  | 43.9   | 41.6         | 39.3         | 44.6   | 37.2  | 37.8   | 38.4   | 20.4  |
| adj AFT                 | 8.6    | 7.3          | 7.5          | 7.9    | 9.8   | 9.2    | 10.1   | 12.0  |
| % new AC                | 100    | 100          | 86.2         | 84.4   | 100   | 85.9   | 100    | 100   |
| % RAP                   | 0      | 0            | 17           | 20     | 0     | 20     | 0      | 0     |
| TSR                     | 88.8   | 92.4         |              | 86.8   | 82.9  |        | 82.0   |       |
| Drain down              | 0      | 0            | 0            | 0      | 0     | 0      | 0      | 0     |
| CAA 1face               |        |              |              |        |       |        |        | 100   |
| CAA 2face               |        |              |              |        |       |        |        | 100   |
| Flat-Elongated          |        |              |              |        |       |        |        | 13    |
| Unit Wt at 7.0% AV, pcf | 146.2  | 156.5        | 155.0        | 146.8  | 152.1 | 151.7  | 152.9  | 158.6 |

#### *Tensile Strength of 4.75-mm SMA Mixtures*

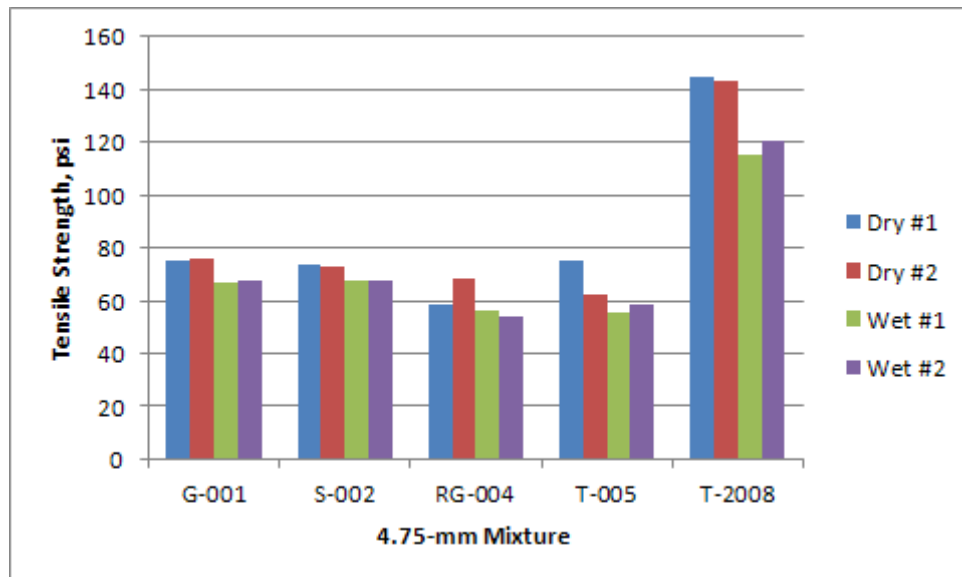
All mixtures tested at tensile strength ratios (TSR) above 80 percent. Mixture tensile strength results are shown in Table 41 and Figure 40. Most notable is the pronounced difference between the high strength values obtained for the MnROAD mixture (T-2008) and T-005. Factors contributing to the differences may be:



- Asphalt binders were produced from different production runs;
- Different proportion of taconite tailings to manufactured sand (65:35 for T-2008; and 55:45 for T-005);
- Binders with similar PG grade categories will differ in actual grades and modification levels; and
- Coarse taconite tailings, although similar in gradation and aggregate properties, were from two different producers and production runs.

**Table 41. Tensile Strength Results, psi.**

| Test Trial  | G-001 | S-002 | RG-004 | T-005 | T-2008 |
|-------------|-------|-------|--------|-------|--------|
| Dry #1      | 75.0  | 73.4  | 58.4   | 75.5  | 144.5  |
| Dry #2      | 76.0  | 72.8  | 68.0   | 62.0  | 143.1  |
| Wet #1      | 66.9  | 67.7  | 56.1   | 55.8  | 115    |
| Wet #2      | 67.2  | 67.3  | 53.6   | 58.2  | 120.7  |
| TSR Voids,% | 8.7   | 8.7   | 9.1    | 9.1   | 9.1    |



**Figure 40. Tensile strength measurements.**

## MIXTURE TESTING

The mixtures were evaluated with additional methods after the completion of the 4.75-mm mixture designs, including:

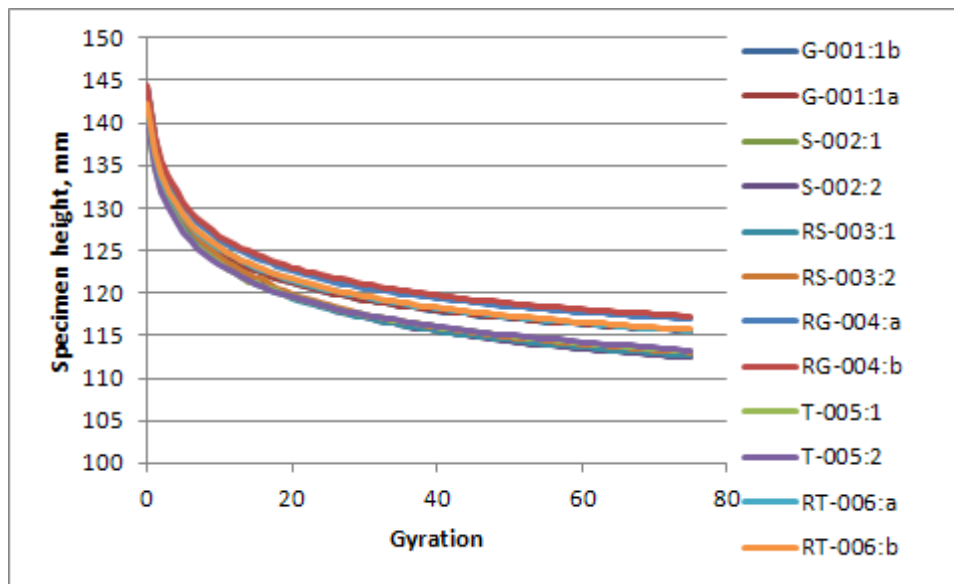
- Work of Compaction (MnDOT Lab);
- Mixture Permeability (MnDOT Lab);

- Asphalt Pavement Analyzer (APA) Rut Testing (MnDOT Lab);
- Dynamic Modulus (MnDOT Lab);
- Semi-Circular Bend (SCB) Fracture Testing (University of Minnesota Lab);
- Indirect Tensile Test (IDT) Fracture Testing (University of Minnesota Lab); and
- Related Fatigue Testing (Iowa State University).

### Mixture Compaction

The 4.75-mm mixtures were compared using compaction records from the gyratory compactor when producing specimens to design conditions. A total of twelve (12) specimens were evaluated. A data table is included as an Appendix.

75 standard gyrations (600 kPa and an internal angle of 1.16°) were applied to 150-mm diameter specimens. Figure 41 plots specimen height versus gyration number for the 4.75-mm mixtures. From the figure, it is apparent that the rate of densification varies between mixtures.



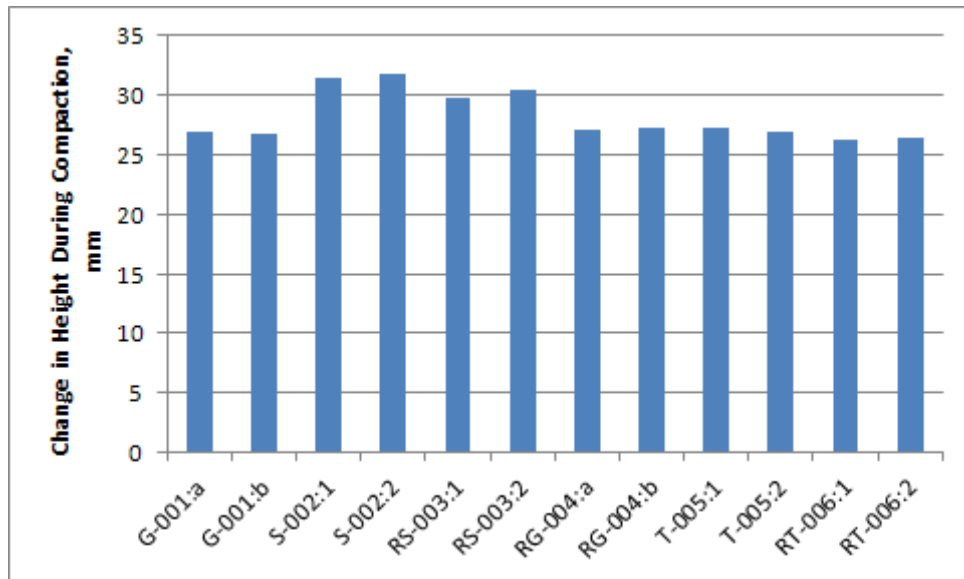
**Figure 41. Mixture densification curves for 4.75-mm mixtures, max 75 gyrations.**

Table 42 and Figure 42 show changes in specimen dimensions and air voids due to compaction. Change in specimen height is proportional to the work of compaction required to densify the mixture. Comparison shows the two 4.75-mm SMA mixtures had the greatest initial volume and required the greatest amount of compaction to achieve volumetric design conditions. The SMA mixtures also had the highest design air void content. The compaction requirements of the remaining four mixtures were all similar. The taconite tailings mixture experienced the greatest amount of air void reduction overall.

A benchmark of 99.8% design voids was also used to compare the rate of compaction. It was found that the granite mixtures (G-001 and RG-004) were fastest to achieve the benchmark, and the taconite tailings mixture (T-005) was the slowest.

**Table 42. Comparison of Laboratory Compaction.**

| 4.75-mm Mixture: Specimen | Delta H, mm | H final/H initial | Delta %Air Voids | Delta %Gmm | Delta %Design Voids | Gyrations to 99.8% Design Voids |
|---------------------------|-------------|-------------------|------------------|------------|---------------------|---------------------------------|
| G-001:a                   | -26.9       | 0.1888            | -0.9             | 0.9        | 0.9%                | 21                              |
| G-001:b                   | -26.8       | 0.1883            | -0.9             | 0.9        | 0.9%                | 20                              |
| S-002:1                   | -31.5       | 0.2186            | -1.2             | 1.2        | 1.3%                | 30                              |
| S-002:2                   | -31.8       | 0.2205            | -1.2             | 1.2        | 1.3%                | 30                              |
| RS-003:1                  | -29.7       | 0.2086            | -1.2             | 1.2        | 1.2%                | 33                              |
| RS-003:2                  | -30.4       | 0.2120            | -1.2             | 1.2        | 1.2%                | 33                              |
| RG-004:a                  | -27.1       | 0.1881            | -0.9             | 0.9        | 1.0%                | 25                              |
| RG-004:b                  | -27.2       | 0.1882            | -0.9             | 0.9        | 1.0%                | 26                              |
| T-005:1                   | -27.2       | 0.1937            | -1.3             | 1.3        | 1.3%                | 37                              |
| T-005:2                   | -27         | 0.1924            | -1.3             | 1.3        | 1.3%                | 36                              |
| RT-006:1                  | -26.3       | 0.1853            | -1.1             | 1.1        | 1.2%                | 33                              |
| RT-006:2                  | -26.5       | 0.1864            | -1.1             | 1.1        | 1.2%                | 33                              |



**Figure 42. Change in specimen height due to gyratory compaction.**

## Permeability

Permeability measurements were obtained in the laboratory using a 4-in. diameter Karol-Warner Flexible Wall Permeameter. The laboratory permeameter was capable of maintaining pressure along the cylindrical specimen side, creating a vertical path for water movement (Figure 43). These conditions are suitable for a falling-head measurement of permeability in one-dimension.

Cylindrical specimens were conditioned in water then placed in the permeameter at a side pressure of 10-14 psi and tested under falling-head conditions. Results were corrected for permeability at 20°C and reported in terms of cm/s. A total of eight (8) specimens were evaluated.



**Figure 43. Laboratory flexible-wall asphalt permeameter.**

Permeability of an asphalt mixture can depend on air void content and mixture type, so the 4.75-mm mixtures were tested and compared with a 12.5-mm dense graded control mixture from Minnesota. The 12.5-mm mixture was evaluated at 4 and 7% air voids while the 4.75-mm mixtures were evaluated within a range of 6.9 to 10.3% air voids (Table 43). This range is similar to the air voids resulting from field compaction of an asphalt mat. Specimen heights for the 12.5-mm mixture were approximately 30 percent less than for the 4.75-mm mixtures.

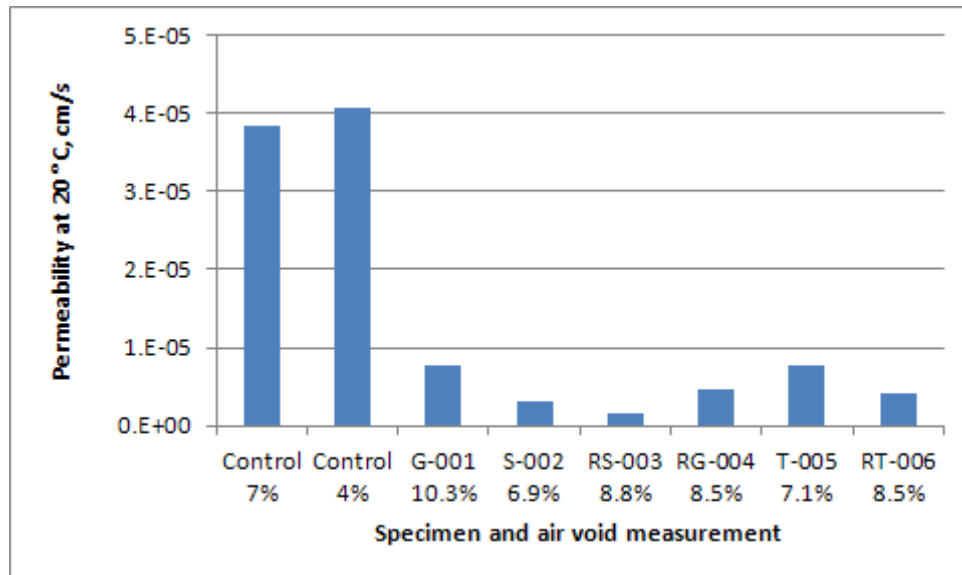
Results showed that the permeability of the 12.5-mm mixture specimens was approximately  $4E-5$  cm/s, and the 4.75-mm mixtures were all below  $7.7E-6$  cm/s. The reduction in permeability was 88% on average. Although MnDOT currently has no established criteria related to good or

bad performance in permeability testing, other research has proposed threshold values in the range of 1.25E-2 (Cooley et al., 2002; Maupin, 2001). All of the mixtures fell below the threshold.

Note that all of the 4.75-mm mixtures containing RAP tested as less permeable than their virgin aggregate counterparts. The regression of permeability as a function of air voids produced an R-squared value of 0.035, suggesting that air void levels contributed less to differences between 4.75-mm mixtures than other mix properties or measurement error sources.

**Table 43. Laboratory Permeability (k) of 4.75-mm and 12.5-mm Asphalt Mixtures.**

| Mixture ID      | Air Void, % | Diameter, mm | Height, mm | k at 20 °C, cm/s | Average k | % Reduction of k | Average Reduction |
|-----------------|-------------|--------------|------------|------------------|-----------|------------------|-------------------|
| 12.5-mm Control | 7.0         | 101.0        | 66.8       | 3.84E-05         | 3.95E-05  | 0.0%             | 0.0%              |
|                 | 4.0         | 101.0        | 66.8       | 4.06E-05         |           |                  |                   |
| G-001           | 10.3        | 100.7        | 96.8       | 7.68E-06         | 4.25E-06  | 81%              | 88%               |
| S-002           | 6.9         | 101.5        | 99.5       | 3.13E-06         |           | 92%              |                   |
| RS-003          | 8.8         | 100.6        | 94.0       | 1.50E-06         |           | 96%              |                   |
| RG-004          | 8.5         | 100.5        | 98.0       | 4.71E-06         |           | 88%              |                   |
| T-005           | 7.1         | 101.5        | 82.7       | 7.68E-06         |           | 81%              |                   |
| RT-006          | 8.5         | 100.2        | 98.2       | 4.23E-06         |           | 89%              |                   |



**Figure 44. Laboratory permeability of 4.75-mm and 12.5-mm mixtures.**

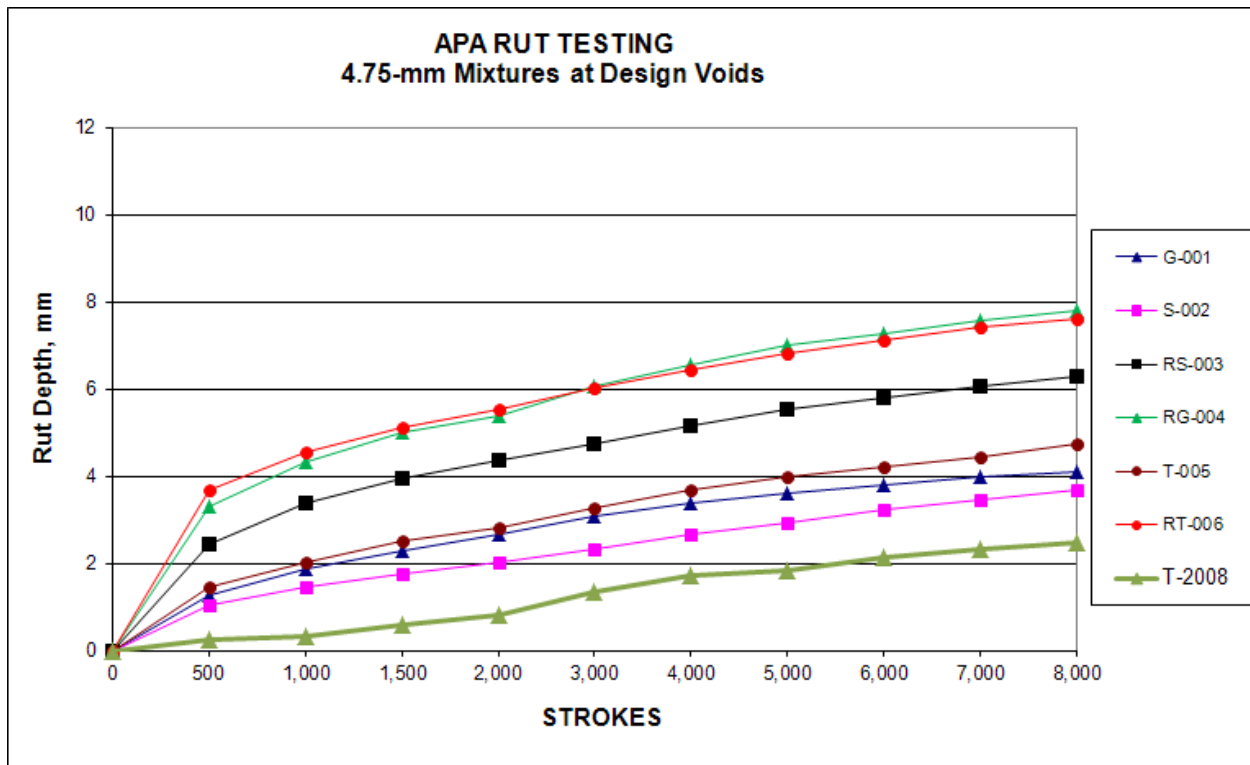
### Asphalt Pavement Analyzer (APA) Testing

APA testing was performed on the six 4.75-mm mixtures. During APA testing mixture specimens are placed in a sealed chamber and heated to a desired temperature. Mixture susceptibility to rutting is tested by applying successive wheel passes at a constant load of 100 lb per stroke (MnDOT, 2000, 2011; Olson et al., 2006; Skok et al., 2002). The constant load was applied to 1-in. diameter hose pressurized to 100 psi. A total of three (3) specimens per mixture were tested at design air void and at 137°F (58°C), which conforms to the high-temperature for the standard Superpave performance grade for Minnesota.

