



Silica Fume - Quality Assurance Manual 2016

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1 COMPANY BACKGROUND

Norchem, Inc., a wholly owned affiliate of Globe Metallurgical, Inc., and part of the Ferroglobe group of companies, with offices in New York and Florida, is a leading supplier of silica fume products and developer of silica fume end-user applications. Since 1975, Norchem, Inc. has been an innovator of silica fume products in cementitious applications, primarily as a concrete admixture in the construction industry.

Silica Fume is the by-product of ferrosilicon and silicon alloy production. The material, an amorphous silicon dioxide, acts as a Pozzolan in cementitious applications, is generated in electric-arc furnaces as a gas during the reduction of very pure quartz and metallurgical grade coal.

The Ferroglobe group operates 26 plants worldwide, with 6 based in North America located in Beverly, Ohio; Selma, Alabama; Alloy, West Virginia; Niagara Falls, New York; Bridgeport, Alabama and Bécancour, Québec. These plants have an annual silica fume production capacity of over 100,000 tones.

Silica fume from these plants is supplied to both the construction market and non-cementitious industries throughout North America and is exported worldwide. Some other market applications for silica fume include oil/gas well cementing, refractory products, fiber-cement and wallboard products, polymers and elastomers. Through research and development programs, Norchem continues to remain on the leading edge of admixture technology with high quality, and uniform, silica fume products.

Norchem has developed and successfully deployed silica fume in end-use applications that include:

- Highway and bridge decks
- Secondary containment vessels
- Shotcrete highway, rail, mining and water tunnels
- Hazardous waste and water treatment facilities
- Rehabilitation of concrete sub-structures
- Parking Garages
- Oil and Gas well grouting
- High-strength structures
- Chemical Environments
- Maritime Structures

These applications have rigorous demands for quality, durability and strength that benefit from Norchem's reputation for technical expertise and customer support. Norchem Silica Fume is distributed to ready-mix and pre-cast concrete producers via a wide network of concrete admixture distributors (see section 9 for a full list of those distributors).

Silica fume can be packaged in 25lb concrete ready bags, 50lb paper bags and 2,200lb. supersacks. For bulk deliveries silica fume is loaded into tanker trucks, freight cars and shipping containers.

2 INTRODUCTION

This manual outlines the basic requirements of the Norchem, Inc. Quality Assurance program. The program is structured for the multiple plant environments of our operations and is based on the testing requirements of American Society of Testing Materials (ASTM) Standard Specification for Silica Fume Used in Cementitious Mixtures ASTM C 1240. In addition to compliance with the ASTM standard our testing practices also conform to American Association of State Highway and Transportation Officials (AASHTO) Standard Specification for Use of Silica Fume as a Mineral Admixture in Hydraulic-Cement Concrete, Mortar, and Grout, AASHTO M 307, High-Reactivity Pozzolans for Use in Hydraulic-Cement Concrete, Mortar, and Grout AASHTO M 321 and the Canadian Standards Association (CSA) Cementitious Materials Compendium Standard specification A3000-13.

The manual references Standard Operating Procedures (SOP) used in our daily operations. These SOP procedures include practices designed by specific scientific equipment manufacturers as well as procedures implemented “in house” to assure test or operational reproducibility and repeatability. More information on these procedures is available on request.

DEFINITIONS

Silica Fume – very fine pozzolanic material, composed of amorphous silica produced by electric arc furnaces as a by-product of the production of elemental silicon or ferrosilicon alloys (also known as condensed silica fume and microsilica).

Pozzolan – a siliceous and aluminous material, which in itself possesses little or no cementitious value but which will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties.

Highly Reactive Pozzolans – amorphous silica of high silica content and purity possessing high pozzolanic activity, meeting the requirements of AASHTO M 321.

Amorphous – non-crystalline, or devoid of regular structure.

OBJECTIVES

The main purpose of this manual is to identify the Norchem, Inc. quality requirements employed in the production, processing, packaging and delivery of ASTM C 1240 Silica Fume. In cases where no specific job or customer-defined requirements are applied, the requirements outlined in this manual shall apply.