APA results show that the mixtures containing RAP and PG 49-34 asphalt binder generally developed the most rutting, and performed similar to MnDOT Traffic Level 2 and 3 designs. Mixtures containing PG 64-34 asphalt binder and no RAP generally performed similar to MnDOT Traffic Level 3 and 4 designs.



**Figure 45. Example of APA specimens after testing. Clockwise from upper left: Mixture RG-004, T-005, RT-006, RS-003, S-002, and G-001.**



**Figure 46. Asphalt Pavement Analyzer test results for 4.75-mm mixtures, max 8,000 strokes.**

**Table 44. Rut Depth (mm) at APA Stroke Count.**

| Mixture | Stroke Count  |      |      |      |      |      |      |      |      |      |       |
|---------|---------------|------|------|------|------|------|------|------|------|------|-------|
|         | 0             | 500  | 1000 | 1500 | 2000 | 3000 | 4000 | 5000 | 6000 | 7000 | 8000* |
|         | Rut Depth, mm |      |      |      |      |      |      |      |      |      |       |
| G-001   | 0.00          | 1.28 | 1.87 | 2.28 | 2.65 | 3.07 | 3.37 | 3.61 | 3.81 | 3.98 | 4.11  |
| S-002   | 0.00          | 1.04 | 1.45 | 1.77 | 2.01 | 2.33 | 2.65 | 2.93 | 3.24 | 3.46 | 3.69  |
| RS-003  | 0.00          | 2.45 | 3.37 | 3.95 | 4.36 | 4.75 | 5.15 | 5.54 | 5.80 | 6.07 | 6.29  |
| RG-004  | 0.00          | 3.31 | 4.32 | 5.00 | 5.39 | 6.05 | 6.55 | 6.99 | 7.28 | 7.58 | 7.81  |
| T-005   | 0.00          | 1.45 | 2.01 | 2.50 | 2.80 | 3.28 | 3.69 | 3.97 | 4.20 | 4.45 | 4.72  |
| RT-006  | 0.00          | 3.68 | 4.54 | 5.11 | 5.53 | 6.02 | 6.44 | 6.81 | 7.10 | 7.40 | 7.60  |
| T-2008  | 0.00          | 0.25 | 0.33 | 0.58 | 0.81 | 1.35 | 1.70 | 1.84 | 2.14 | 2.33 | 2.49  |

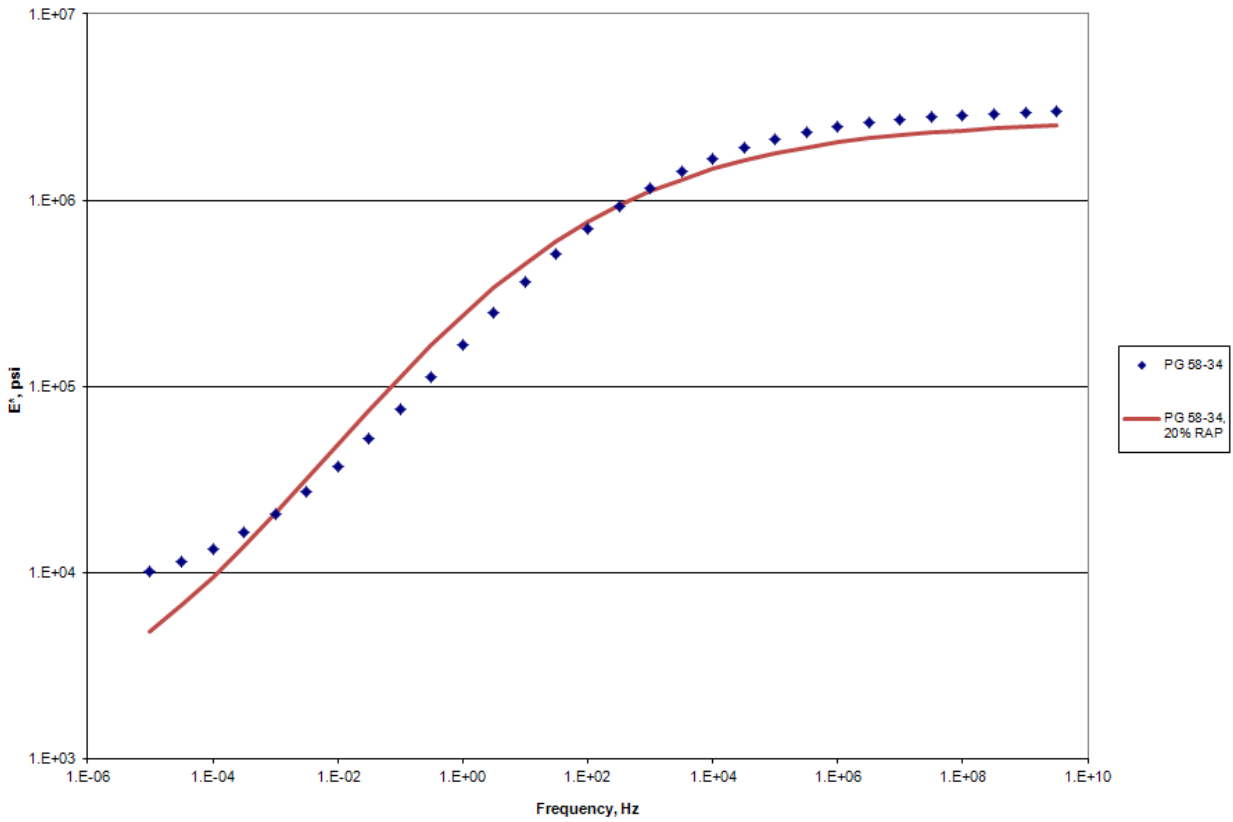
(\*) Maximum 8,000 strokes

### Dynamic Modulus

Dynamic modulus ( $|E^*|$ ) testing was performed on the 4.75-mm mixtures. A total of 24 specimens were produced for the 6 mixtures at a targeted 8% air void content.  $|E^*|$  testing evaluates mixtures response to cyclic loading at five temperatures and six frequencies. In this work the test frequencies included 0.1, 0.5, 1, 5, 10, and 25 Hz. The set of test temperatures was -10, 4.4, 21.1, 37.8, and 54.4°C. Sigmoidal-shaped master curves were fitted to a reference temperature of 21.1°C.

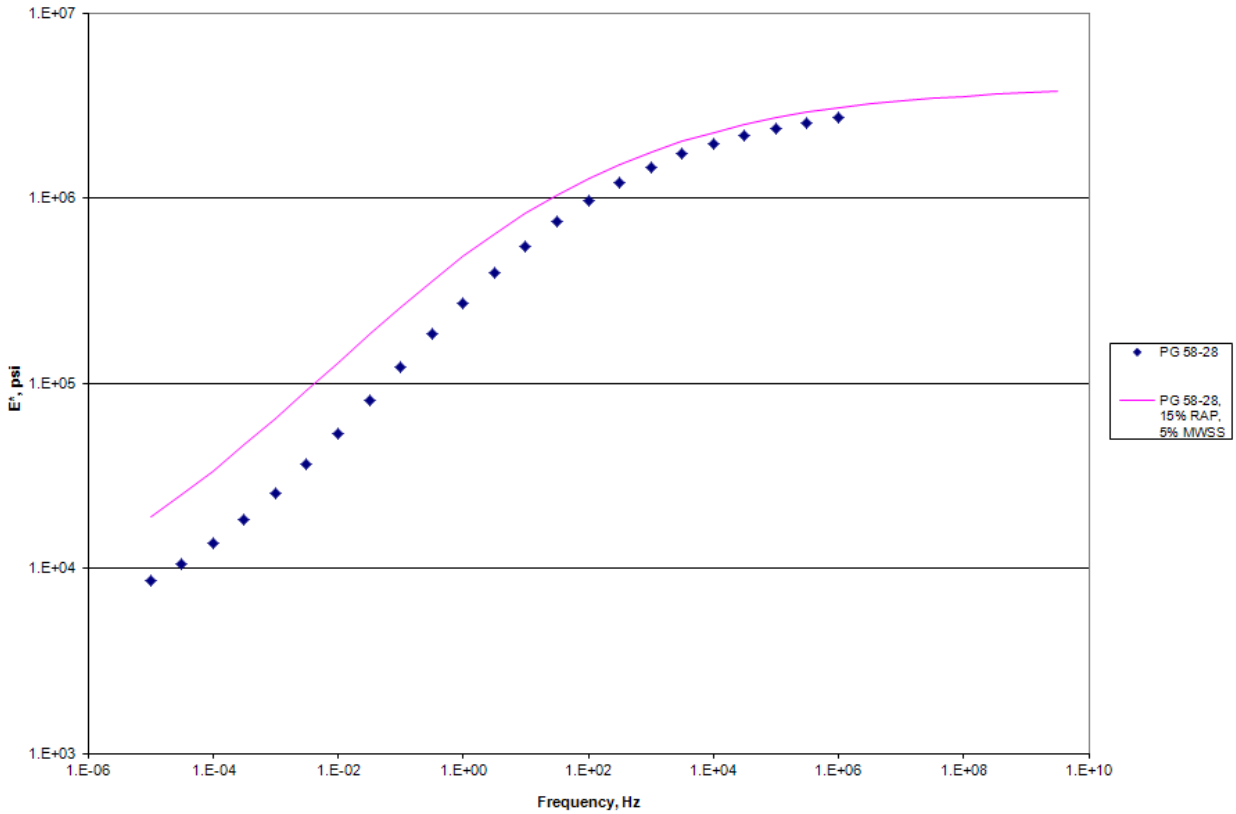
Figure 47 and Figure 48 are plots of master curves for two different 12.5-mm mixtures that contain 0% and 20% recycled material. The curves are presented here as a frame of reference regarding the performance of the 4.75-mm mixtures and the effect of RAP on asphalt mixtures.

The curves in Figure 47 were generated from field cores of a county highway research project, and the curves in Figure 48 were generated from gyratory specimens mixed in the laboratory. The plots show that it is difficult to precisely anticipate the effect of RAP. From the plots, it can be seen that significant stiffening occurred for laboratory mixtures using PG 58-28 asphalt binder. There was less separation in the performance of the PG 64-34 field samples except that extreme high and low frequency ranges showed that mixture softening had occurred. In the case of the 12.5-mm mixtures no extra sizing was done for RAP material. RAP was processed and screened to a maximum size matching the maximum size of virgin aggregates in the blend.



**Figure 47. Dynamic Modulus master curve of 12.5-mm mixtures with and without RAP: PG 58-34.**





**Figure 48. Dynamic Modulus master curve for 12.5-mm mixtures with and without RAP & shingles: PG 58-28.**

Fitted master curves for individual  $E^*$  specimens are presented in the top half of Figure 49 through Figure 54.  $E^*$  data points for the specimens are presented in the lower half of the figures, and are plotted along with the master curve fitted to the entire set of data points. The figures show that portions of the fitted master curves extrapolated beyond the measured data. The data points from individual specimens and the degree to which fitted curves overlap both illustrate the level of variability for a particular mixture.

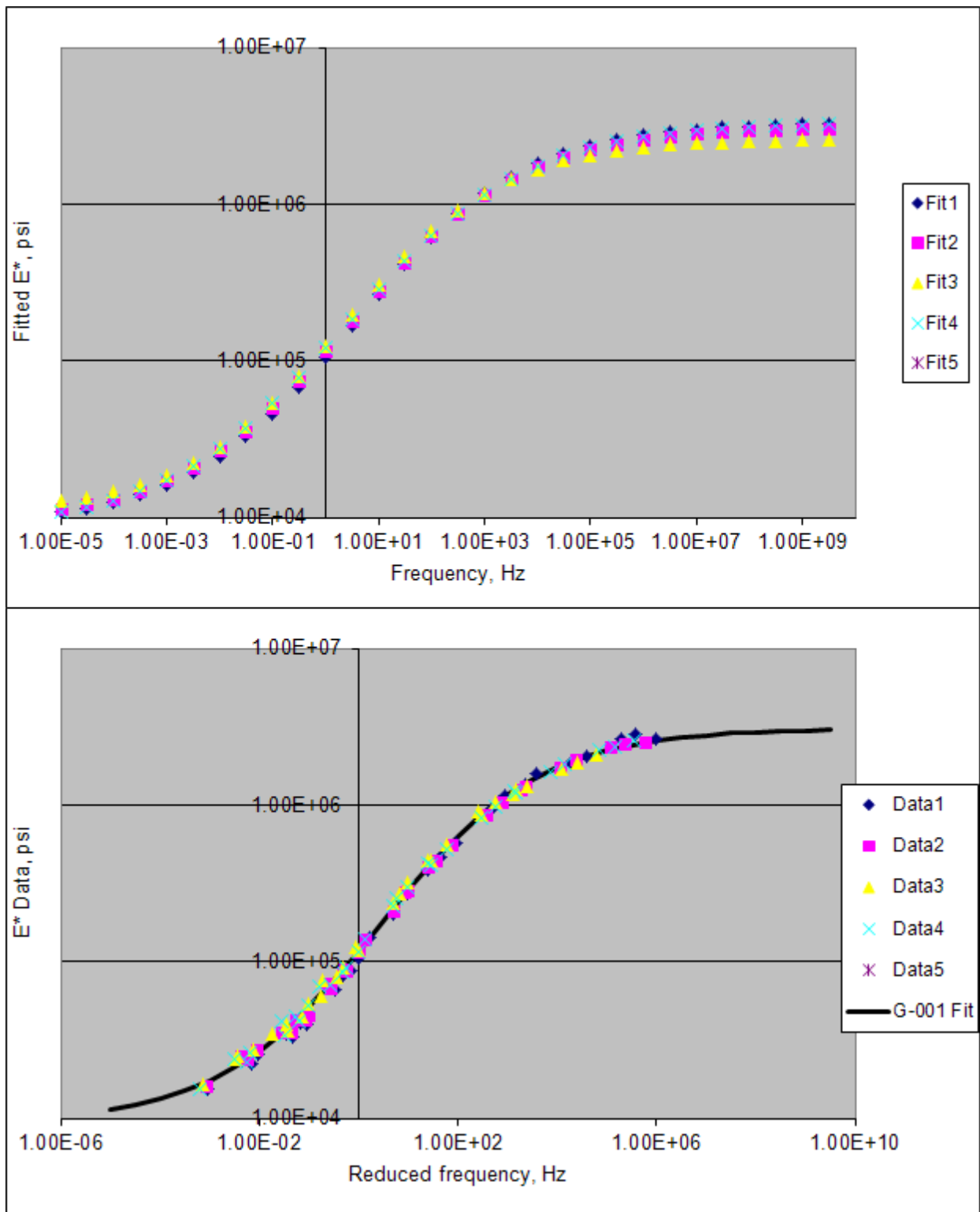
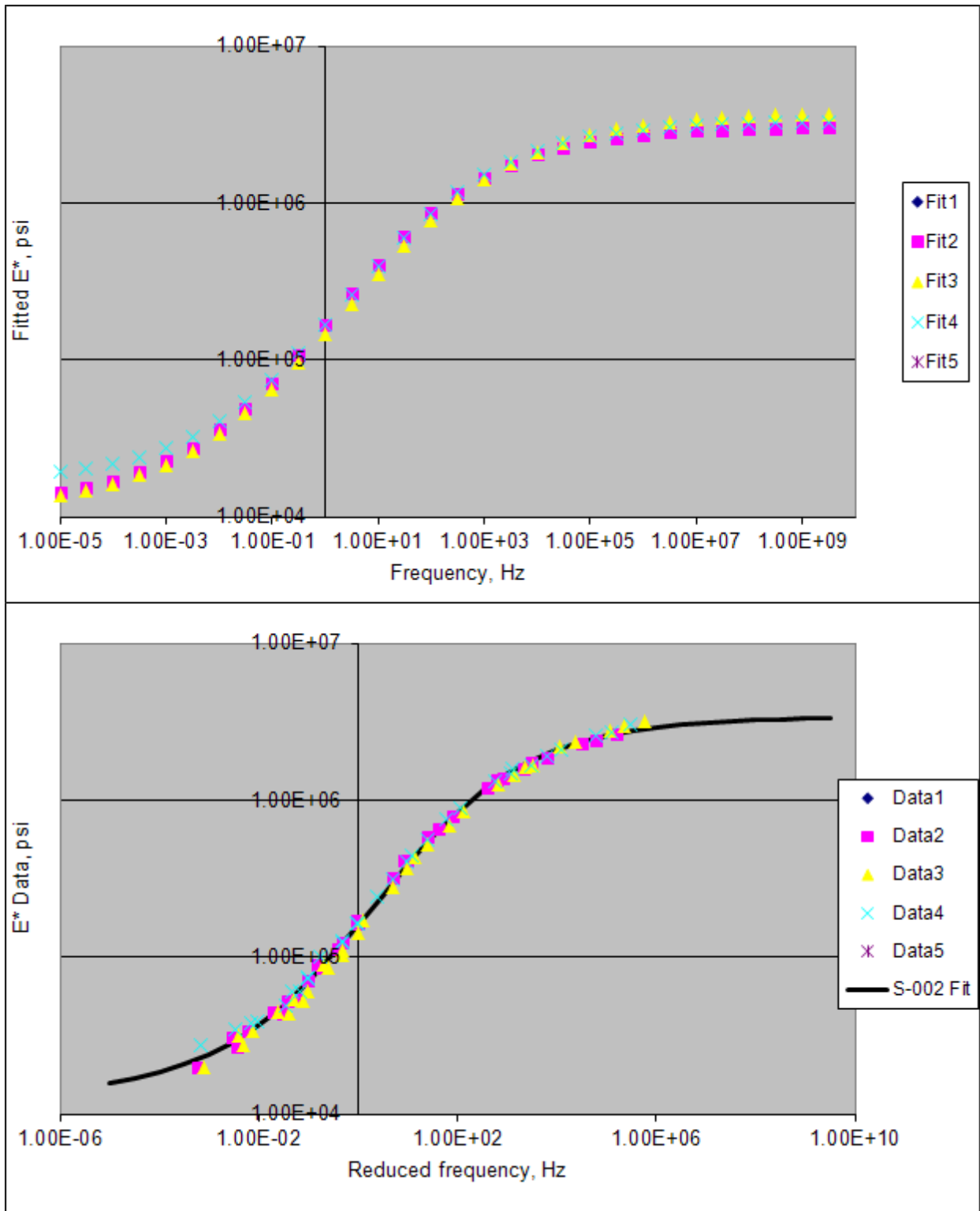
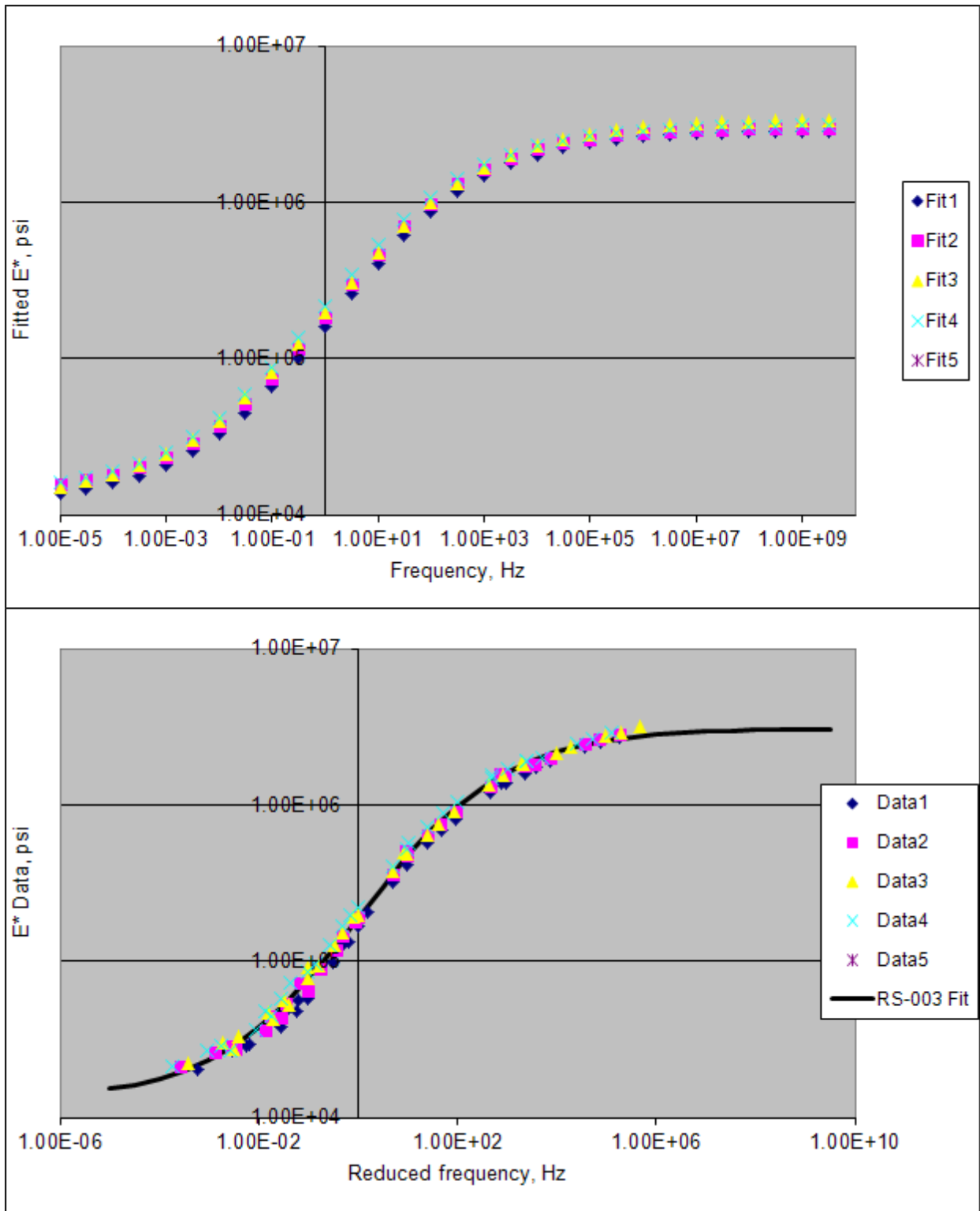


Figure 49. E\* Specimen master-curve and data: G-001.



**Figure 50. E\* Specimen master-curve and data: S-002.**



**Figure 51.  $E^*$  Specimen master-curve and data: RS-003.**

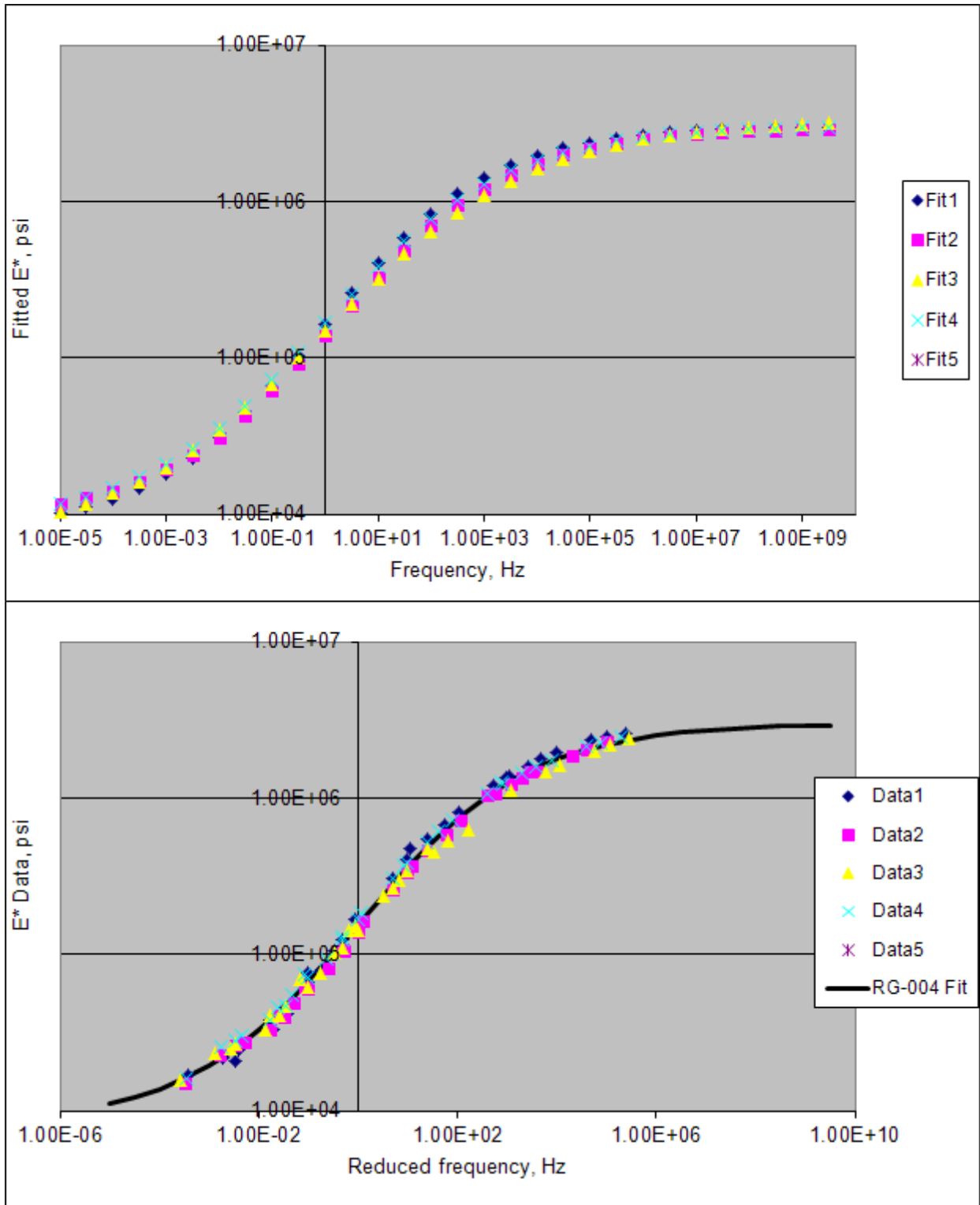
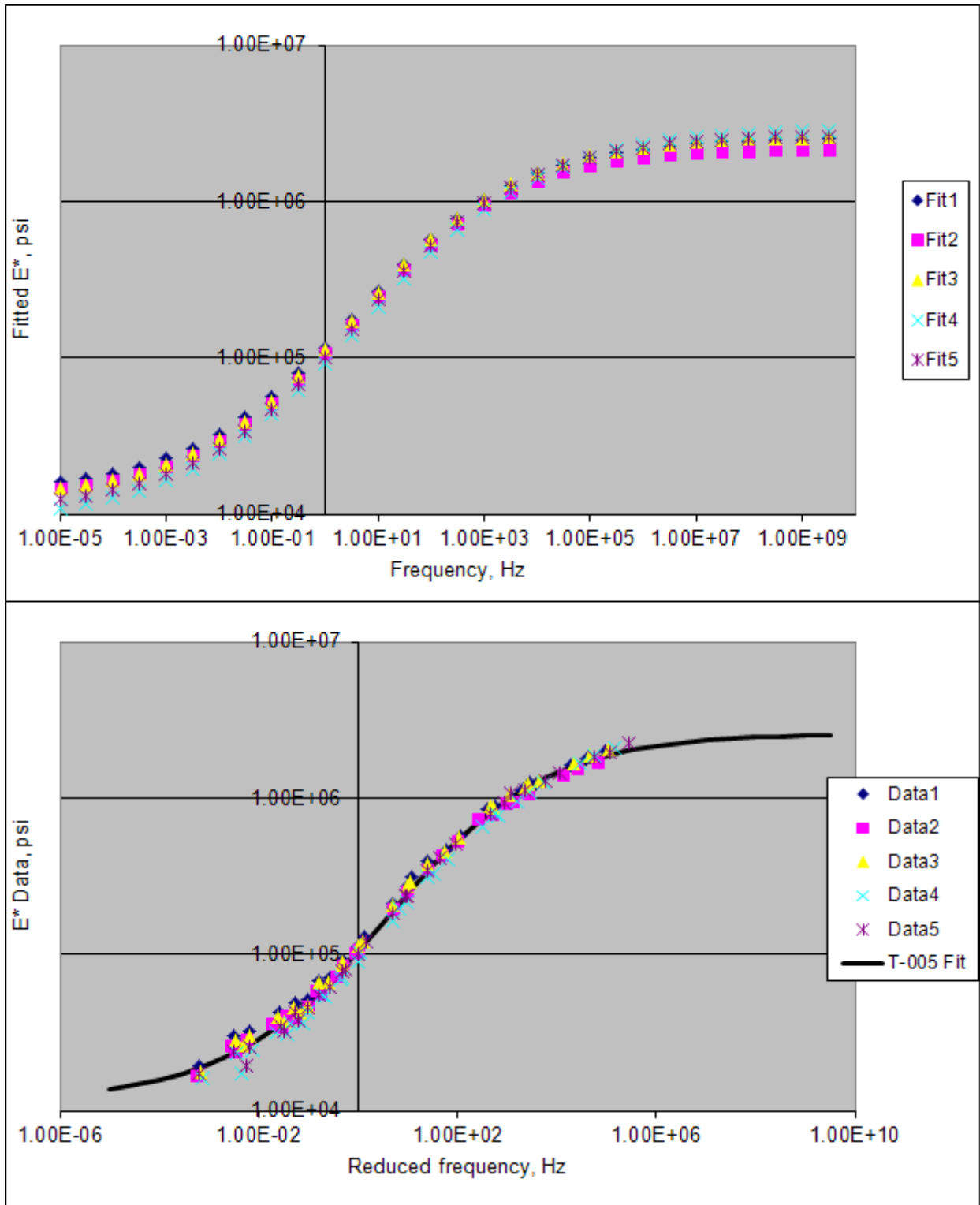
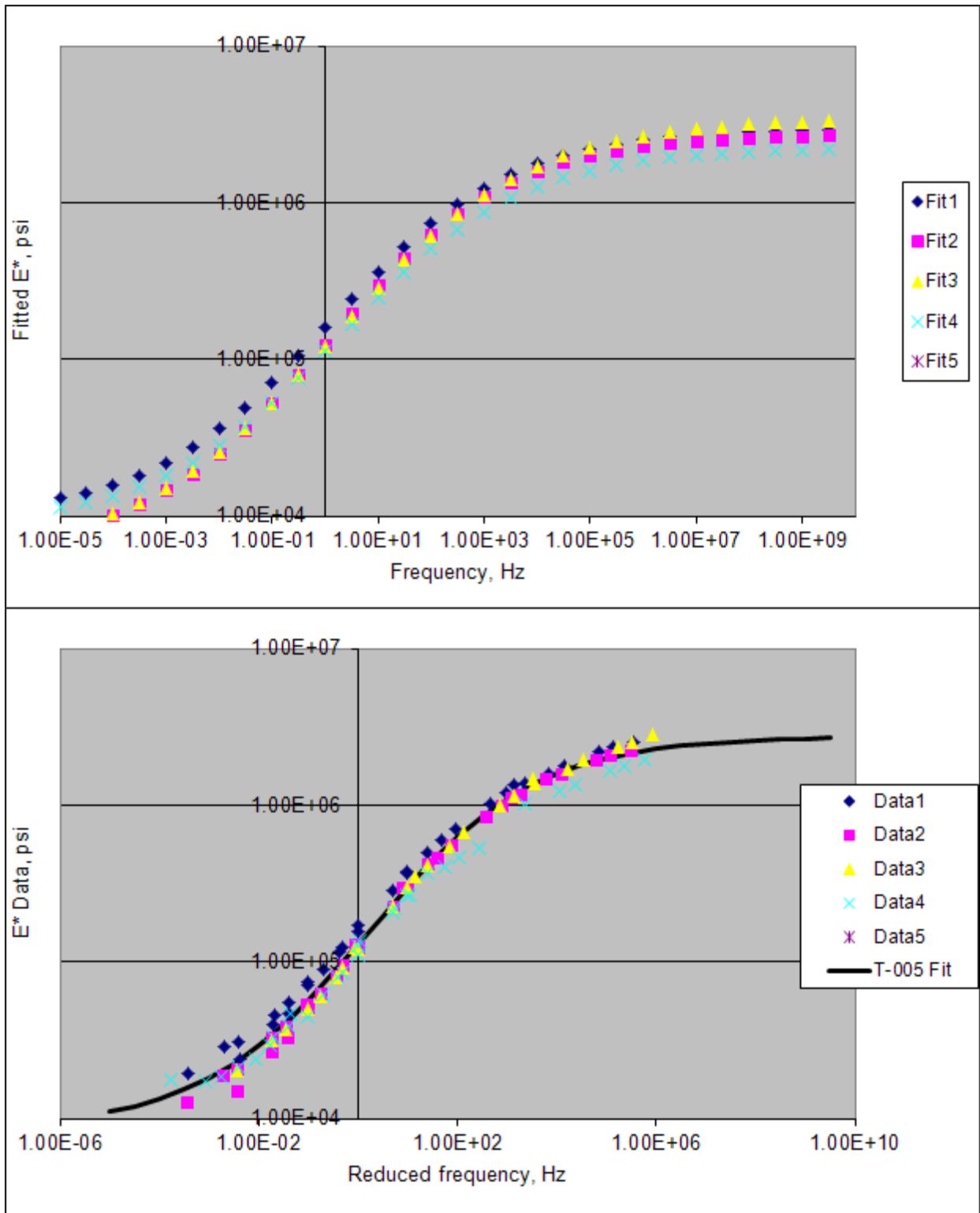


Figure 52.  $E^*$  Specimen master-curve and data: RG-004.

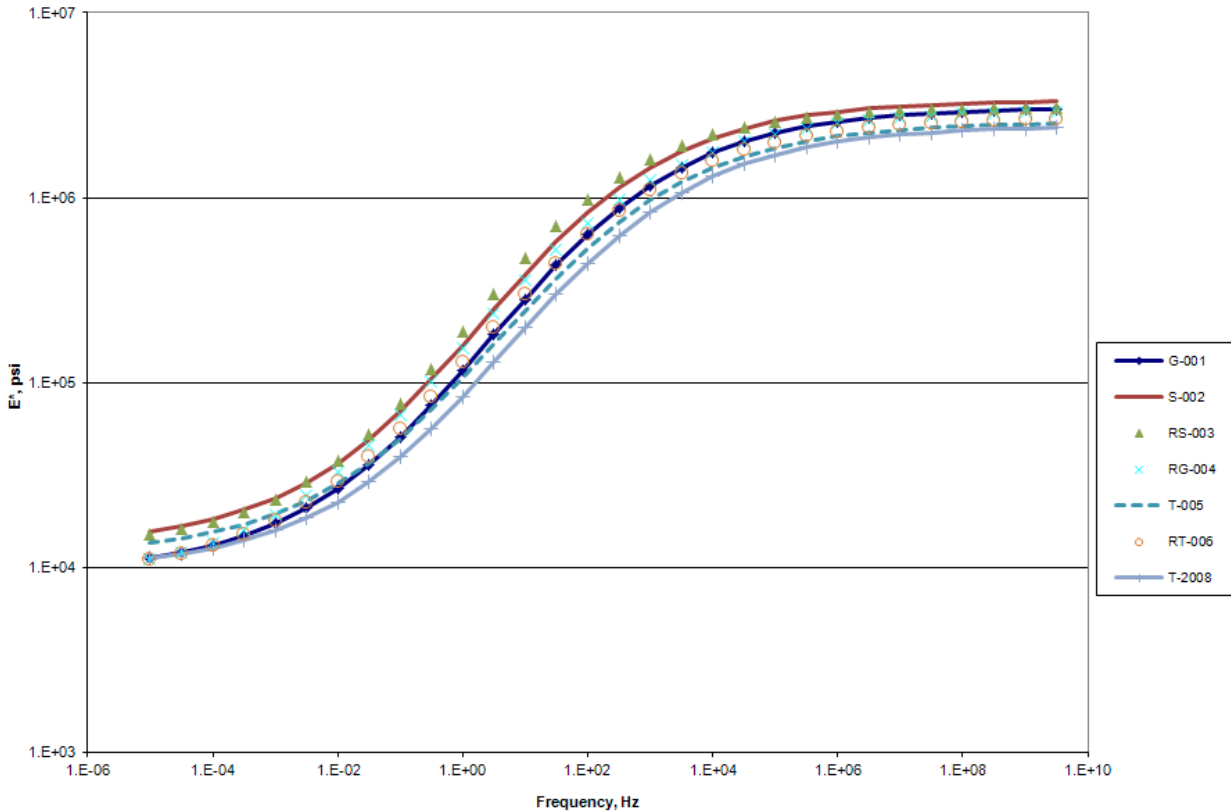


**Figure 53. E\* Specimen master-curve and data: T-005.**



**Figure 54. E\* Specimen master-curve and data: RT-006.**

Master curves for the set of six 4.75-mm mixtures are given in Figure 55, along with the master curve for T-2008.