Norchem Quality Assurance requires the attention and cooperation of all management and production personnel. Thus it is our goal to make every effort to implement the necessary changes in equipment, procedures or personnel to produce the highest quality products achievable.

3 NORTH AMERICAN PLANT LOCATIONS

Beverly, Ohio
 County Road 32
 Beverly, Ohio 45715

Selma, Alabama
 2401 Old Montgomery Hwy.
 Selma, Alabama 36701

Bridgeport, Alabama
 101 Garner Rd.
 Bridgeport, Alabama 35740

Alloy, West Virginia
 Route 60 East
 Alloy, West Virginia 25002

Niagara Falls, New York
 3807 Highland Avenue
 Niagara Falls, New York 14305

Bécancour, Québec
 6500 Yvon-Trudeau Street
 Bécancour, Québec G9H 2V8

SILICON SMELTING OVERVIEW

To understand silica fume, one must first understand the smelting of silicon alloys. Silicon is smelted in large submerged arc electric furnaces (see photo below). A typical 20 MW furnace might be as large as 40 feet in diameter and 15 feet deep.



Submerged Arc Electric Furnaces

The furnace charge is quartzite, charcoal, wood chips and coal. The quartzite used for smelting silicon must be very pure and contain more than 99% silicon dioxide, typically called river rock, which has been washed to remove fines. A specific ratio of quartzite, coal and wood chips is continuously added to the top of the furnace while the silicon alloy is tapped from the bottom. The coal and wood chips provide an extremely high reducing atmosphere near the bottom of the furnace and especially around the ends of the electrodes. Hot gases rising through the burden, preheat it and the extreme temperature at the tips of the electrodes volatilizes the quartzite when it gets close to them. This silicon dioxide vapor reacts with the now porous, pure carbon to form carbon monoxide, silicon monoxide, silicon carbide and eventually silicon alloy.

The gas that rises through the burden and oxidizes at the top of the furnace gives us silica fume. When silica fume reaches the top of the furnace it is sucked up into the hood that covers the top of the furnace by powerful baghouse fans.



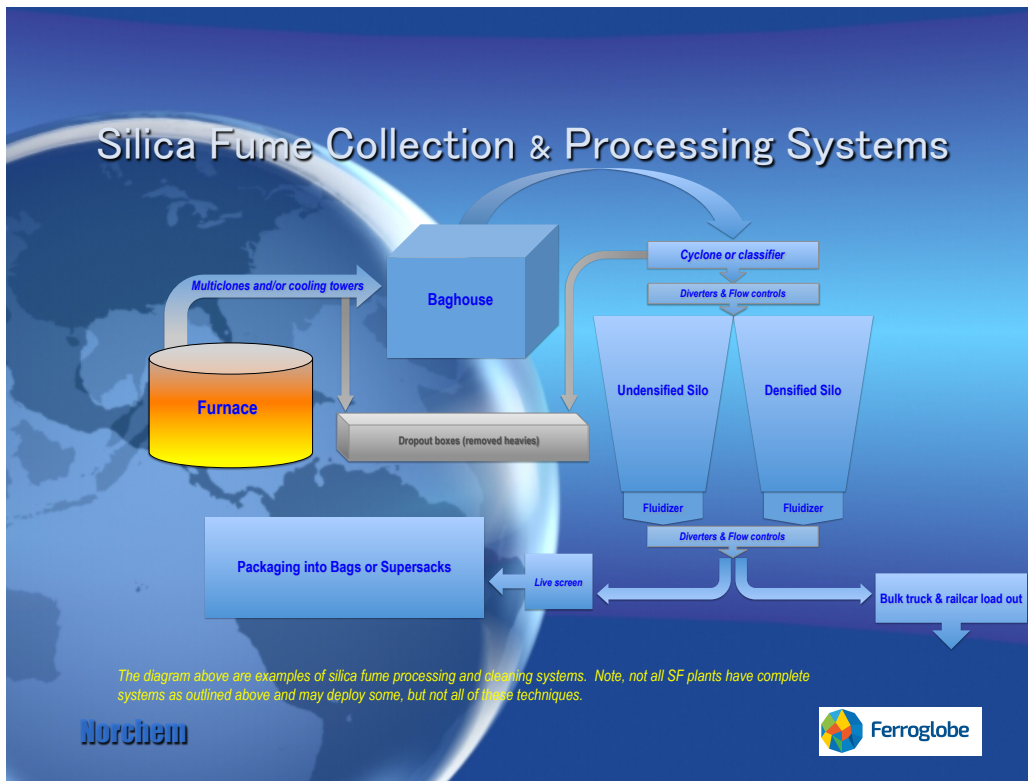
Silica Fume Collection & Processing Schematic