**Figure 55. Dynamic Modulus master curve for 4.75-mm mixtures.**

Performance of the 4.75-mm mixtures in Figure 55 showed relatively similar  $|E^*|$  master curves between the six mixtures. The following observations were made based on  $|E^*|$  data:

- The virgin aggregate SMA mixture containing taconite tailings was stiffer than the dense-graded control mixture (PG's 64-34);
- The SMA and RAP mixture showed the stiffest performance along the majority of fitted data points (PG 49-34);
- The granite and RAP mixture showed similar performance compared to the virgin SMA along the majority of the fitted points, but was softer at SMA modulus values below 100 kpsi (PG 49-34 versus 64-34);
- The six mixtures were all stiffer than the MnROAD T-2008 mixture;
- Contrary to expectations, large differences were not observed between RAP and non-RAP mixtures. This performance may be due to utilization of the fine RAP fraction, compatible RAP and virgin asphalt binder, or the interaction of other mixture parameters; and
- $E^*$  values for RAP versus non-RAP mixtures in the low frequency (high temperature) portion of the master curve shows that similar performance may be obtained when substituting an appropriate softer high-PG binder along with RAP.



## DISCUSSION

Outcomes of this study suggest that Mesabi rock and tailings products show promise as components of 4.75-mm Dense-graded, Stone Matrix Asphalt, and Ultra-Thin Bonded Wearing asphalt mixtures. Laboratory and field investigations of taconite tailings should continue.

The Mesabi rock can be incorporated in standard Superpave, SMA, and fine/sand asphalt mixtures in upcoming construction projects. In each case construction and long term field performance should be evaluated.

Results support that in order to ensure adequate mixture performance, care should be applied when using RAP. RAP and aggregates may also benefit from extra margins of gradation control.

Material control enhancements provide benefits whenever they are available. Efforts should be made to promote the use of washing and sizing of taconite tailing and RAP stockpiles.

Differences were found between the coarse tailings mixtures produced in 2008 and 2011. It should not be assumed that all tailings mixtures are alike, and will yield similar laboratory or field performance, because each taconite mine will generate tailings having a gradation curve specific to that particular operation. Other work should include additional evaluation of mixture performance in fatigue and moisture susceptibility tests for 4.75-mm designs containing a range of materials from various sources.

Agencies should be able to specify similar mixtures with only minor changes to construction specifications.

### Design Observations:

- 4.75-mm dense-graded:
  - Gradation: current broadband recommendations are appropriate;
  - Use of RAP is acceptable, but does affect cracking resistance of mixtures;
  - Use of 4.0 design voids is adequate. Designers should also track AFT, mixture volumetric properties, and effective AC; and
  - TSR of 80 is realistic expectation.
  
- 4.75-mm SMA:
  - Gradation: current broadband recommendations are appropriate;
  - Use of RAP is acceptable, but does affect cracking resistance of mixtures;
  - Use of fiber may not be required. None of the SMA or dense-graded mixtures exhibited drain-down issues;
  - Use of 5.0 design voids, or range from 4.0 – 6.0, is adequate. Designers should also track AFT, mixture volumetric properties, and effective AC;
  - Design criteria were satisfied using a total AC percentage of 5.8 percent. This percentage is regarded as somewhat low in comparison to SMA's designed with larger sized aggregate. Low AC levels cause concern about durability, and alternate designs should be explored; and

- TSR of 80 is realistic expectation.
- Ultra-Thin Bonded Wear:
  - MnDOT Gradation is adequate. Better overall economy may be obtained by designing toward the coarse limit;
  - RAP not used; and
  - Design voids, AFT, Effective AC.
- All mixtures were acceptable for use in wear-course applications:
  - Traffic Level 2-3: All mixtures PG 49-34, PG 64-34, 0-20% RAP; and
  - Traffic Level 4: Mixtures PG 64-34, 0% RAP.

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## APPENDIX A

### Selected Excerpts from MnDOT 2360 (Superpave) Asphalt Mixture Requirements, February 4, 2011.

| <b>Table 2360-1<br/>Traffic Levels</b>  |                             |
|---|-----------------------------|
| <b>Traffic Level</b>  | <b>20 Year Design ESALs</b> |
| 2 *   | < 1                         |
| 3   | 1 - < 3                     |
| 4   | 1 - < 10                    |
| 5   | 10 - ≤ 30                   |
| NOTE: The requirements for gyratory mixtures in this specification are based on the 20 year design traffic level of the project, expressed in Equivalent Single Axle Loads (ESAL's) $1 \times 10^6$ ESALs<br>* AADT < 2,300<br>   AADT > 2,300 to < 6,000 |                             |

(5) The last two digits indicate the air void requirement:

(5.1) 40 = 4.0 percent for wear mixtures, and

(5.2) 30 = 3.0 percent for non-wear and shoulder.

(6) The letter at the end of the mixture designation identifies the asphalt binder grade in accordance with Table 2360-2, "Asphalt Grades."

| <b>Table 2360-2<br/>Asphalt Grades</b> |              |
|--|--------------|
| <b>Letter</b>                          | <b>Grade</b> |
| A                                      | PG 52 - 34   |
| B                                      | PG 58 - 28   |
| C                                      | PG 58 - 34   |
| E                                      | PG 64 - 28   |
| F                                      | PG 64 - 34   |
| H                                      | PG 70 - 28   |
| L                                      | PG 64 - 22   |

Ex: Gyratory Mixture Designation -- SPWEB540E (Design Type, Lift, Aggr. Size, Traffic Level, Voids, Binder)

**2360.2 MATERIALS**

**A Aggregate**

Use aggregate materials in accordance with 3139.2.

**B Asphalt Binder Material ..... 3151**

| <b>Table 2360-3<br/>Asphalt Binder Selection Criteria for all Mixtures with RAP</b>   |                     |                       |
|---|---------------------|-----------------------|
| <b>Asphalt Binder Selection Criteria<br/>for all Mixtures with RAP<br/>Specified PG Asphalt Binder<br/>Grade</b>  | <b>≤ 20 % RAP</b>   | <b>&gt; 20 % RAP*</b> |
| PG XX-28 and PG 52-34   | Use specified grade | Use specified grade   |
| PG XX-34  | Use specified grade | Use blending chart*   |
| * Use the blending chart on file with the Mn/DOT Chemical Laboratory to verify compliance with the specified binder grade when RAP is greater than 20 percent. The Department may take production samples to ensure the the asphalt binder material meets the requirements. |                     |                       |

**E.5.a(1) Aggregate**

| <b>Table 2360-4<br/>Aggregate Sample Size</b> |                             |  |
|---|-----------------------------|--|
| <b>Classification</b>                         | <b>Sieve</b>                | <b>Weight</b>                                      |
| Virgin  | Retained on No. 4 [4.75 mm] | 80 lb [35 kg]                                      |
| Virgin  | Passing No. 4 [4.75 mm]     | 35 lb [15 kg]                                      |
| Recycled asphalt pavement (RAP)               | —                           | 80 lb [35 kg]                                      |
| Recycled asphalt shingles (RAS)               | —                           | 10 lb [5 kg] sample of representative RAS material |

**E.6 Mixture Requirements**

The Department will base mixture evaluation on the trial mix tests and in accordance with Table 2360-7, “Mixture Requirements.”

| <b>Table 2360-7<br/>Mixture Requirements</b>   |                |                |                |                 |
|--|----------------|----------------|----------------|-----------------|
| <b>Traffic Level</b>   | <b>2</b>       | <b>3</b>       | <b>4</b>       | <b>5</b>        |
| 20 year design ESALs   | < 1 million    | 1 – 3 million  | 3 – 10 million | 10 – 30 million |
| Gyratory mixture requirements:   |                |                |                |                 |
| Gyrations for $N_{design}$   | 40             | 60             | 90             | 100             |
| % Air voids at $N_{design}$ , wear   | 4.0            | 4.0            | 4.0            | 4.0             |
| % Air voids at $N_{design}$ , Non-wear and all shoulder  | 3.0            | 3.0            | 3.0            | 3.0             |
| Adjusted Asphalt Film Thickness, minimum $\mu$   | 8.5            | 8.5            | 8.5            | 8.5             |
| Ratio of Added New Asphalt Binder to Total Asphalt Binder, <sup>(1)</sup> min%   | 70             | 70             | 70             | 70              |
| TSR*, minimum %  | 75 $\parallel$ | 75 $\parallel$ | 80 $\dagger$   | 80 $\dagger$    |
| Fines/effective asphalt  | 0.6 – 1.2      | 0.6 – 1.2      | 0.6 – 1.2      | 0.6 – 1.2       |
| <p>* Use 6 in [150 mm] specimens in accordance with 2360.2.I, “Field Tensile Strength Ratio (TSR).”</p> <p><math>\parallel</math> Mn/DOT minimum = 65</p> <p><math>\dagger</math> Mn/DOT minimum = 70</p> <p><sup>1</sup> The ratio of added new asphalt binder to total asphalt binder needs to be 70% or greater ((added binder/total binder) x 100 <math>\geq</math> 70) in both mixtures that contain RAP and in mixtures that include shingles as part of the allowable RAP percentage.</p> |                |                |                |                 |

### E.7 Coarse/Fine Mixture Determination

Base the determination of coarse and fine graded mixtures on the percentage of material passing the No. 8 [2.36 mm] sieve in accordance with Table 2360-8, “Coarse/Fine Mixture Determination.”

| <b>Table 2360-8<br/>Coarse/Fine Mixture Determination</b> |  |  |
|---|--|--|
| <b>Gradation</b>  | <b>Fine Mixture,<br/>% passing No. 8 [2.36 mm]</b> | <b>Coarse Mixture,<br/>% passing No. 8 [2.36 mm]</b> |
| A   | > 47   | $\leq$ 47  |
| B   | > 39   | $\leq$ 39  |
| C   | > 35   | $\leq$ 35  |
| D   | —  | —  |

### E.8 Adjusted Asphalt Film Thickness (Adj. AFT) ..... MnDOT Laboratory Manual Method 1854

Ensure the adjusted asphalt film thickness (Adj. AFT) of the mixture at design and during production meets the requirements of Table 2360-7, “Mixture Requirements.” Base the Adj. AFT on the calculated aggregate surface area (SA) and the effective asphalt binder content.

### G.14.a Ratio of New Added Asphalt Binder to Total Asphalt Binder – Acceptance Criteria

The minimum design ratio of new added asphalt binder to total asphalt binder is 70%.

## APPENDIX B

### Select Excerpts from MnDOT S-157 (2356) ULTRATHIN BONDED WEARING COURSE (UTBWC) (2011 Version)

#### S-157.1 DESCRIPTION

This work is the construction of an ultrathin bonded wearing course on a prepared pavement. An ultrathin bonded wearing course is the application of a warm polymer modified emulsion membrane followed immediately with an ultrathin wearing course.

#### S-157.2 MATERIAL REQUIREMENTS

##### A Bituminous Materials

##### A.1 Polymer Modified Emulsion Membrane

Provide a polymer modified emulsion membrane meeting the requirements of Table 2356-1, "Polymer Modified Emulsion Membrane Requirements."

| <b>Table 2356-1</b>   |               |      |      |
|---|---------------|------|------|
| <b>Polymer Modified Emulsion Membrane Requirements</b>  |               |      |      |
| Tests on Emulsion   | AASHTO Method | Min. | Max. |
| Viscosity, Saybolt Furol @ 77°F [25°C],s  | T59           | 20   | 100  |
| Storage Stability Test <sup>1</sup> ,24 h. %  | T59           |      | 1    |
| Sieve Test  | T59           |      | 0.05 |
| Residue by Distillation <sup>2</sup> ,%   | T59           | 63   |      |
| Oil Distillate by Distillation, %   |               |      | 2    |
| Demulsibility, 35ml, 0.8% dioctyl sodium sulfosuccinate, %  | T59           | 60   |      |
| Tests on Residue From Distillation  |               |      |      |
| Penetration @ 77°F [25°C]   | T49           | 60   | 150  |
| Solubility in trichlorethylene, %   | T44           | 97.5 |      |
| Elastic Recovery, %   | T301          | 60   |      |
| <sup>1</sup> After standing undisturbed for 24 hours, the surface shall be a smooth homogeneous color throughout.<br><sup>2</sup> AASTHO T59 with modifications to include a 392°F ± 9°F [200°C±5°C] maximum temperature to be held for a period of 15 minutes. |               |      |      |

##### A.2 Asphalt Binder

Use a Performance Graded binder, PG 64-34 that meets MnDOT 3151.2A.

##### B Aggregate

**Do not use recycled materials including glass, concrete, bituminous, shingles, ash, and steel slag.**

##### B.1 Coarse Aggregate

Provide Class A aggregate, as defined in 3139.2 in Section S-\_\_ (GRADED AGGREGATE FOR BITUMINOUS MIXTURES) of these Special Provisions, that meets the requirements in Table 2356-2, "Coarse Aggregate Requirements."

| Table 2356-2<br>Coarse Aggregate Requirements |                              |  |
|---|------------------------------|--|
| Test  | Test Reference               | Limit  |
| Flat & Elongated Ratio @ 3:1, %               | MnDOT Laboratory Manual 1208 | 25 max                                       |
| LAR   | MnDOT Laboratory Manual 1210 | 40 max                                       |
| Soundness (Magnesium Sulfate)                 | MnDOT Laboratory Manual 1219 | No more than 18% loss for the composite loss |
| Bulk Specific Gravity                         | MnDOT Laboratory Manual 1204 | NA   |

### B.2 Fine Aggregate

Provide fine aggregate, passing the No. 4 [4.75 mm] sieve, meeting the requirements in Table 2356-3, "Fine Aggregate Requirements." The fine aggregate will be part of the asphalt mastic.

| Table 2356-3<br>Fine Aggregate Requirements |                              |        |
|---|------------------------------|--------|
| Test  | Test Reference               | Limit  |
| Uncompacted Void Content                    | MnDOT Laboratory Manual 1206 | 40 min |
| Sand Equivalent                             | AASHTO T 176                 | 45 min |
| Bulk Specific Gravity                       | MnDOT Laboratory Manual 1205 | NA     |

### B.3 Mineral Filler

Mineral filler shall meet the requirements in AASHTO M17.

### C. Mix Design

It is the Contractor's responsibility to design the UTBWC mixture that meets the requirements of this specification.

The mixture design's optimum binder content is first established so that a minimum adjusted film thickness (Adj. AFT) requirement of 10.5 microns is met. Calculate the Adj. AFT according to MnDOT Lab Procedure 1854.

At the optimum binder content the mixture must meet the Drain down and Lottman (TSR) requirements in Table 2356-4, "Mixture Requirements."

Lottman Testing (TSR) shall use 6" gyratory specimens compacted to 7-8% voids. One freeze-thaw cycle shall be included prior to testing.

Each design shall include the additional design trial points that bracket the optimum AC content and with at least one point at 0.4 above and below the optimum AC content.



## D. Mix Design Submittal

| <b>Table 2356-4<br/>Aggregate Gradation Broadband</b> |        |  |
|---|--------|--|
| Aggregate Size<br>Typical application rates           |        | 3/8-in [9.5 mm ]<br>65-75 lbs/sy           |
| Sieve Size  |        | Gradation<br>Broadband Limits<br>% Passing |
| inch  | mm     |  |
| 3/4   | 19.0   |  |
| 1/2   | 12.5   | 100  |
| 3/8   | 9.5    | 85 – 100                                   |
| #4  | 4.75   | 28 – 42                                    |
| #8  | 2.36   | 22 – 32                                    |
| #16   | 1.18   | 15 – 23                                    |
| #30   | 600 µm | 10 – 18                                    |
| #50   | 300 µm | 8 – 13                                     |
| #100  | 150 µm | 6 – 10                                     |
| #200  | 75 µm  | 4 – 5.5                                    |

| <b>Table 2356-5<br/>Mixture Requirements</b> |                  |   |
|--|------------------|---|
| Test   | Criteria         | Test Reference                          |
| Asphalt Content                              | 4.8-6.0          | MnDOT Laboratory<br>Manual 1853 or 1852 |
| Adj. AFT (Calculated)                        | 10.5 µm minimum. | MnDOT Laboratory<br>Manual 1854         |
| Draindown Test                               | 0.10% max        | AASHTO T 305                            |
| Lottman (TSR)                                | 80% min          | MnDOT Laboratory<br>Manual 1813         |

## APPENDIX C

### Gyratory Specimen Height (mm) Through 75 Gyration

| gyration | G-001:<br>1b | G-001:<br>1a | S-002B:<br>1 | S-002B:<br>2 | RS-003:<br>1 | RS-003:<br>2 | RG-004:<br>a | RG-004:<br>b | T-005:<br>1 | T-005:<br>2 | RT-006:<br>a | RT-006:<br>b |
|----------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|-------------|-------------|--------------|--------------|
| 0        | 142.3        | 142.5        | 144.1        | 144.2        | 142.4        | 143.4        | 144.1        | 144.5        | 140.4       | 140.3       | 141.9        | 142.2        |
| 1        | 136.6        | 136.7        | 137.9        | 138          | 136.4        | 137.4        | 138.2        | 138.6        | 134.8       | 134.8       | 136.4        | 136.7        |
| 2        | 133.7        | 133.7        | 134.6        | 134.6        | 133.3        | 134.3        | 135.2        | 135.6        | 132.1       | 131.9       | 133.6        | 133.9        |
| 3        | 131.6        | 133.6        | 132.3        | 132.3        | 131.1        | 132.1        | 133.1        | 133.6        | 130.1       | 129.9       | 131.6        | 131.9        |
| 4        | 130          | 130          | 130.5        | 130.4        | 129.4        | 130.3        | 131.5        | 132          | 128.6       | 128.4       | 130.2        | 130.4        |
| 5        | 128          | 128.8        | 129          | 128.9        | 128          | 128.9        | 130.3        | 130.7        | 127.3       | 127.2       | 129          | 129.2        |
| 6        | 127.7        | 127.7        | 127.8        | 127.7        | 126.9        | 127.7        | 129.2        | 129.6        | 126.3       | 126.2       | 128          | 128.2        |
| 7        | 126.9        | 126.8        | 126.7        | 126.7        | 125.9        | 126.6        | 128.3        | 128.8        | 125.5       | 125.3       | 127.1        | 127.3        |
| 8        | 126.1        | 126.1        | 125.8        | 125.7        | 125          | 125.8        | 127.6        | 128          | 124.7       | 124.6       | 126.4        | 126.6        |
| 9        | 125.5        | 125.4        | 125          | 124.9        | 124.3        | 125          | 126.9        | 127.3        | 124         | 123.9       | 125.8        | 125.9        |
| 10       | 124.9        | 124.8        | 124.3        | 124.2        | 123.6        | 124.3        | 126.3        | 126.7        | 123.5       | 123.3       | 125.2        | 125.4        |
| 11       | 124.3        | 124.3        | 123.6        | 123.6        | 123          | 123.7        | 125.8        | 126.2        | 122.9       | 122.8       | 124.7        | 124.8        |
| 12       | 123.8        | 123.8        | 123.1        | 123          | 122.5        | 123.1        | 125.3        | 125.7        | 122.4       | 122.3       | 124.2        | 124.3        |
| 13       | 123.4        | 123.4        | 122.5        | 122.4        | 122          | 122.6        | 124.9        | 125.3        | 122         | 121.9       | 123.8        | 123.9        |
| 14       | 123          | 123          | 122          | 121.9        | 121.5        | 122.1        | 124.6        | 124.9        | 121.3       | 121.5       | 123.4        | 123.5        |
| 15       | 122.7        | 122.6        | 121.6        | 121.5        | 121.1        | 121.7        | 124.1        | 124.5        | 121.2       | 121.1       | 123          | 123.2        |
| 16       | 122.3        | 122.3        | 121.2        | 121.1        | 120.7        | 121.6        | 123.8        | 124.2        | 120.8       | 120.7       | 122.7        | 122.8        |
| 17       | 122          | 122          | 120.8        | 120.7        | 120.3        | 120.9        | 123.5        | 123.9        | 120.5       | 120.4       | 122.4        | 122.5        |
| 18       | 121.7        | 121.7        | 120.4        | 120.3        | 120          | 120.5        | 123.2        | 123.6        | 120.2       | 120.1       | 122.1        | 122.2        |
| 19       | 121.4        | 121.4        | 120.1        | 120          | 119.7        | 120.2        | 122.9        | 123.3        | 119.9       | 119.8       | 121.8        | 121.9        |
| 20       | 121.2        | 121.2        | 119.7        | 119.6        | 119.4        | 119.9        | 122.6        | 123          | 119.6       | 119.5       | 121.5        | 121.6        |
| 21       | 120.9        | 120.9        | 119.4        | 119.3        | 119.1        | 119.6        | 122.4        | 122.8        | 119.4       | 119.3       | 121.3        | 121.4        |
| 22       | 120.7        | 120.7        | 119.2        | 119          | 118.8        | 119.3        | 122.1        | 122.5        | 119.1       | 119.1       | 121          | 121.2        |
| 23       | 120.5        | 120.5        | 118.9        | 118.8        | 118.6        | 119.1        | 121.9        | 122.3        | 118.9       | 118.8       | 120.8        | 120.9        |
| 24       | 120.3        | 120.3        | 118.6        | 118.5        | 118.3        | 118.8        | 121.7        | 122.1        | 118.6       | 118.6       | 120.6        | 120.7        |
| 25       | 120.1        | 120.1        | 118.4        | 118.3        | 118.1        | 118.6        | 121.5        | 121.9        | 118.4       | 118.4       | 120.4        | 120.5        |
| 26       | 119.9        | 119.9        | 118.1        | 118          | 117.9        | 118.3        | 121.3        | 121.7        | 118.2       | 118.2       | 120.2        | 120.3        |