To meet environmental regulations, the bag house fans provide enough vacuuming action at the top of the furnace so that no gases escape into the atmosphere. Small particles of wood chips, coal fines, quartzite fines, are separated from the silica fume in the coolers and then in a series of cyclones that drop out what are known as “Heavies” from the silica fume. After this cleaning and cooling operation, silica fume is captured in baghouses. The silica fume is then transported from the baghouse into a silo either pneumatically or with a screw conveyor.

PROCESSING AND HANDLING

Typically the plants will have two different silo setups: one is for the storage, bulk loading and packaging of undensified silica fume and the other for the storage, bulk loading and packaging of densified silica fume. Undensified silica fume is used in refractories, gypsum wallboard and concrete rehabilitation products, whereas the densified product is made using undensified silica fume and some additional processing steps. Silica fume densification silos have a very fine screen that covers the bottom section upon which the silica fume rests. Air is blown into the silo under the screen. This air gently rises through the silica fume causing the individual silica particles to rub against each other. As the particles touch, the naturally occurring van der Waals

forces on their surface cause the particles to be attracted to each other. This attraction causes the particles to adhere to each other. The longer the air is allowed to flow through the silica fume bed, the greater the degree of agglomeration and correspondingly the density. Once the desired density is reached, the airflow to the silo is stopped. The newly densified silica fume can be shipped in bulk in pneumatic trucks or packaged in supersacks or paper bags. The value of the densification process is most evident in pneumatic bulk handling, i.e. silo storage and economic transportation because of the reduced cost of transporting 45-lbs/ft³ silica fume versus 15-lbs/ft³ material.



Typical Production Flow Chart for Silica Fume

4 QUALITY ASSURANCE DEPARTMENT

The QA team includes several members that jointly oversee Quality Assurance and Laboratory operations and are based in our Florida location and can be contacted at (772) 468-6110, or via email to info@norchem.com

LABORATORY

Norchem's laboratory includes both advanced material (silica fume and other pozzolans) and concrete testing. Internally the laboratory performs composite sample, random production, and lot number conformance, verification testing as well as research and product development work on silica fume and cementitious compounds.

The laboratory and its personnel have received recognition for work in developing the National Institute of Standards and Technology (NIST) Silica Fume Reference Material (SRM)[®] #2696 standard. Values are a product of extensive round-robin testing by a group of participants representing State Departments of Transportation, Universities, Commercial Test Laboratories, Distributors and Manufacturers. The Standard Reference Material is applicable for calibration curves, validation of existing laboratory values, confirmation of laboratory practices and simplifying the test process through standardization.

The laboratory and its principals have ongoing involvement in test development and coordination of round-robin programs for the American Society of Testing Materials (ASTM) C 1240 and AASHTO the American Association of State Highway and Transportation (AASHTO) M 307 standard specifications. Together with AASHTO a High-Reactive Pozzolans for Use in Hydraulic-Cement Concrete, Mortar, and Grout specification (M321) has been developed. It is the Norchem continued commitment to quality, product knowledge and research that has driven our growth.

LABORATORY TEST CAPABILITIES

In addition to the capabilities necessary for performing the conformance testing listed in ASTM C 1240, AASHTO M307 and CSA A3000-13, the laboratory has the following specialized abilities.

- **Specific Surface Area** – by ASTM C 1069 – Specific Surface Area of Alumina or Quartz by Nitrogen Adsorption.
- **Particle Size Distribution** – Using a Horiba Laser Scattering Particle Size Distribution Analyzer with built in 600-watt ultrasonic processor & temperature control.
- **Determination of the Silicon Content in Silica Fume** - Deutsche Norm DIN 51075 Part 4, testing of Ceramic materials, Chemical analysis of silicon carbide, Determination of the free Silicon Content.
- **Four Pin Resistivity and Rapid Chloride Permeability Testing (RCP)** - (estimated charge passed in coulombs) – by the TMB Associates Methodology and Formulation.
- **Water Demand** - of Hydraulic-Cement & Pozzolanic Materials in Mortars – Norchem Method.

ASTM C 305 – Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency.

ASTM C 109/C 109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or 50-mm Cube Specimens).

5 NORCHEM QUALITY ASSURANCE PROGRAM

The section of the plan outlined below is relevant to the sampling, testing and shipment of silica fume in accordance with the specifications and standards of ASTM, AASHTO and CSA.

SAMPLING

1. Sampling is conducted in accordance with ASTM C183 as modified by ASTM C 1240.
2. All samples, whether grab or composite, shall have a mass of at least 1 kg (2 lb.)
3. Daily “grab” samples are collected and combined into composite samples representing each 100 Mg (110 ton) of silica fume. Each subsequent 100 Mg (110 ton) of silica fume follows the same procedure. They are labeled, assigned lot number(s) and sent to the plant laboratory for chemical and physical analysis.
4. When four (4) composite samples or 400 Mg (440 ton) is collected the samples are combined (typically monthly) and sent to the Norchem Florida laboratory for extended physical testing.
5. At the end of three (3) months or collection of samples representing 3200 Mg (3520 ton), the three (3) individual monthly composite samples are combined and tested for Specific Surface Area (SSA), Accelerated Pozzolanic Strength Activity Index (PSAI), and all other required optional testing at the Florida Laboratory.

TESTING

1. All testing is in accordance with the current requirements of ASTM C 1240 and the appropriate referenced standards.
2. Daily samples are combined into weekly samples representing each 100 Mg (110 ton) of silica fume. They are analyzed for chemistry of the elemental oxides including silicon dioxide, sulfur trioxide, sodium oxide, potassium oxide, etc. along with Moisture Content, Loss on Ignition (LOI), Oversize (percent retained on No. 325 sieve), Specific Gravity (SG), and Bulk Density (BD) at each plant laboratory.
3. Four (4) weekly composite samples representing each 100 Mg (110 ton) of silica fume are combined into a 400 Mg (440 ton) composite sample (typically monthly) and sent to the Norchem Florida laboratory for additional chemistry and physical testing.
4. At the end of three (3) months or collection of samples representing 3200 Mg (3520 ton), a composite sample is tested in the Norchem Florida laboratory per ASTM C-1240 for Pozzolanic Strength Activity Index (PSAI), Specific Surface Area (SSA), and any optional testing specifically required.
5. Norchem SOP procedures call for laser scattering particle size distribution analysis and four-pin resistivity cup and RCP testing on all samples and any samples tested for Pozzolanic Strength Activity Index (PSAI).

EQUIPMENT CALIBRATION

1. Calibration of the test equipment is conducted as necessary and in accordance with applicable requirements of ISO 9001:2008 – ANSI/ISO/ASQ Q9001-2008, and CCRL laboratories.
2. Forney Break Machine calibrations are conducted annually by a certified outside calibration specialist.
3. The Nitrogen Adsorption Specific Surface Area (SSA) analyzer, Light Scattering Laser Particle Size Analyzer (PSA), X-Ray Fluorescence Spectrometer, ovens, and constant temperature water baths, are calibrated with each use.
4. The flow table, pH meter and various analytical balances are calibrated annually.
5. Wet wash sieves are inspected with each use (for wear) and calibrated bi-annually. If excessive wear is evident they are replaced or disposed of.