| gyration | G-001:<br>1b | G-001:<br>1a | S-002B:<br>1 | S-002B:<br>2 | RS-003:<br>1 | RS-003:<br>2 | RG-004:<br>a | RG-004:<br>b | T-005:<br>1 | T-005:<br>2 | RT-006:<br>a | RT-006:<br>b |
|----------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|-------------|-------------|--------------|--------------|
| 27       | 119.7        | 119.7        | 117.9        | 117.8        | 117.6        | 118.1        | 121.1        | 121.6        | 118         | 118         | 120          | 120.1        |
| 28       | 119.5        | 119.5        | 117.7        | 117.6        | 117.5        | 117.9        | 121          | 121.4        | 117.8       | 117.8       | 119.8        | 120          |
| 29       | 119.4        | 119.4        | 117.5        | 117.4        | 117.3        | 117.7        | 120.8        | 121.2        | 117.6       | 117.6       | 119.7        | 119.8        |
| 30       | 119.2        | 119.2        | 117.3        | 117.2        | 117.1        | 117.5        | 120.6        | 121.1        | 117.5       | 117.5       | 119.5        | 119.6        |
| 31       | 119          | 119.1        | 117.1        | 117          | 116.9        | 117.3        | 120.5        | 120.9        | 117.3       | 117.3       | 119.3        | 119.5        |
| 32       | 118.9        | 118.9        | 116.9        | 116.8        | 116.7        | 117.2        | 120.3        | 120.8        | 117.1       | 117.2       | 119.2        | 119.3        |
| 33       | 118.8        | 118.8        | 116.8        | 116.6        | 116.6        | 117          | 120.2        | 120.6        | 117         | 117         | 119.1        | 119.2        |
| 34       | 118.6        | 118.7        | 116.6        | 116.4        | 116.4        | 116.8        | 120.1        | 120.5        | 116.8       | 116.9       | 118.9        | 119          |
| 35       | 118.5        | 118.5        | 116.4        | 116.3        | 116.3        | 116.7        | 119.9        | 120.3        | 116.7       | 116.7       | 118.8        | 118.9        |
| 36       | 118.4        | 118.4        | 116.3        | 116.1        | 116.1        | 116.5        | 119.8        | 120.2        | 116.5       | 116.6       | 118.6        | 118.8        |
| 37       | 118.3        | 118.3        | 116.1        | 115.9        | 116          | 116.4        | 119.7        | 120.1        | 116.4       | 116.5       | 118.5        | 118.7        |
| 38       | 118.1        | 118.2        | 115.9        | 115.8        | 115.8        | 116.2        | 119.6        | 120          | 116.3       | 116.3       | 118.4        | 118.5        |
| 39       | 118          | 118.1        | 115.8        | 115.7        | 115.7        | 116.1        | 119.5        | 119.9        | 116.2       | 116.2       | 118.3        | 118.4        |
| 40       | 117.9        | 118          | 115.7        | 115.5        | 115.6        | 116          | 119.4        | 119.8        | 116         | 116.1       | 118.2        | 118.3        |
| 41       | 117.8        | 117.9        | 115.5        | 115.4        | 115.4        | 115.8        | 119.3        | 119.6        | 115.9       | 116         | 118.1        | 118.2        |
| 42       | 117.7        | 117.8        | 115.4        | 115.3        | 115.3        | 115.7        | 119.2        | 119.5        | 115.8       | 115.8       | 118          | 118.1        |
| 43       | 117.7        | 117.7        | 115.3        | 115          | 115.2        | 115.6        | 119.1        | 119.4        | 115.7       | 115.7       | 117.9        | 118          |
| 44       | 117.6        | 117.6        | 115.2        | 115          | 115.1        | 115.5        | 119          | 119.3        | 115.6       | 115.6       | 117.7        | 117.9        |
| 45       | 117.4        | 117.5        | 115          | 114.9        | 115          | 115.4        | 118.9        | 119.2        | 115.5       | 115.5       | 117.7        | 117.8        |
| 46       | 117.3        | 117.4        | 114.9        | 114.8        | 114.9        | 115.3        | 118.8        | 119.2        | 115.4       | 115.4       | 117.6        | 117.7        |
| 47       | 117.3        | 117.3        | 114.8        | 114.7        | 114.8        | 115.1        | 118.7        | 119.1        | 115.3       | 115.3       | 117.5        | 117.6        |
| 48       | 117.2        | 117.2        | 114.7        | 114.6        | 114.7        | 115          | 118.6        | 119          | 115.2       | 115.2       | 117.4        | 117.5        |
| 49       | 117.1        | 117.1        | 114.6        | 114.4        | 114.6        | 114.9        | 118.5        | 118.9        | 115.1       | 115.1       | 117.3        | 117.4        |
| 50       | 117          | 117.1        | 114.5        | 114.3        | 114.5        | 114.8        | 118.4        | 118.8        | 115         | 115         | 117.2        | 117.3        |
| 51       | 116.9        | 117          | 114.4        | 114.2        | 114.4        | 114.7        | 118.4        | 118.7        | 114.9       | 115         | 117.1        | 117.2        |
| 52       | 116.9        | 116.9        | 114.3        | 114.1        | 114.3        | 114.7        | 118.3        | 118.7        | 114.8       | 114.9       | 117          | 117.2        |
| 53       | 116.8        | 116.8        | 114.2        | 114          | 114.2        | 114.6        | 118.2        | 118.6        | 114.7       | 114.8       | 117          | 117.1        |
| 54       | 116.7        | 116.8        | 114.1        | 114          | 114.1        | 114.5        | 118.1        | 118.5        | 114.6       | 114.7       | 116.9        | 117          |
| 55       | 116.6        | 116.7        | 114          | 113.9        | 114          | 114.4        | 118.1        | 118.4        | 114.5       | 114.6       | 116.8        | 116.9        |
| 56       | 116.6        | 116.6        | 113.9        | 113.8        | 113.9        | 114.3        | 118          | 118.4        | 114.5       | 114.5       | 116.7        | 116.9        |

| gyration | G-001:<br>1b | G-001:<br>1a | S-002B:<br>1 | S-002B:<br>2 | RS-003:<br>1 | RS-003:<br>2 | RG-004:<br>a | RG-004:<br>b | T-005:<br>1 | T-005:<br>2 | RT-006:<br>a | RT-006:<br>b |
|----------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|-------------|-------------|--------------|--------------|
| 57       | 116.5        | 116.6        | 113.8        | 113.7        | 113.9        | 114.2        | 117.9        | 118.3        | 114.4       | 114.5       | 116.7        | 116.8        |
| 58       | 116.4        | 116.5        | 113.8        | 113.6        | 113.8        | 114.1        | 117.9        | 118.2        | 114.3       | 114.4       | 116.6        | 116.7        |
| 59       | 116.4        | 116.4        | 113.7        | 113.5        | 113.7        | 114.1        | 117.8        | 118.2        | 114.2       | 114.3       | 116.5        | 116.6        |
| 60       | 116.3        | 116.4        | 113.6        | 113.4        | 113.6        | 114          | 117.7        | 118.1        | 114.2       | 114.2       | 116.4        | 116.6        |
| 61       | 116.2        | 116.3        | 113.5        | 113.4        | 113.6        | 113.9        | 117.7        | 118          | 114.1       | 114.2       | 116.4        | 116.5        |
| 62       | 116.2        | 116.3        | 113.4        | 113.3        | 113.5        | 113.8        | 117.6        | 118          | 114         | 114.1       | 116.3        | 116.4        |
| 63       | 116.1        | 116.2        | 113.4        | 113.2        | 113.4        | 113.8        | 117.6        | 117.9        | 113.9       | 114         | 116.2        | 116.4        |
| 64       | 116.1        | 116.1        | 113.3        | 113.2        | 113.3        | 113.7        | 117.5        | 117.9        | 113.9       | 114         | 116.2        | 116.3        |
| 65       | 116          | 116.1        | 113.2        | 113.1        | 113.3        | 113.6        | 117.4        | 117.8        | 113.8       | 113.9       | 116.1        | 116.3        |
| 66       | 116          | 116          | 113.2        | 113          | 113.2        | 113.6        | 117.4        | 117.7        | 113.7       | 113.8       | 116.1        | 116.2        |
| 67       | 115.9        | 116          | 113.1        | 112.9        | 113.2        | 113.5        | 117.3        | 117.7        | 113.7       | 113.8       | 116          | 116.1        |
| 68       | 115.9        | 115.9        | 113          | 112.9        | 113.1        | 113.4        | 117.3        | 117.6        | 113.6       | 113.7       | 115.9        | 116.1        |
| 69       | 115.8        | 115.9        | 112.9        | 112.8        | 113          | 113.4        | 117.2        | 117.6        | 113.5       | 113.6       | 115.9        | 116          |
| 70       | 115.8        | 115.8        | 112.9        | 112.7        | 113          | 113.3        | 117.2        | 117.5        | 113.5       | 113.6       | 115.8        | 116          |
| 71       | 115.7        | 115.8        | 112.8        | 112.7        | 112.9        | 113.2        | 117.1        | 117.5        | 113.4       | 113.5       | 115.8        | 115.9        |
| 72       | 115.7        | 115.7        | 112.8        | 112.6        | 112.8        | 113.2        | 117.1        | 117.4        | 113.4       | 113.5       | 115.7        | 115.9        |
| 73       | 115.6        | 115.7        | 112.7        | 112.6        | 112.8        | 113.1        | 117          | 117.4        | 113.3       | 113.4       | 115.7        | 115.8        |
| 74       | 115.6        | 115.6        | 112.6        | 112.5        | 112.7        | 113.1        | 117          | 117.3        | 113.2       | 113.3       | 115.6        | 115.7        |
| 75       | 115.5        | 115.6        | 112.6        | 112.4        | 112.7        | 113          | 117          | 117.3        | 113.2       | 113.3       | 115.6        | 115.7        |

## APPENDIX D

### MnDOT Inventory of Dynamic Modulus Test Specimens.

| Mix #      | TP62 Specimens | Air Voids |
|------------|----------------|-----------|
| 2011-001   | #1             | 6.6       |
|            | #2             | 7.1       |
|            | #3             | 7.0       |
|            | #4             | 6.7       |
| 2011-002   | #1             | 5.3       |
|            | #2             | 7.1       |
|            | #3             | 7.3       |
|            | #4             | 7.2       |
| 2011-003   | #1             | 8.3       |
|            | #2             | 7.9       |
|            | #3             | 7.5       |
|            | #4             | 7.6       |
| 2011-004   | #1             | 6.6       |
|            | #2             | 7.1       |
|            | #3             | 7.1       |
|            | #4             | 6.8       |
| 2011-005   | #1             | 6.4       |
|            | #2             | 7.1       |
|            | #3             | 7.0       |
|            | #4             | 7.2       |
|            | #5             | 6.9       |
| 2011-006   | #1             | 6.3       |
|            | #2             | 7.3       |
|            | #3             | 7.0       |
|            | #4             | 7.2       |
| 6 mixtures | 25 specimens   |           |

## APPENDIX E

### E\* Results: Data and Fit

Mix G-001 E\* Results: Data

| Red freq | Data1    | Red freq | Data2    | Red freq | Data3    | Red freq | Data4    |
|----------|----------|----------|----------|----------|----------|----------|----------|
| 3.91E+03 | 1.59E+06 | 2.30E+03 | 1.33E+06 | 2.51E+02 | 9.02E+05 | 1.42E+03 | 1.24E+06 |
| 1.95E+04 | 1.88E+06 | 1.15E+04 | 1.75E+06 | 1.25E+03 | 1.18E+06 | 7.11E+03 | 1.63E+06 |
| 3.91E+04 | 2.06E+06 | 2.30E+04 | 1.95E+06 | 2.51E+03 | 1.31E+06 | 1.42E+04 | 1.80E+06 |
| 1.95E+05 | 2.63E+06 | 1.15E+05 | 2.33E+06 | 1.25E+04 | 1.70E+06 | 7.11E+04 | 2.24E+06 |
| 3.91E+05 | 2.84E+06 | 2.30E+05 | 2.48E+06 | 2.51E+04 | 1.87E+06 | 1.42E+05 | 2.36E+06 |
| 9.76E+05 | 2.65E+06 | 5.76E+05 | 2.51E+06 | 6.27E+04 | 2.10E+06 | 3.56E+05 | 2.59E+06 |
| 9.96E+00 | 2.87E+05 | 8.13E+00 | 2.79E+05 | 6.51E+00 | 2.73E+05 | 5.86E+00 | 2.55E+05 |
| 4.53E+01 | 4.60E+05 | 3.71E+01 | 4.47E+05 | 2.96E+01 | 4.45E+05 | 2.93E+01 | 4.12E+05 |
| 9.06E+01 | 5.75E+05 | 7.41E+01 | 5.56E+05 | 5.91E+01 | 5.59E+05 | 5.86E+01 | 5.15E+05 |
| 4.53E+02 | 9.33E+05 | 3.71E+02 | 8.70E+05 | 2.95E+02 | 8.70E+05 | 2.93E+02 | 8.33E+05 |
| 9.07E+02 | 1.14E+06 | 7.41E+02 | 1.03E+06 | 5.91E+02 | 1.03E+06 | 5.86E+02 | 9.82E+05 |
| 2.27E+03 | 1.36E+06 | 1.85E+03 | 1.24E+06 | 1.48E+03 | 1.26E+06 | 1.46E+03 | 1.20E+06 |
| 1.00E-01 | 4.40E+04 | 1.00E-01 | 4.59E+04 | 1.00E-01 | 5.42E+04 | 1.00E-01 | 5.27E+04 |
| 5.00E-01 | 8.19E+04 | 5.00E-01 | 8.94E+04 | 5.00E-01 | 9.10E+04 | 5.00E-01 | 8.47E+04 |
| 1.00E+00 | 1.05E+05 | 1.00E+00 | 1.15E+05 | 1.00E+00 | 1.19E+05 | 1.00E+00 | 1.16E+05 |
| 5.00E+00 | 2.01E+05 | 5.00E+00 | 2.12E+05 | 5.00E+00 | 2.34E+05 | 5.00E+00 | 2.24E+05 |
| 1.00E+01 | 2.70E+05 | 1.00E+01 | 2.83E+05 | 1.00E+01 | 3.17E+05 | 1.00E+01 | 2.99E+05 |
| 2.50E+01 | 3.81E+05 | 2.50E+01 | 4.05E+05 | 2.50E+01 | 4.44E+05 | 2.50E+01 | 4.23E+05 |
| 6.89E-03 | 2.25E+04 | 5.58E-03 | 2.37E+04 | 3.52E-03 | 2.37E+04 | 5.58E-03 | 2.29E+04 |
| 3.44E-02 | 3.42E+04 | 2.79E-02 | 3.54E+04 | 1.76E-02 | 3.42E+04 | 2.79E-02 | 4.13E+04 |
| 6.88E-02 | 4.05E+04 | 5.58E-02 | 4.22E+04 | 3.52E-02 | 3.97E+04 | 5.59E-02 | 4.47E+04 |
| 3.44E-01 | 6.59E+04 | 2.79E-01 | 6.70E+04 | 1.76E-01 | 6.08E+04 | 2.79E-01 | 7.07E+04 |
| 6.89E-01 | 8.74E+04 | 5.58E-01 | 8.81E+04 | 3.52E-01 | 7.95E+04 | 5.58E-01 | 9.18E+04 |
| 1.72E+00 | 1.44E+05 | 1.39E+00 | 1.39E+05 | 8.80E-01 | 1.22E+05 | 1.40E+00 | 1.39E+05 |
| 9.37E-04 | 1.53E+04 | 8.69E-04 | 1.62E+04 | 7.44E-04 | 1.64E+04 | 6.44E-04 | 1.54E+04 |
| 4.69E-03 | 2.35E+04 | 4.34E-03 | 2.48E+04 | 3.72E-03 | 2.50E+04 | 3.21E-03 | 2.36E+04 |
| 9.37E-03 | 2.54E+04 | 8.69E-03 | 2.72E+04 | 7.44E-03 | 2.76E+04 | 6.43E-03 | 2.64E+04 |
| 4.69E-02 | 3.26E+04 | 4.34E-02 | 3.55E+04 | 3.72E-02 | 3.61E+04 | 3.22E-02 | 3.37E+04 |
| 9.38E-02 | 3.94E+04 | 8.69E-02 | 4.30E+04 | 7.44E-02 | 4.42E+04 | 6.44E-02 | 4.17E+04 |
| 2.34E-01 | 6.97E+04 | 2.17E-01 | 7.24E+04 | 1.86E-01 | 7.52E+04 | 1.61E-01 | 6.95E+04 |