REPORTING

1. Chemical & Physical Analysis (CPA) reporting is weekly from each plant by the Chief Chemist to Norchem Florida Technical Center.
2. Data is compiled and maintained in a central database for retrieval as needed. These records are kept for a minimum of five (5) years. However, most test data is maintained indefinitely.
3. Information regarding concrete plants or customers receiving shipments of silica fume from Norchem, Inc. are kept and maintained for record purposes at least 5 years. This information is typically found in the shipment Bills of Lading (BL) that can be made available from Norchem, Inc. or the appropriate distributor.

SHIPMENT CERTIFICATION

1. Each shipment of product can be certified for quality by use of a Chemical and Physical Analysis (CPA) report. These reports are based on the reporting requirements of ASTM C 1240 and some additional customer requested data. The certificate lists: the Customer name, Delivery address, Date, Quantity, Shipping number, Plant of Manufacture, Product type and Lot Number. The test data includes SiO₂ content, %, SO₃, %, CL, %, Total Alkalies, as equivalent Na₂O, %, Moisture Content, %, Loss on Ignition, %, pH, and the Physical Test Data for Oversize, % retained, Density (specific gravity), Mg/m³, Bulk Density, lb./ft³ & kg/m³, Specific Surface Area m²/g, and the Accelerated Pozzolanic Strength Activity Index.
2. The CPA is available via email in electronic form, US Mail or by facsimile.
3. A Project-specific Statement of Compliance (in letter form) is available upon request.
4. DOT-specific statements of compliance are either found on the CPA document or on an accompanying cover letter/facsimile cover sheet for the specific shipment.
5. Automatic load certifications for every shipment can be arranged by notifying Norchem's customer service department.

LOAD TRACEABILITY

1. Norchem provides complete traceability for all shipments of silica fume, from the date of production to the final shipment destination. The system uses a combination of the shipment Bills of Lading and the material Lot Numbers to recognize the loads.
2. The Bills of Lading produced in triplicate identify the plant or warehouse of origin, shipping date, customer, carrier, shipment number, delivery address, product number,

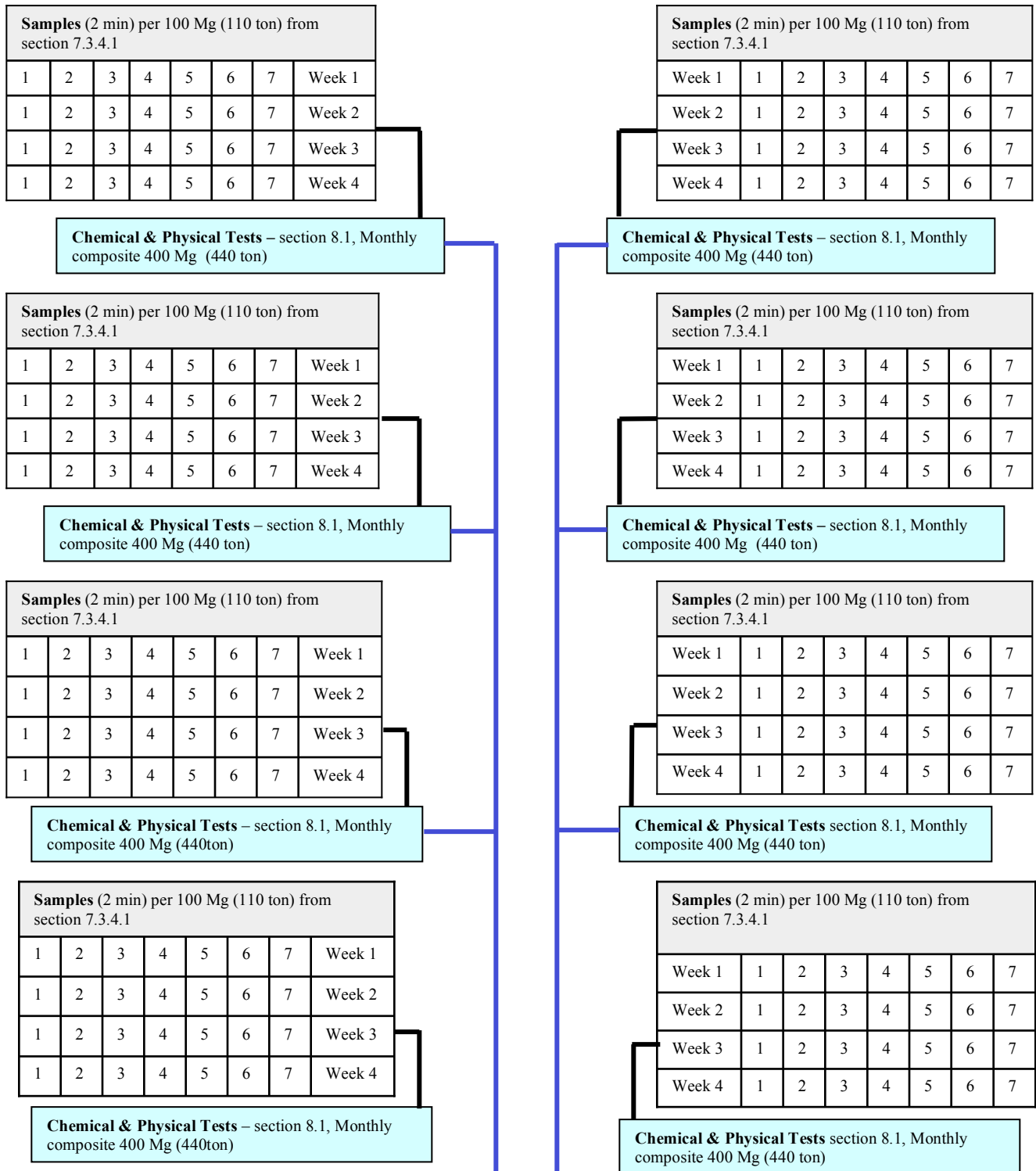
quantity, material classification number, product type, shipper's signature and normally the lot number. A copy of the BL is given to the carrier and said document is carried with the load from plant or warehouse to the final destination where it is handed to the customer.