Mix G-001 E\* Results: Fit

| freq     | Fit1     | Fit2     | Fit3     | Fit4     | AlldataFit | deltaE/avgE |
|----------|----------|----------|----------|----------|------------|-------------|
| 1.00E-05 | 1.08E+04 | 1.13E+04 | 1.26E+04 | 1.08E+04 | 1.13E+04   | 16.7%       |
| 3.16E-05 | 1.14E+04 | 1.20E+04 | 1.34E+04 | 1.16E+04 | 1.21E+04   | 16.1%       |
| 1.00E-04 | 1.24E+04 | 1.32E+04 | 1.45E+04 | 1.29E+04 | 1.32E+04   | 15.5%       |
| 3.16E-04 | 1.39E+04 | 1.48E+04 | 1.61E+04 | 1.47E+04 | 1.49E+04   | 15.0%       |
| 1.00E-03 | 1.61E+04 | 1.72E+04 | 1.86E+04 | 1.74E+04 | 1.73E+04   | 14.5%       |
| 3.16E-03 | 1.93E+04 | 2.09E+04 | 2.24E+04 | 2.14E+04 | 2.10E+04   | 14.3%       |
| 1.00E-02 | 2.45E+04 | 2.66E+04 | 2.83E+04 | 2.76E+04 | 2.67E+04   | 14.3%       |
| 3.16E-02 | 3.26E+04 | 3.57E+04 | 3.79E+04 | 3.74E+04 | 3.59E+04   | 14.6%       |
| 1.00E-01 | 4.60E+04 | 5.04E+04 | 5.37E+04 | 5.31E+04 | 5.08E+04   | 15.1%       |
| 3.16E-01 | 6.83E+04 | 7.45E+04 | 8.01E+04 | 7.85E+04 | 7.54E+04   | 15.7%       |
| 1.00E+00 | 1.06E+05 | 1.14E+05 | 1.24E+05 | 1.20E+05 | 1.16E+05   | 16.0%       |
| 3.16E+00 | 1.67E+05 | 1.78E+05 | 1.95E+05 | 1.85E+05 | 1.81E+05   | 15.7%       |
| 1.00E+01 | 2.65E+05 | 2.78E+05 | 3.05E+05 | 2.85E+05 | 2.83E+05   | 14.3%       |
| 3.16E+01 | 4.11E+05 | 4.23E+05 | 4.62E+05 | 4.29E+05 | 4.31E+05   | 11.9%       |
| 1.00E+02 | 6.14E+05 | 6.19E+05 | 6.67E+05 | 6.23E+05 | 6.32E+05   | 8.5%        |
| 3.16E+02 | 8.72E+05 | 8.65E+05 | 9.11E+05 | 8.66E+05 | 8.80E+05   | 5.2%        |
| 1.00E+03 | 1.17E+06 | 1.15E+06 | 1.17E+06 | 1.15E+06 | 1.16E+06   | 2.5%        |
| 3.16E+03 | 1.49E+06 | 1.44E+06 | 1.44E+06 | 1.45E+06 | 1.46E+06   | 4.0%        |
| 1.00E+04 | 1.81E+06 | 1.73E+06 | 1.68E+06 | 1.74E+06 | 1.74E+06   | 7.7%        |
| 3.16E+04 | 2.10E+06 | 2.00E+06 | 1.88E+06 | 2.03E+06 | 2.01E+06   | 10.9%       |
| 1.00E+05 | 2.36E+06 | 2.23E+06 | 2.06E+06 | 2.28E+06 | 2.24E+06   | 13.5%       |
| 3.16E+05 | 2.57E+06 | 2.43E+06 | 2.19E+06 | 2.50E+06 | 2.43E+06   | 15.6%       |
| 1.00E+06 | 2.75E+06 | 2.59E+06 | 2.30E+06 | 2.68E+06 | 2.58E+06   | 17.3%       |
| 3.16E+06 | 2.88E+06 | 2.72E+06 | 2.38E+06 | 2.82E+06 | 2.70E+06   | 18.6%       |
| 1.00E+07 | 2.99E+06 | 2.82E+06 | 2.44E+06 | 2.94E+06 | 2.79E+06   | 19.6%       |
| 3.16E+07 | 3.07E+06 | 2.89E+06 | 2.49E+06 | 3.03E+06 | 2.86E+06   | 20.4%       |
| 1.00E+08 | 3.13E+06 | 2.95E+06 | 2.52E+06 | 3.10E+06 | 2.92E+06   | 20.9%       |
| 3.16E+08 | 3.18E+06 | 2.99E+06 | 2.54E+06 | 3.15E+06 | 2.96E+06   | 21.4%       |
| 1.00E+09 | 3.21E+06 | 3.03E+06 | 2.56E+06 | 3.19E+06 | 2.99E+06   | 21.7%       |
| 3.16E+09 | 3.24E+06 | 3.05E+06 | 2.57E+06 | 3.23E+06 | 3.01E+06   | 22.0%       |
|          |          |          |          |          | Sum =      | 439.2%      |

deltaE = Max - Min

Mix S-002 E\* Results: Data

| Red freq | Data1   | Red freq | Data2    | Red freq | Data3    | Red freq | Data4    |
|----------|---------|----------|----------|----------|----------|----------|----------|
| No Data  | No Data | 6.23E+02 | 1.34E+06 | 2.33E+03 | 1.61E+06 | 1.24E+03 | 1.57E+06 |
| No Data  | No Data | 3.12E+03 | 1.72E+06 | 1.16E+04 | 2.20E+06 | 6.20E+03 | 1.93E+06 |
| No Data  | No Data | 6.23E+03 | 1.87E+06 | 2.33E+04 | 2.35E+06 | 1.24E+04 | 2.12E+06 |
| No Data  | No Data | 3.12E+04 | 2.28E+06 | 1.16E+05 | 2.74E+06 | 6.20E+04 | 2.57E+06 |
| No Data  | No Data | 6.23E+04 | 2.41E+06 | 2.33E+05 | 2.96E+06 | 1.24E+05 | 2.73E+06 |
| No Data  | No Data | 1.56E+05 | 2.62E+06 | 5.82E+05 | 3.20E+06 | 3.10E+05 | 3.01E+06 |
| No Data  | No Data | 8.25E+00 | 4.08E+05 | 1.34E+01 | 4.33E+05 | 1.19E+01 | 4.44E+05 |
| No Data  | No Data | 4.12E+01 | 6.50E+05 | 6.71E+01 | 6.84E+05 | 5.93E+01 | 7.48E+05 |
| No Data  | No Data | 8.25E+01 | 7.89E+05 | 1.34E+02 | 8.38E+05 | 1.19E+02 | 8.95E+05 |
| No Data  | No Data | 4.12E+02 | 1.19E+06 | 6.70E+02 | 1.25E+06 | 5.93E+02 | 1.30E+06 |
| No Data  | No Data | 8.25E+02 | 1.38E+06 | 1.34E+03 | 1.45E+06 | 1.19E+03 | 1.49E+06 |
| No Data  | No Data | 2.06E+03 | 1.58E+06 | 3.35E+03 | 1.65E+06 | 2.96E+03 | 1.67E+06 |
| No Data  | No Data | 1.00E-01 | 7.04E+04 | 1.00E-01 | 6.04E+04 | 1.00E-01 | 7.36E+04 |
| No Data  | No Data | 5.00E-01 | 1.24E+05 | 5.00E-01 | 1.10E+05 | 5.00E-01 | 1.27E+05 |
| No Data  | No Data | 1.00E+00 | 1.64E+05 | 1.00E+00 | 1.43E+05 | 9.99E-01 | 1.64E+05 |
| No Data  | No Data | 5.00E+00 | 3.18E+05 | 5.00E+00 | 2.79E+05 | 5.00E+00 | 3.12E+05 |
| No Data  | No Data | 1.00E+01 | 4.16E+05 | 1.00E+01 | 3.67E+05 | 1.00E+01 | 4.04E+05 |
| No Data  | No Data | 2.50E+01 | 5.86E+05 | 2.50E+01 | 5.15E+05 | 2.50E+01 | 5.52E+05 |
| No Data  | No Data | 3.79E-03 | 2.69E+04 | 4.99E-03 | 2.72E+04 | 9.48E-03 | 3.92E+04 |
| No Data  | No Data | 1.90E-02 | 4.49E+04 | 2.49E-02 | 4.51E+04 | 4.75E-02 | 5.96E+04 |
| No Data  | No Data | 3.78E-02 | 5.30E+04 | 4.99E-02 | 5.38E+04 | 9.48E-02 | 7.26E+04 |
| No Data  | No Data | 1.89E-01 | 8.58E+04 | 2.50E-01 | 8.59E+04 | 4.74E-01 | 1.23E+05 |
| No Data  | No Data | 3.79E-01 | 1.12E+05 | 4.99E-01 | 1.03E+05 | 9.48E-01 | 1.62E+05 |
| No Data  | No Data | 9.47E-01 | 1.71E+05 | 1.25E+00 | 1.70E+05 | 2.37E+00 | 2.42E+05 |
| No Data  | No Data | 5.97E-04 | 1.99E+04 | 7.83E-04 | 1.99E+04 | 6.85E-04 | 2.72E+04 |
| No Data  | No Data | 2.98E-03 | 3.10E+04 | 3.91E-03 | 3.12E+04 | 3.42E-03 | 3.46E+04 |
| No Data  | No Data | 5.97E-03 | 3.37E+04 | 7.84E-03 | 3.36E+04 | 6.85E-03 | 3.82E+04 |
| No Data  | No Data | 2.99E-02 | 4.40E+04 | 3.92E-02 | 4.31E+04 | 3.43E-02 | 4.94E+04 |
| No Data  | No Data | 5.97E-02 | 5.41E+04 | 7.83E-02 | 5.27E+04 | 6.85E-02 | 6.03E+04 |
| No Data  | No Data | 1.49E-01 | 8.86E+04 | 1.96E-01 | 8.97E+04 | 1.71E-01 | 9.95E+04 |



Mix S-002 E\* Results: Fit

| freq     | Fit1    | Fit2     | Fit3     | Fit4     | AlldataFit | deltaE/avgE |
|----------|---------|----------|----------|----------|------------|-------------|
| 1.00E-05 | No Data | 1.45E+04 | 1.37E+04 | 1.92E+04 | 1.58E+04   | 34.9%       |
| 3.16E-05 | No Data | 1.55E+04 | 1.47E+04 | 2.02E+04 | 1.68E+04   | 33.0%       |
| 1.00E-04 | No Data | 1.70E+04 | 1.61E+04 | 2.18E+04 | 1.83E+04   | 30.7%       |
| 3.16E-04 | No Data | 1.92E+04 | 1.83E+04 | 2.40E+04 | 2.05E+04   | 28.1%       |
| 1.00E-03 | No Data | 2.25E+04 | 2.14E+04 | 2.74E+04 | 2.38E+04   | 25.2%       |
| 3.16E-03 | No Data | 2.77E+04 | 2.62E+04 | 3.26E+04 | 2.89E+04   | 22.2%       |
| 1.00E-02 | No Data | 3.59E+04 | 3.36E+04 | 4.06E+04 | 3.67E+04   | 19.2%       |
| 3.16E-02 | No Data | 4.91E+04 | 4.53E+04 | 5.35E+04 | 4.94E+04   | 16.5%       |
| 1.00E-01 | No Data | 7.11E+04 | 6.44E+04 | 7.45E+04 | 7.00E+04   | 14.4%       |
| 3.16E-01 | No Data | 1.08E+05 | 9.56E+04 | 1.09E+05 | 1.04E+05   | 12.9%       |
| 1.00E+00 | No Data | 1.68E+05 | 1.47E+05 | 1.66E+05 | 1.60E+05   | 13.2%       |
| 3.16E+00 | No Data | 2.64E+05 | 2.28E+05 | 2.57E+05 | 2.50E+05   | 14.1%       |
| 1.00E+01 | No Data | 4.07E+05 | 3.54E+05 | 3.97E+05 | 3.86E+05   | 13.8%       |
| 3.16E+01 | No Data | 6.06E+05 | 5.35E+05 | 5.96E+05 | 5.80E+05   | 12.3%       |
| 1.00E+02 | No Data | 8.58E+05 | 7.77E+05 | 8.55E+05 | 8.32E+05   | 9.7%        |
| 3.16E+02 | No Data | 1.15E+06 | 1.08E+06 | 1.16E+06 | 1.13E+06   | 7.7%        |
| 1.00E+03 | No Data | 1.45E+06 | 1.41E+06 | 1.49E+06 | 1.46E+06   | 5.5%        |
| 3.16E+03 | No Data | 1.75E+06 | 1.77E+06 | 1.82E+06 | 1.79E+06   | 4.0%        |
| 1.00E+04 | No Data | 2.02E+06 | 2.11E+06 | 2.13E+06 | 2.10E+06   | 5.0%        |
| 3.16E+04 | No Data | 2.26E+06 | 2.43E+06 | 2.39E+06 | 2.37E+06   | 7.4%        |
| 1.00E+05 | No Data | 2.45E+06 | 2.71E+06 | 2.61E+06 | 2.60E+06   | 10.1%       |
| 3.16E+05 | No Data | 2.60E+06 | 2.94E+06 | 2.78E+06 | 2.78E+06   | 12.3%       |
| 1.00E+06 | No Data | 2.72E+06 | 3.13E+06 | 2.92E+06 | 2.93E+06   | 14.1%       |
| 3.16E+06 | No Data | 2.81E+06 | 3.28E+06 | 3.02E+06 | 3.04E+06   | 15.6%       |
| 1.00E+07 | No Data | 2.87E+06 | 3.40E+06 | 3.10E+06 | 3.12E+06   | 16.7%       |
| 3.16E+07 | No Data | 2.92E+06 | 3.49E+06 | 3.15E+06 | 3.19E+06   | 17.6%       |
| 1.00E+08 | No Data | 2.96E+06 | 3.55E+06 | 3.20E+06 | 3.23E+06   | 18.3%       |
| 3.16E+08 | No Data | 2.99E+06 | 3.61E+06 | 3.23E+06 | 3.27E+06   | 18.9%       |
| 1.00E+09 | No Data | 3.01E+06 | 3.64E+06 | 3.25E+06 | 3.29E+06   | 19.3%       |
| 3.16E+09 | No Data | 3.02E+06 | 3.67E+06 | 3.26E+06 | 3.31E+06   | 19.6%       |
|          |         |          |          |          | Sum =      | 492.5%      |

Mix RS-003 E\* Results: Data

| Red freq | Data1    | Red freq | Data2    | Red freq | Data3    | Red freq | Data4    |
|----------|----------|----------|----------|----------|----------|----------|----------|
| 7.52E+02 | 1.37E+06 | 7.12E+02 | 1.57E+06 | 1.93E+03 | 1.88E+06 | 5.10E+02 | 1.55E+06 |
| 3.76E+03 | 1.76E+06 | 3.56E+03 | 1.81E+06 | 9.66E+03 | 2.14E+06 | 2.55E+03 | 1.93E+06 |
| 7.52E+03 | 1.93E+06 | 7.12E+03 | 2.01E+06 | 1.93E+04 | 2.33E+06 | 5.10E+03 | 2.02E+06 |
| 3.76E+04 | 2.33E+06 | 3.56E+04 | 2.48E+06 | 9.66E+04 | 2.78E+06 | 2.55E+04 | 2.45E+06 |
| 7.52E+04 | 2.50E+06 | 7.12E+04 | 2.65E+06 | 1.93E+05 | 2.92E+06 | 5.10E+04 | 2.63E+06 |
| 1.88E+05 | 2.69E+06 | 1.78E+05 | 2.84E+06 | 4.83E+05 | 3.15E+06 | 1.28E+05 | 2.91E+06 |
| 9.38E+00 | 4.64E+05 | 9.01E+00 | 5.08E+05 | 8.39E+00 | 4.92E+05 | 1.01E+01 | 5.69E+05 |
| 4.69E+01 | 6.81E+05 | 4.50E+01 | 7.50E+05 | 4.19E+01 | 7.55E+05 | 5.03E+01 | 8.82E+05 |
| 9.38E+01 | 8.16E+05 | 9.01E+01 | 9.07E+05 | 8.39E+01 | 9.10E+05 | 1.01E+02 | 1.04E+06 |
| 4.69E+02 | 1.20E+06 | 4.50E+02 | 1.32E+06 | 4.19E+02 | 1.33E+06 | 5.03E+02 | 1.47E+06 |
| 9.38E+02 | 1.38E+06 | 9.01E+02 | 1.54E+06 | 8.39E+02 | 1.55E+06 | 1.01E+03 | 1.69E+06 |
| 2.34E+03 | 1.57E+06 | 2.25E+03 | 1.77E+06 | 2.10E+03 | 1.81E+06 | 2.51E+03 | 1.90E+06 |
| 1.00E-01 | 5.71E+04 | 1.00E-01 | 6.51E+04 | 1.00E-01 | 7.73E+04 | 1.00E-01 | 8.48E+04 |
| 5.00E-01 | 1.27E+05 | 5.00E-01 | 1.46E+05 | 5.00E-01 | 1.53E+05 | 4.99E-01 | 1.67E+05 |
| 1.00E+00 | 1.67E+05 | 1.00E+00 | 1.91E+05 | 1.00E+00 | 1.97E+05 | 1.00E+00 | 2.19E+05 |
| 5.00E+00 | 3.18E+05 | 5.00E+00 | 3.62E+05 | 5.00E+00 | 3.73E+05 | 5.00E+00 | 4.07E+05 |
| 1.00E+01 | 4.17E+05 | 1.00E+01 | 4.69E+05 | 1.00E+01 | 4.85E+05 | 1.00E+01 | 5.24E+05 |
| 2.50E+01 | 5.76E+05 | 2.50E+01 | 6.41E+05 | 2.50E+01 | 6.43E+05 | 2.50E+01 | 7.12E+05 |
| 6.45E-03 | 2.92E+04 | 3.50E-03 | 2.72E+04 | 3.25E-03 | 2.73E+04 | 2.79E-03 | 2.67E+04 |
| 3.22E-02 | 4.62E+04 | 1.75E-02 | 4.44E+04 | 1.62E-02 | 4.68E+04 | 1.39E-02 | 4.73E+04 |
| 6.45E-02 | 5.68E+04 | 3.50E-02 | 5.36E+04 | 3.25E-02 | 5.65E+04 | 2.79E-02 | 5.74E+04 |
| 3.22E-01 | 9.81E+04 | 1.75E-01 | 8.86E+04 | 1.62E-01 | 9.41E+04 | 1.40E-01 | 9.52E+04 |
| 6.45E-01 | 1.33E+05 | 3.50E-01 | 1.19E+05 | 3.25E-01 | 1.27E+05 | 2.79E-01 | 1.28E+05 |
| 1.61E+00 | 2.05E+05 | 8.76E-01 | 1.80E+05 | 8.11E-01 | 1.92E+05 | 6.98E-01 | 1.98E+05 |
| 5.74E-04 | 2.03E+04 | 2.72E-04 | 2.12E+04 | 3.92E-04 | 2.24E+04 | 1.78E-04 | 2.13E+04 |
| 2.87E-03 | 2.66E+04 | 1.36E-03 | 2.64E+04 | 1.96E-03 | 3.04E+04 | 8.87E-04 | 2.69E+04 |
| 5.74E-03 | 2.90E+04 | 2.72E-03 | 2.84E+04 | 3.92E-03 | 3.30E+04 | 1.78E-03 | 2.85E+04 |
| 2.87E-02 | 3.81E+04 | 1.36E-02 | 3.62E+04 | 1.96E-02 | 4.29E+04 | 8.88E-03 | 3.63E+04 |
| 5.74E-02 | 4.78E+04 | 2.72E-02 | 4.38E+04 | 3.92E-02 | 5.23E+04 | 1.78E-02 | 4.43E+04 |
| 1.44E-01 | 8.86E+04 | 6.79E-02 | 7.22E+04 | 9.79E-02 | 9.08E+04 | 4.44E-02 | 7.18E+04 |

Mix RS-003 E\* Results: Fit

| freq     | Fit1     | Fit2     | Fit3     | Fit4     | AlldataFit | deltaE/avgE |
|----------|----------|----------|----------|----------|------------|-------------|
| 1.00E-05 | 1.38E+04 | 1.58E+04 | 1.52E+04 | 1.60E+04 | 1.52E+04   | 14.3%       |
| 3.16E-05 | 1.47E+04 | 1.68E+04 | 1.63E+04 | 1.71E+04 | 1.62E+04   | 15.1%       |
| 1.00E-04 | 1.59E+04 | 1.82E+04 | 1.80E+04 | 1.88E+04 | 1.77E+04   | 16.1%       |
| 3.16E-04 | 1.78E+04 | 2.03E+04 | 2.06E+04 | 2.13E+04 | 2.00E+04   | 17.5%       |
| 1.00E-03 | 2.08E+04 | 2.35E+04 | 2.44E+04 | 2.53E+04 | 2.35E+04   | 19.1%       |
| 3.16E-03 | 2.53E+04 | 2.87E+04 | 3.04E+04 | 3.15E+04 | 2.90E+04   | 21.1%       |
| 1.00E-02 | 3.27E+04 | 3.70E+04 | 4.00E+04 | 4.15E+04 | 3.78E+04   | 23.3%       |
| 3.16E-02 | 4.50E+04 | 5.08E+04 | 5.56E+04 | 5.84E+04 | 5.25E+04   | 25.5%       |
| 1.00E-01 | 6.58E+04 | 7.45E+04 | 8.16E+04 | 8.69E+04 | 7.72E+04   | 27.3%       |
| 3.16E-01 | 1.01E+05 | 1.15E+05 | 1.25E+05 | 1.35E+05 | 1.19E+05   | 28.5%       |
| 1.00E+00 | 1.61E+05 | 1.84E+05 | 1.96E+05 | 2.16E+05 | 1.89E+05   | 28.6%       |
| 3.16E+00 | 2.59E+05 | 2.95E+05 | 3.09E+05 | 3.42E+05 | 3.01E+05   | 27.6%       |
| 1.00E+01 | 4.08E+05 | 4.64E+05 | 4.76E+05 | 5.28E+05 | 4.69E+05   | 25.6%       |
| 3.16E+01 | 6.16E+05 | 6.96E+05 | 7.05E+05 | 7.76E+05 | 6.98E+05   | 23.0%       |
| 1.00E+02 | 8.77E+05 | 9.83E+05 | 9.90E+05 | 1.07E+06 | 9.81E+05   | 20.2%       |
| 3.16E+02 | 1.17E+06 | 1.30E+06 | 1.31E+06 | 1.40E+06 | 1.30E+06   | 17.4%       |
| 1.00E+03 | 1.48E+06 | 1.62E+06 | 1.65E+06 | 1.72E+06 | 1.62E+06   | 15.0%       |
| 3.16E+03 | 1.77E+06 | 1.92E+06 | 1.98E+06 | 2.02E+06 | 1.92E+06   | 13.0%       |
| 1.00E+04 | 2.02E+06 | 2.17E+06 | 2.27E+06 | 2.27E+06 | 2.18E+06   | 11.5%       |
| 3.16E+04 | 2.23E+06 | 2.38E+06 | 2.52E+06 | 2.48E+06 | 2.40E+06   | 12.1%       |
| 1.00E+05 | 2.40E+06 | 2.54E+06 | 2.72E+06 | 2.64E+06 | 2.58E+06   | 12.7%       |
| 3.16E+05 | 2.53E+06 | 2.67E+06 | 2.88E+06 | 2.76E+06 | 2.71E+06   | 13.2%       |
| 1.00E+06 | 2.62E+06 | 2.76E+06 | 3.01E+06 | 2.86E+06 | 2.81E+06   | 13.7%       |
| 3.16E+06 | 2.70E+06 | 2.83E+06 | 3.10E+06 | 2.92E+06 | 2.89E+06   | 14.2%       |
| 1.00E+07 | 2.75E+06 | 2.88E+06 | 3.18E+06 | 2.97E+06 | 2.94E+06   | 14.5%       |
| 3.16E+07 | 2.79E+06 | 2.91E+06 | 3.23E+06 | 3.01E+06 | 2.98E+06   | 14.8%       |
| 1.00E+08 | 2.81E+06 | 2.93E+06 | 3.27E+06 | 3.04E+06 | 3.01E+06   | 15.1%       |
| 3.16E+08 | 2.83E+06 | 2.95E+06 | 3.30E+06 | 3.06E+06 | 3.03E+06   | 15.3%       |
| 1.00E+09 | 2.85E+06 | 2.96E+06 | 3.32E+06 | 3.07E+06 | 3.05E+06   | 15.4%       |
| 3.16E+09 | 2.86E+06 | 2.97E+06 | 3.33E+06 | 3.08E+06 | 3.06E+06   | 15.5%       |
|          |          |          |          |          | Sum =      | 546.1%      |

Mix RG-004 E\* Results: Data

| Red freq | Data1    | Red freq | Data2    | Red freq | Data3    | Red freq | Data4    |
|----------|----------|----------|----------|----------|----------|----------|----------|
| 9.84E+02 | 1.35E+06 | 4.04E+02 | 1.03E+06 | 1.17E+03 | 1.13E+06 | 7.85E+02 | 1.26E+06 |
| 4.92E+03 | 1.77E+06 | 2.02E+03 | 1.35E+06 | 5.83E+03 | 1.47E+06 | 3.92E+03 | 1.59E+06 |
| 9.84E+03 | 1.93E+06 | 4.04E+03 | 1.48E+06 | 1.17E+04 | 1.62E+06 | 7.85E+03 | 1.73E+06 |
| 4.92E+04 | 2.36E+06 | 2.02E+04 | 1.88E+06 | 5.83E+04 | 2.02E+06 | 3.92E+04 | 2.12E+06 |
| 9.84E+04 | 2.45E+06 | 4.04E+04 | 2.06E+06 | 1.17E+05 | 2.19E+06 | 7.85E+04 | 2.27E+06 |
| 2.46E+05 | 2.59E+06 | 1.01E+05 | 2.27E+06 | 2.91E+05 | 2.41E+06 | 1.96E+05 | 2.43E+06 |
| 1.10E+01 | 4.75E+05 | 1.17E+01 | 3.64E+05 | 6.59E-01 | 1.41E+05 | 8.11E+00 | 3.64E+05 |
| 5.49E+01 | 6.66E+05 | 5.86E+01 | 5.85E+05 | 3.29E+00 | 2.36E+05 | 4.06E+01 | 6.07E+05 |
| 1.10E+02 | 8.10E+05 | 1.17E+02 | 7.14E+05 | 6.59E+00 | 2.95E+05 | 8.11E+01 | 7.27E+05 |
| 5.49E+02 | 1.19E+06 | 5.86E+02 | 1.07E+06 | 3.29E+01 | 4.53E+05 | 4.06E+02 | 1.07E+06 |
| 1.10E+03 | 1.38E+06 | 1.17E+03 | 1.24E+06 | 6.59E+01 | 5.29E+05 | 8.11E+02 | 1.24E+06 |
| 2.75E+03 | 1.59E+06 | 2.93E+03 | 1.48E+06 | 1.65E+02 | 6.22E+05 | 2.03E+03 | 1.44E+06 |
| 1.00E-01 | 6.67E+04 | 1.00E-01 | 6.03E+04 | 1.00E-01 | 6.19E+04 | 1.00E-01 | 7.02E+04 |
| 5.00E-01 | 1.24E+05 | 5.00E-01 | 1.07E+05 | 5.00E-01 | 1.11E+05 | 5.01E-01 | 1.29E+05 |
| 1.00E+00 | 1.62E+05 | 1.00E+00 | 1.38E+05 | 1.00E+00 | 1.43E+05 | 1.00E+00 | 1.67E+05 |
| 5.00E+00 | 3.09E+05 | 5.00E+00 | 2.58E+05 | 5.00E+00 | 2.66E+05 | 5.00E+00 | 3.05E+05 |
| 1.00E+01 | 4.03E+05 | 1.00E+01 | 3.36E+05 | 1.00E+01 | 3.45E+05 | 1.00E+01 | 3.85E+05 |
| 2.50E+01 | 5.50E+05 | 2.50E+01 | 4.65E+05 | 2.50E+01 | 4.70E+05 | 2.50E+01 | 5.17E+05 |
| 3.43E-03 | 2.07E+04 | 5.13E-03 | 2.77E+04 | 3.46E-03 | 2.69E+04 | 4.71E-03 | 3.01E+04 |
| 1.71E-02 | 3.80E+04 | 2.57E-02 | 4.05E+04 | 1.73E-02 | 4.08E+04 | 2.36E-02 | 4.52E+04 |
| 3.42E-02 | 4.59E+04 | 5.13E-02 | 4.87E+04 | 3.46E-02 | 4.70E+04 | 4.71E-02 | 5.48E+04 |
| 1.71E-01 | 7.79E+04 | 2.57E-01 | 8.09E+04 | 1.73E-01 | 7.64E+04 | 2.35E-01 | 9.39E+04 |
| 3.43E-01 | 1.05E+05 | 5.13E-01 | 1.06E+05 | 3.46E-01 | 1.02E+05 | 4.71E-01 | 1.24E+05 |
| 8.56E-01 | 1.69E+05 | 1.28E+00 | 1.62E+05 | 8.66E-01 | 1.52E+05 | 1.18E+00 | 1.85E+05 |
| 3.89E-04 | 1.67E+04 | 3.30E-04 | 1.48E+04 | 2.70E-04 | 1.56E+04 | 3.44E-04 | 1.61E+04 |
| 1.94E-03 | 2.17E+04 | 1.65E-03 | 2.28E+04 | 1.35E-03 | 2.35E+04 | 1.72E-03 | 2.55E+04 |
| 3.89E-03 | 2.42E+04 | 3.30E-03 | 2.63E+04 | 2.70E-03 | 2.50E+04 | 3.44E-03 | 2.82E+04 |
| 1.95E-02 | 3.32E+04 | 1.65E-02 | 3.27E+04 | 1.35E-02 | 3.32E+04 | 1.72E-02 | 3.79E+04 |
| 3.89E-02 | 4.19E+04 | 3.30E-02 | 3.94E+04 | 2.70E-02 | 4.08E+04 | 3.44E-02 | 4.70E+04 |
| 9.72E-02 | 7.66E+04 | 8.25E-02 | 6.23E+04 | 6.75E-02 | 6.85E+04 | 8.60E-02 | 7.39E+04 |

Mix RG-004 E\* Results: Fit

| freq     | Fit1     | Fit2     | Fit3     | Fit4     | AlldataFit | deltaE/avgE |
|----------|----------|----------|----------|----------|------------|-------------|
| 1.00E-05 | 1.02E+04 | 1.17E+04 | 1.04E+04 | 1.17E+04 | 1.10E+04   | 14.0%       |
| 3.16E-05 | 1.11E+04 | 1.27E+04 | 1.17E+04 | 1.29E+04 | 1.21E+04   | 14.4%       |
| 1.00E-04 | 1.26E+04 | 1.41E+04 | 1.35E+04 | 1.46E+04 | 1.37E+04   | 14.7%       |
| 3.16E-04 | 1.47E+04 | 1.61E+04 | 1.61E+04 | 1.70E+04 | 1.60E+04   | 14.8%       |
| 1.00E-03 | 1.79E+04 | 1.92E+04 | 2.00E+04 | 2.07E+04 | 1.94E+04   | 14.7%       |
| 3.16E-03 | 2.29E+04 | 2.38E+04 | 2.57E+04 | 2.64E+04 | 2.47E+04   | 14.1%       |
| 1.00E-02 | 3.10E+04 | 3.11E+04 | 3.43E+04 | 3.53E+04 | 3.29E+04   | 12.9%       |
| 3.16E-02 | 4.43E+04 | 4.26E+04 | 4.76E+04 | 4.94E+04 | 4.59E+04   | 14.8%       |
| 1.00E-01 | 6.65E+04 | 6.12E+04 | 6.81E+04 | 7.21E+04 | 6.70E+04   | 16.3%       |
| 3.16E-01 | 1.03E+05 | 9.15E+04 | 9.98E+04 | 1.09E+05 | 1.01E+05   | 17.0%       |
| 1.00E+00 | 1.64E+05 | 1.40E+05 | 1.48E+05 | 1.66E+05 | 1.55E+05   | 16.9%       |
| 3.16E+00 | 2.59E+05 | 2.17E+05 | 2.20E+05 | 2.55E+05 | 2.37E+05   | 17.7%       |
| 1.00E+01 | 3.99E+05 | 3.31E+05 | 3.22E+05 | 3.83E+05 | 3.58E+05   | 21.5%       |
| 3.16E+01 | 5.91E+05 | 4.90E+05 | 4.61E+05 | 5.56E+05 | 5.23E+05   | 24.8%       |
| 1.00E+02 | 8.32E+05 | 6.97E+05 | 6.39E+05 | 7.74E+05 | 7.32E+05   | 26.2%       |
| 3.16E+02 | 1.11E+06 | 9.43E+05 | 8.54E+05 | 1.03E+06 | 9.77E+05   | 25.8%       |
| 1.00E+03 | 1.40E+06 | 1.21E+06 | 1.10E+06 | 1.30E+06 | 1.24E+06   | 24.1%       |
| 3.16E+03 | 1.68E+06 | 1.49E+06 | 1.36E+06 | 1.57E+06 | 1.51E+06   | 21.4%       |
| 1.00E+04 | 1.95E+06 | 1.75E+06 | 1.62E+06 | 1.83E+06 | 1.77E+06   | 18.3%       |
| 3.16E+04 | 2.18E+06 | 1.99E+06 | 1.87E+06 | 2.07E+06 | 2.00E+06   | 14.9%       |
| 1.00E+05 | 2.37E+06 | 2.20E+06 | 2.11E+06 | 2.27E+06 | 2.20E+06   | 11.5%       |
| 3.16E+05 | 2.52E+06 | 2.37E+06 | 2.32E+06 | 2.44E+06 | 2.37E+06   | 8.4%        |
| 1.00E+06 | 2.65E+06 | 2.50E+06 | 2.50E+06 | 2.57E+06 | 2.51E+06   | 5.6%        |
| 3.16E+06 | 2.74E+06 | 2.61E+06 | 2.66E+06 | 2.68E+06 | 2.62E+06   | 4.8%        |
| 1.00E+07 | 2.81E+06 | 2.70E+06 | 2.79E+06 | 2.76E+06 | 2.71E+06   | 4.2%        |
| 3.16E+07 | 2.87E+06 | 2.76E+06 | 2.89E+06 | 2.83E+06 | 2.78E+06   | 4.5%        |
| 1.00E+08 | 2.91E+06 | 2.81E+06 | 2.98E+06 | 2.88E+06 | 2.83E+06   | 5.7%        |
| 3.16E+08 | 2.94E+06 | 2.85E+06 | 3.05E+06 | 2.92E+06 | 2.87E+06   | 6.6%        |
| 1.00E+09 | 2.96E+06 | 2.88E+06 | 3.10E+06 | 2.95E+06 | 2.90E+06   | 7.5%        |
| 3.16E+09 | 2.98E+06 | 2.90E+06 | 3.14E+06 | 2.97E+06 | 2.92E+06   | 8.1%        |
|          |          |          |          |          | Sum =      | 426.1%      |

Mix T-005 E\* Results: Data

| Red freq | Data1    | Red freq | Data2    | Red freq | Data3    | Red freq | Data4    | Red freq | Data5    |
|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|
| 3.90E+02 | 8.54E+05 | 2.64E+02 | 7.33E+05 | 4.51E+02 | 8.82E+05 | 5.71E+02 | 7.89E+05 | 1.15E+03 | 1.06E+06 |
| 1.95E+03 | 1.12E+06 | 1.32E+03 | 9.43E+05 | 2.26E+03 | 1.16E+06 | 2.85E+03 | 1.09E+06 | 5.76E+03 | 1.29E+06 |
| 3.90E+03 | 1.24E+06 | 2.64E+03 | 1.06E+06 | 4.51E+03 | 1.30E+06 | 5.71E+03 | 1.26E+06 | 1.15E+04 | 1.44E+06 |
| 1.95E+04 | 1.61E+06 | 1.32E+04 | 1.41E+06 | 2.26E+04 | 1.68E+06 | 2.85E+04 | 1.65E+06 | 5.76E+04 | 1.81E+06 |
| 3.90E+04 | 1.77E+06 | 2.64E+04 | 1.55E+06 | 4.51E+04 | 1.85E+06 | 5.71E+04 | 1.84E+06 | 1.15E+05 | 1.97E+06 |
| 9.75E+04 | 2.01E+06 | 6.60E+04 | 1.70E+06 | 1.13E+05 | 2.02E+06 | 1.43E+05 | 2.07E+06 | 2.88E+05 | 2.22E+06 |
| 1.18E+01 | 3.13E+05 | 9.87E+00 | 2.53E+05 | 1.09E+01 | 2.90E+05 | 6.48E+00 | 1.96E+05 | 9.02E+00 | 2.36E+05 |
| 5.91E+01 | 4.65E+05 | 4.94E+01 | 4.28E+05 | 5.45E+01 | 4.58E+05 | 3.24E+01 | 3.24E+05 | 4.51E+01 | 4.11E+05 |
| 1.18E+02 | 5.67E+05 | 9.87E+01 | 5.27E+05 | 1.09E+02 | 5.61E+05 | 6.48E+01 | 4.01E+05 | 9.02E+01 | 5.07E+05 |
| 5.91E+02 | 8.78E+05 | 4.93E+02 | 7.96E+05 | 5.45E+02 | 8.70E+05 | 3.24E+02 | 6.54E+05 | 4.51E+02 | 7.94E+05 |
| 1.18E+03 | 1.03E+06 | 9.87E+02 | 9.36E+05 | 1.09E+03 | 1.03E+06 | 6.48E+02 | 7.80E+05 | 9.02E+02 | 9.32E+05 |
| 2.95E+03 | 1.24E+06 | 2.47E+03 | 1.09E+06 | 2.72E+03 | 1.23E+06 | 1.62E+03 | 9.54E+05 | 2.25E+03 | 1.12E+06 |
| 1.00E-01 | 5.12E+04 | 1.00E-01 | 4.65E+04 | 1.00E-01 | 4.65E+04 | 1.00E-01 | 4.29E+04 | 1.00E-01 | 4.56E+04 |
| 5.00E-01 | 9.07E+04 | 5.01E-01 | 8.53E+04 | 5.01E-01 | 8.89E+04 | 5.00E-01 | 7.10E+04 | 5.01E-01 | 7.81E+04 |
| 1.00E+00 | 1.14E+05 | 1.00E+00 | 1.08E+05 | 1.00E+00 | 1.13E+05 | 1.00E+00 | 9.03E+04 | 1.00E+00 | 9.94E+04 |
| 5.00E+00 | 2.10E+05 | 5.00E+00 | 1.96E+05 | 5.00E+00 | 2.08E+05 | 5.00E+00 | 1.63E+05 | 5.00E+00 | 1.82E+05 |
| 1.00E+01 | 2.75E+05 | 1.00E+01 | 2.55E+05 | 1.00E+01 | 2.72E+05 | 1.00E+01 | 2.16E+05 | 1.00E+01 | 2.39E+05 |
| 2.50E+01 | 3.91E+05 | 2.50E+01 | 3.53E+05 | 2.50E+01 | 3.73E+05 | 2.50E+01 | 3.10E+05 | 2.50E+01 | 3.41E+05 |
| 5.27E-03 | 2.90E+04 | 3.50E-03 | 2.39E+04 | 4.90E-03 | 2.64E+04 | 4.45E-03 | 1.72E+04 | 5.60E-03 | 1.95E+04 |
| 2.63E-02 | 4.25E+04 | 1.75E-02 | 3.63E+04 | 2.45E-02 | 4.00E+04 | 2.23E-02 | 3.15E+04 | 2.80E-02 | 3.44E+04 |
| 5.27E-02 | 4.86E+04 | 3.50E-02 | 4.05E+04 | 4.90E-02 | 4.53E+04 | 4.45E-02 | 3.59E+04 | 5.60E-02 | 4.30E+04 |
| 2.63E-01 | 7.04E+04 | 1.75E-01 | 5.68E+04 | 2.45E-01 | 6.66E+04 | 2.23E-01 | 5.31E+04 | 2.80E-01 | 6.22E+04 |
| 5.27E-01 | 9.03E+04 | 3.50E-01 | 7.19E+04 | 4.90E-01 | 8.46E+04 | 4.45E-01 | 6.89E+04 | 5.60E-01 | 7.95E+04 |
| 1.32E+00 | 1.30E+05 | 8.76E-01 | 1.03E+05 | 1.22E+00 | 1.20E+05 | 1.11E+00 | 1.04E+05 | 1.40E+00 | 1.20E+05 |
| 6.31E-04 | 1.92E+04 | 5.57E-04 | 1.69E+04 | 6.58E-04 | 1.75E+04 | 7.56E-04 | 1.62E+04 | 6.45E-04 | 1.72E+04 |
| 3.16E-03 | 3.00E+04 | 2.78E-03 | 2.60E+04 | 3.29E-03 | 2.83E+04 | 3.78E-03 | 2.32E+04 | 3.23E-03 | 2.39E+04 |
| 6.32E-03 | 3.19E+04 | 5.56E-03 | 2.90E+04 | 6.58E-03 | 3.01E+04 | 7.56E-03 | 2.45E+04 | 6.45E-03 | 2.57E+04 |
| 3.16E-02 | 3.90E+04 | 2.78E-02 | 3.42E+04 | 3.29E-02 | 3.68E+04 | 3.78E-02 | 3.05E+04 | 3.23E-02 | 3.19E+04 |
| 6.31E-02 | 4.50E+04 | 5.57E-02 | 3.99E+04 | 6.58E-02 | 4.26E+04 | 7.56E-02 | 3.58E+04 | 6.45E-02 | 3.80E+04 |
| 1.58E-01 | 6.76E+04 | 1.39E-01 | 5.93E+04 | 1.65E-01 | 6.55E+04 | 1.89E-01 | 5.51E+04 | 1.61E-01 | 5.47E+04 |