3. A copy is maintained at the plant or warehouse, and in both the Norchem New York and Florida Offices.
4. The internal lot number system uses a unique alphanumeric identification code to distinguish the material's makeup. This coding gives Quality Control the ability to quickly identify the materials and match them to production and test data.
5. Lot numbers can be found in several places depending on the shipment type: bulk, bag, or supersack. Aside from the Bill of Lading, lot numbers are clearly visible on palletized package products (detail can be found in SOP package procedures).

PROTECTION AGAINST CONTAMINATION

1. Contamination of the silica fume at the plant locations is non-existent, due to the fact that the plant collection systems are a closed circuit system completely separate from the furnaces. The only possible contamination would be the material defined by ASTM as oversize (un-burnt coal, wood chips, metal fragments) that is filtered out during the silica fume processing in the bag house and cyclone systems.
2. Non-specification silica fume is extremely rare and only produced during furnace maintenance start up or shut down. SOP procedures dictate that when this event is scheduled the non-specification fume is collected in an empty silo and disposed of according to plant policies.
3. Damaged silica fume products typically only occur after production or during storage. These materials are red tagged, physically painted with a red strip and set aside for disposal according to local, state, and federal requirements.
4. Materials identified as having a potential problem (by customers or the warehouses) are segregated, sampled and tested to determine if the materials still have a value. When possible the materials are recycled, otherwise they are disposed of in accordance with local regulations.

6 SAMPLING DIAGRAM PER ASTM C 1240



7 PRODUCT PACKAGING AND WAREHOUSE PREPARATION PROCEDURES

The following Standard Operating Procedure (SOP) procedures apply to all plants and facilities packaging silica fume products.