Mix T-005 E\* Results: Fit

| freq     | Fit1     | Fit2     | Fit3     | Fit4     | Fit5     | AlldataFit | deltaE/avgE |
|----------|----------|----------|----------|----------|----------|------------|-------------|
| 1.00E-05 | 1.61E+04 | 1.47E+04 | 1.46E+04 | 1.08E+04 | 1.25E+04 | 1.37E+04   | 38.4%       |
| 3.16E-05 | 1.70E+04 | 1.55E+04 | 1.54E+04 | 1.16E+04 | 1.32E+04 | 1.45E+04   | 37.1%       |
| 1.00E-04 | 1.82E+04 | 1.65E+04 | 1.66E+04 | 1.26E+04 | 1.42E+04 | 1.56E+04   | 35.5%       |
| 3.16E-04 | 1.99E+04 | 1.81E+04 | 1.83E+04 | 1.41E+04 | 1.57E+04 | 1.72E+04   | 33.8%       |
| 1.00E-03 | 2.25E+04 | 2.04E+04 | 2.07E+04 | 1.63E+04 | 1.79E+04 | 1.96E+04   | 31.8%       |
| 3.16E-03 | 2.64E+04 | 2.39E+04 | 2.44E+04 | 1.95E+04 | 2.12E+04 | 2.31E+04   | 29.8%       |
| 1.00E-02 | 3.23E+04 | 2.92E+04 | 3.01E+04 | 2.44E+04 | 2.62E+04 | 2.85E+04   | 27.9%       |
| 3.16E-02 | 4.14E+04 | 3.76E+04 | 3.89E+04 | 3.18E+04 | 3.40E+04 | 3.68E+04   | 26.2%       |
| 1.00E-01 | 5.60E+04 | 5.10E+04 | 5.30E+04 | 4.36E+04 | 4.66E+04 | 5.01E+04   | 24.8%       |
| 3.16E-01 | 7.93E+04 | 7.27E+04 | 7.58E+04 | 6.23E+04 | 6.70E+04 | 7.15E+04   | 23.8%       |
| 1.00E+00 | 1.17E+05 | 1.08E+05 | 1.13E+05 | 9.22E+04 | 1.00E+05 | 1.06E+05   | 23.1%       |
| 3.16E+00 | 1.76E+05 | 1.64E+05 | 1.71E+05 | 1.40E+05 | 1.54E+05 | 1.61E+05   | 22.4%       |
| 1.00E+01 | 2.66E+05 | 2.49E+05 | 2.61E+05 | 2.13E+05 | 2.37E+05 | 2.45E+05   | 21.6%       |
| 3.16E+01 | 3.95E+05 | 3.71E+05 | 3.91E+05 | 3.20E+05 | 3.59E+05 | 3.67E+05   | 20.3%       |
| 1.00E+02 | 5.67E+05 | 5.32E+05 | 5.65E+05 | 4.70E+05 | 5.26E+05 | 5.31E+05   | 18.3%       |
| 3.16E+02 | 7.78E+05 | 7.26E+05 | 7.80E+05 | 6.63E+05 | 7.35E+05 | 7.35E+05   | 16.0%       |
| 1.00E+03 | 1.02E+06 | 9.40E+05 | 1.02E+06 | 8.94E+05 | 9.76E+05 | 9.68E+05   | 13.3%       |
| 3.16E+03 | 1.26E+06 | 1.16E+06 | 1.28E+06 | 1.15E+06 | 1.23E+06 | 1.21E+06   | 10.3%       |
| 1.00E+04 | 1.50E+06 | 1.36E+06 | 1.52E+06 | 1.42E+06 | 1.48E+06 | 1.45E+06   | 10.9%       |
| 3.16E+04 | 1.71E+06 | 1.54E+06 | 1.74E+06 | 1.67E+06 | 1.71E+06 | 1.67E+06   | 11.9%       |
| 1.00E+05 | 1.89E+06 | 1.69E+06 | 1.93E+06 | 1.91E+06 | 1.91E+06 | 1.86E+06   | 12.8%       |
| 3.16E+05 | 2.04E+06 | 1.82E+06 | 2.09E+06 | 2.12E+06 | 2.08E+06 | 2.02E+06   | 15.0%       |
| 1.00E+06 | 2.16E+06 | 1.91E+06 | 2.22E+06 | 2.30E+06 | 2.22E+06 | 2.15E+06   | 17.8%       |
| 3.16E+06 | 2.26E+06 | 1.99E+06 | 2.31E+06 | 2.44E+06 | 2.32E+06 | 2.25E+06   | 20.2%       |
| 1.00E+07 | 2.33E+06 | 2.04E+06 | 2.39E+06 | 2.56E+06 | 2.40E+06 | 2.33E+06   | 22.2%       |
| 3.16E+07 | 2.38E+06 | 2.08E+06 | 2.45E+06 | 2.65E+06 | 2.47E+06 | 2.39E+06   | 23.8%       |
| 1.00E+08 | 2.42E+06 | 2.11E+06 | 2.49E+06 | 2.73E+06 | 2.51E+06 | 2.44E+06   | 25.0%       |
| 3.16E+08 | 2.45E+06 | 2.14E+06 | 2.52E+06 | 2.78E+06 | 2.55E+06 | 2.47E+06   | 26.0%       |
| 1.00E+09 | 2.47E+06 | 2.15E+06 | 2.54E+06 | 2.83E+06 | 2.58E+06 | 2.50E+06   | 26.8%       |
| 3.16E+09 | 2.49E+06 | 2.16E+06 | 2.56E+06 | 2.86E+06 | 2.60E+06 | 2.52E+06   | 27.5%       |
|          |          |          |          |          | Sum =    |            | 694.2%      |

Mix RT-006 E\* Results: Data

| Red freq | Data1    | Red freq | Data2    | Red freq | Data3    | Red freq | Data4    |
|----------|----------|----------|----------|----------|----------|----------|----------|
| 1.41E+03 | 1.34E+06 | 1.22E+03 | 1.12E+06 | 3.32E+03 | 1.47E+06 | 2.32E+03 | 1.02E+06 |
| 7.03E+03 | 1.60E+06 | 6.10E+03 | 1.46E+06 | 1.66E+04 | 1.71E+06 | 1.16E+04 | 1.22E+06 |
| 1.40E+04 | 1.80E+06 | 1.22E+04 | 1.59E+06 | 3.32E+04 | 1.93E+06 | 2.32E+04 | 1.34E+06 |
| 7.03E+04 | 2.17E+06 | 6.10E+04 | 1.94E+06 | 1.66E+05 | 2.35E+06 | 1.16E+05 | 1.67E+06 |
| 1.41E+05 | 2.34E+06 | 1.22E+05 | 2.08E+06 | 3.32E+05 | 2.52E+06 | 2.32E+05 | 1.80E+06 |
| 3.51E+05 | 2.51E+06 | 3.05E+05 | 2.25E+06 | 8.30E+05 | 2.83E+06 | 5.81E+05 | 1.95E+06 |
| 9.47E+00 | 3.80E+05 | 7.48E+00 | 2.97E+05 | 1.38E+01 | 3.50E+05 | 1.09E+00 | 1.35E+05 |
| 4.73E+01 | 5.95E+05 | 3.74E+01 | 4.59E+05 | 6.91E+01 | 5.50E+05 | 5.47E+00 | 2.21E+05 |
| 9.47E+01 | 7.10E+05 | 7.49E+01 | 5.58E+05 | 1.38E+02 | 6.68E+05 | 1.09E+01 | 2.67E+05 |
| 4.74E+02 | 1.03E+06 | 3.74E+02 | 8.53E+05 | 6.91E+02 | 9.90E+05 | 5.47E+01 | 4.00E+05 |
| 9.47E+02 | 1.19E+06 | 7.48E+02 | 9.99E+05 | 1.38E+03 | 1.15E+06 | 1.09E+02 | 4.60E+05 |
| 2.37E+03 | 1.37E+06 | 1.87E+03 | 1.18E+06 | 3.46E+03 | 1.38E+06 | 2.74E+02 | 5.28E+05 |
| 1.00E-01 | 7.04E+04 | 1.00E-01 | 5.17E+04 | 1.00E-01 | 4.97E+04 | 1.00E-01 | 4.44E+04 |
| 5.00E-01 | 1.23E+05 | 5.00E-01 | 9.54E+04 | 5.00E-01 | 9.23E+04 | 5.00E-01 | 8.58E+04 |
| 9.99E-01 | 1.57E+05 | 1.00E+00 | 1.24E+05 | 1.00E+00 | 1.20E+05 | 1.00E+00 | 1.09E+05 |
| 5.00E+00 | 2.84E+05 | 5.00E+00 | 2.28E+05 | 5.00E+00 | 2.26E+05 | 5.00E+00 | 2.04E+05 |
| 1.00E+01 | 3.65E+05 | 1.00E+01 | 3.01E+05 | 1.00E+01 | 2.97E+05 | 1.00E+01 | 2.65E+05 |
| 2.50E+01 | 4.95E+05 | 2.50E+01 | 4.20E+05 | 2.50E+01 | 4.17E+05 | 2.50E+01 | 3.65E+05 |
| 4.11E-03 | 2.39E+04 | 3.56E-03 | 1.49E+04 | 3.57E-03 | 2.03E+04 | 3.97E-03 | 2.12E+04 |
| 2.06E-02 | 4.58E+04 | 1.78E-02 | 3.27E+04 | 1.79E-02 | 3.16E+04 | 1.99E-02 | 3.29E+04 |
| 4.11E-02 | 5.51E+04 | 3.56E-02 | 3.89E+04 | 3.57E-02 | 3.74E+04 | 3.97E-02 | 3.85E+04 |
| 2.06E-01 | 8.92E+04 | 1.78E-01 | 6.26E+04 | 1.79E-01 | 6.00E+04 | 1.98E-01 | 6.13E+04 |
| 4.11E-01 | 1.15E+05 | 3.56E-01 | 8.23E+04 | 3.57E-01 | 7.93E+04 | 3.97E-01 | 8.04E+04 |
| 1.03E+00 | 1.72E+05 | 8.91E-01 | 1.30E+05 | 8.93E-01 | 1.25E+05 | 9.92E-01 | 1.26E+05 |
| 3.94E-04 | 1.93E+04 | 3.66E-04 | 1.28E+04 | No Data  | No Data  | 1.72E-04 | 1.77E+04 |
| 1.97E-03 | 2.86E+04 | 1.83E-03 | 1.89E+04 | No Data  | No Data  | 8.60E-04 | 1.71E+04 |
| 3.94E-03 | 3.05E+04 | 3.66E-03 | 2.06E+04 | No Data  | No Data  | 1.72E-03 | 1.87E+04 |
| 1.97E-02 | 3.95E+04 | 1.83E-02 | 2.70E+04 | No Data  | No Data  | 8.58E-03 | 2.38E+04 |
| 3.94E-02 | 4.72E+04 | 3.66E-02 | 3.30E+04 | No Data  | No Data  | 1.72E-02 | 2.92E+04 |
| 9.86E-02 | 7.34E+04 | 9.15E-02 | 5.32E+04 | No Data  | No Data  | 4.29E-02 | 4.63E+04 |



Mix RT-006 E\* Results: Fit

| freq     | Fit1     | Fit2     | Fit3     | Fit4     | AlldataFit | deltaE/avgE |
|----------|----------|----------|----------|----------|------------|-------------|
| 1.00E-05 | 1.29E+04 | 8.10E+03 | 8.09E+03 | 1.13E+04 | 1.10E+04   | 47.7%       |
| 3.16E-05 | 1.41E+04 | 8.94E+03 | 9.00E+03 | 1.22E+04 | 1.19E+04   | 46.6%       |
| 1.00E-04 | 1.58E+04 | 1.01E+04 | 1.03E+04 | 1.34E+04 | 1.33E+04   | 45.4%       |
| 3.16E-04 | 1.83E+04 | 1.19E+04 | 1.22E+04 | 1.53E+04 | 1.52E+04   | 44.0%       |
| 1.00E-03 | 2.19E+04 | 1.46E+04 | 1.50E+04 | 1.79E+04 | 1.80E+04   | 42.2%       |
| 3.16E-03 | 2.74E+04 | 1.87E+04 | 1.92E+04 | 2.19E+04 | 2.24E+04   | 40.2%       |
| 1.00E-02 | 3.60E+04 | 2.51E+04 | 2.57E+04 | 2.79E+04 | 2.91E+04   | 37.8%       |
| 3.16E-02 | 4.94E+04 | 3.55E+04 | 3.61E+04 | 3.73E+04 | 3.97E+04   | 35.2%       |
| 1.00E-01 | 7.08E+04 | 5.24E+04 | 5.26E+04 | 5.20E+04 | 5.68E+04   | 33.1%       |
| 3.16E-01 | 1.05E+05 | 8.01E+04 | 7.94E+04 | 7.52E+04 | 8.44E+04   | 35.2%       |
| 1.00E+00 | 1.59E+05 | 1.25E+05 | 1.22E+05 | 1.11E+05 | 1.29E+05   | 36.8%       |
| 3.16E+00 | 2.41E+05 | 1.94E+05 | 1.88E+05 | 1.67E+05 | 1.98E+05   | 37.6%       |
| 1.00E+01 | 3.61E+05 | 2.98E+05 | 2.88E+05 | 2.48E+05 | 3.00E+05   | 37.8%       |
| 3.16E+01 | 5.24E+05 | 4.42E+05 | 4.29E+05 | 3.60E+05 | 4.43E+05   | 37.3%       |
| 1.00E+02 | 7.31E+05 | 6.27E+05 | 6.16E+05 | 5.05E+05 | 6.29E+05   | 36.4%       |
| 3.16E+02 | 9.74E+05 | 8.48E+05 | 8.49E+05 | 6.79E+05 | 8.51E+05   | 35.2%       |
| 1.00E+03 | 1.24E+06 | 1.09E+06 | 1.12E+06 | 8.72E+05 | 1.10E+06   | 34.0%       |
| 3.16E+03 | 1.51E+06 | 1.34E+06 | 1.41E+06 | 1.07E+06 | 1.35E+06   | 32.7%       |
| 1.00E+04 | 1.76E+06 | 1.58E+06 | 1.70E+06 | 1.27E+06 | 1.59E+06   | 31.6%       |
| 3.16E+04 | 2.00E+06 | 1.80E+06 | 1.98E+06 | 1.44E+06 | 1.81E+06   | 30.6%       |
| 1.00E+05 | 2.20E+06 | 1.99E+06 | 2.24E+06 | 1.60E+06 | 2.00E+06   | 31.8%       |
| 3.16E+05 | 2.37E+06 | 2.15E+06 | 2.47E+06 | 1.73E+06 | 2.16E+06   | 33.6%       |
| 1.00E+06 | 2.50E+06 | 2.29E+06 | 2.66E+06 | 1.84E+06 | 2.29E+06   | 35.3%       |
| 3.16E+06 | 2.61E+06 | 2.39E+06 | 2.82E+06 | 1.93E+06 | 2.39E+06   | 36.6%       |
| 1.00E+07 | 2.70E+06 | 2.48E+06 | 2.96E+06 | 2.00E+06 | 2.48E+06   | 37.8%       |
| 3.16E+07 | 2.77E+06 | 2.54E+06 | 3.06E+06 | 2.05E+06 | 2.54E+06   | 38.8%       |
| 1.00E+08 | 2.82E+06 | 2.59E+06 | 3.15E+06 | 2.09E+06 | 2.59E+06   | 39.6%       |
| 3.16E+08 | 2.86E+06 | 2.63E+06 | 3.22E+06 | 2.13E+06 | 2.62E+06   | 40.2%       |
| 1.00E+09 | 2.89E+06 | 2.66E+06 | 3.27E+06 | 2.15E+06 | 2.65E+06   | 40.8%       |
| 3.16E+09 | 2.91E+06 | 2.68E+06 | 3.31E+06 | 2.17E+06 | 2.67E+06   | 41.2%       |
|          |          |          |          |          | Sum =      | 1133.2%     |