1. Silica Fume used for packaging must be run through the “safety” screen systems prior to bagging.
2. Confirm bagging machine is set for proper bag weight and check calibration status of load scale prior to starting.
3. Fill bags or supersacks to correct listed weight. The bag tolerance for paper bags is zero underweight and + 1 lb. over weight. Supersack is zero underweight and + 5lb. over weight. Check random bags for correct weight using a platform scale prior to stacking on a pallet. Dispose of any bags outside the tolerance limits.
4. All stored/warehoused materials are to be managed on a first in first out basis. Defined as: Oldest material in storage (determined by lot number) is required to ship out first.
5. For export shipments, Heat Treated (HT) pallets are required, in compliance with the International Pallet Convention (IPPA) requirements.
6. Moisture protection layer must be placed on the pallet, consisting of cardboard liner prior to loading.
7. Bags must be clean and free of excess material or debris before stacking on the pallet and blown clean prior to stretch wrapping.
8. Lot Number identification must be clearly identified on a minimum two sides of each pallet and on each supersack. Typically a white sheet of 8.5” x 11” paper (appendix 2 section 14.3) is used listing the following: Norchem, Inc. or Distributor’s name, Product type, Bag or supersack weight and Lot Number. For paper bags the Lot Number must also be stenciled on a minimum of one bag per side of each pallet.
9. Each pallet must be covered with a plastic bag liner and stretch-wrapped before placing in storage or staged for shipment to customers.
10. Lot number identification and number of pallets of each lot must be listed on the Bill of Lading (BL) at the time of shipment.
11. Warehouse supervisor will sign the Bill of Lading to confirm load and shipping details are correct.
12. Each packaging location will submit a weekly shipment report to the appropriate personnel.

8 BULK TANKER LOADING PROCEDURES

The following Standard Operating Procedures apply to all plants and facilities prior to loading Bulk materials. Note: Norchem Tech Bulletin # 112 Silo Configuration, Storage and Material Handling is included as an Appendix to this manual.

1. Record truck (equipment) tare weight prior to loading.
2. Bulk truck hatches must be opened for visual inspection of material compartments. Make sure compartments are completely empty and dry. Contamination from small amounts of fly ash, lime, cement, or other materials can cause adverse chemical reactions. In addition, small quantities of residual moisture in the truck can cause stickiness, lumps and balling during delivery pump-off.
3. Use of the silo/compartment scalping screen (1/2" maximum opening) is mandatory on all bulk loads.
4. When loading bulk tankers from supersacks, the supersacks must be emptied through a scalping screen (1/2" maximum opening).
5. Supersacks taken from inventory for bulk tanker loading must be cleared by the Quality Control Department prior to use. In addition the materials must be tested for moisture and oversize by use of the +325 (45- μ m) sieve, prior to loading.
6. A shipment record including the customer name, shipment number, test results and lot number of each supersack used to make up bulk load must be maintained and forwarded to the Norchem Florida Quality Control Department each week.
7. 2kg grab samples must be taken from each lot number used to make up the shipment. Combine the individual lot number samples into a single composite and send to the Norchem QA for the load-specific analysis.
8. Send truck to the scales for final weighing and official load documentation.
9. Obtain Trucker signature as certification that the tanker was empty and scalped as loaded.



9 NORCHEM DISTRIBUTION NETWORK

Norchem produces, loads and packages silica fume for many companies worldwide. The primary delivery systems are by bulk tanker, supersacks and paper bags. Many of our distributors request the silica fume product be private labeled or packaged into bags carrying their product name for delivery throughout their system of customers. Below is a listing of our North American Concrete Admixture Distributors and their product names.

<u>Distributor</u>	<u>Product Name</u>
BASF Corporation	MasterLife SF 100
Euclid Chemical Company	Eucon MSA
GCP Applied Technologies (WR Grace)	Force 10,000 D
Sika Corporation	Sikacrete 950 DP
Acme	Silica Fume
Advanced Cement Technology	Silica Fume
Chryso	Chryso DSF
Fritz Pak	Silica Fume
General Resource Technology - Mapei	Silica Fume
Hydration Kontrol	Silica Fume
Peregrine Chemical	Silica Fume
Premiere Concrete Admixture	Silica Fume
RussTech	Silica Fume

If more information regarding these, or other, distributors is required please our sales office at (631) 724-8639, or send an email to info@norchem.com.

10 CONTACT INFORMATION

Sales, Administration & Customer Service

960 Wheeler Rd. # 5537
Hauppauge, NY 11788-0132
Tel. # (631) 724-8639
Fax # (888) 617-8520

QA & Laboratory

985 Seaway Drive, Suite A
Fort Pierce, FL 34949
Tel. # (772) 468-6110
Fax # (772) 468-8702

Silica Fume - Quality Assurance Manual 2016 Appendices

12 APPENDIX 1 – ISO 9000 CERTIFICATES

CERTIFICATE OF REGISTRATION



Having been audited in accordance with requirements of

ISO 9001:2008 – ANSI/ISO/ASQ Q9001-2008

SRI Quality System Registrar, 300 Northpointe Circle, Seven Fields, Pennsylvania, 16046, USA, hereby grants to:

Globe Metallurgical, Inc.

Registration of the management system at its location:

**County Road 32
Beverly, Ohio, 45715, USA**

The conditions for maintaining this certificate of registration are set forth in the SRI registration agreements R20.3 and R20.4. Further clarifications regarding the scope of this certificate and the applicability of ISO 9001:2008 requirements may be obtained by consulting the organization.


Scope of ISO 9001:2008 registration: "Manufacture and distribution of electrometallurgical products and by-products."

Exclusions: Design and Development; Service Provision; Validation of Processes for Production and Service Provision

Initial SRI registration date: March 21, 2005

Current registration period: March 18, 2014 through March 17, 2017

Signed for SRI:


Christopher H. Lake, President & COO

Certificate Date: March 18, 2014
Certificate Number: 012489
Registration Number: 0377-01



C E R T I F I C A T E O F R E G I S T R A T I O N



Having been audited in accordance with requirements of

ISO 9001:2008 – ANSI/ISO/ASQ Q9001-2008

SRI Quality System Registrar, 300 Northpointe Circle, Seven Fields, Pennsylvania, 16046, USA, hereby grants to:

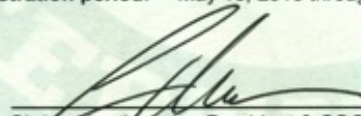
WVA Manufacturing, LLC

Registration of the management system at its location:

**US Route 60 E
 Alloy, West Virginia, 25002, USA**

The conditions for maintaining this certificate of registration are set forth in the SRI registration agreements R20.3 and R20.4. Further clarifications regarding the scope of this certificate and the applicability of ISO 9001:2008 requirements may be obtained by consulting the organization.

- Scope of ISO 9001:2008 registration:** "Manufacture and distribution of electrometallurgical products and by-products."
- Exclusions:** Design and Development; Service Provision; Validation of Processes for Production and Service Provision
- Initial SRI Registration date:** May 18, 2006
- Current registration period:** May 16, 2015 through May 15, 2018

Signed for SRI: 
 Christopher H. Lake, President & COO

Release Date: May 16, 2015
 Certificate Number: 015411
 Registration Number: 0377-05



CERTIFICATE OF REGISTRATION



Having been audited in accordance with requirements of

ISO 9001:2008 – ANSI/ISO/ASQ Q9001-2008

SRI Quality System Registrar, 300 Northpointe Circle, Seven Fields, Pennsylvania, USA, hereby grants to:

Globe Metallurgical, Inc.

Registration of the management system at its location:

**3807 Highland Avenue
Niagara Falls, New York, USA**

The conditions for maintaining this certificate of registration are set forth in the SRI registration agreements R20.3 and R20.4. Further clarifications regarding the scope of this certificate and the applicability of ISO 9001:2008 requirements may be obtained by consulting the organization.

Scope of ISO 9001:2008 registration: "Manufacture and distribution of electrometallurgical products and by-products."

Exclusions: Design and Development; Service Provision; Validation of Processes for Production and Service Provision

Initial SRI registration date: September 10, 2010

Current registration period: September 9, 2013 through September 8, 2016

Signed for SRI:


Christopher H. Lako, President & COO

Certificate Date: September 9, 2013
Certificate Number: 012036
Registration Number: 0377-04



CERTIFICATE OF REGISTRATION



Having been audited in accordance with requirements of

ISO 9001:2008 – ANSI/ISO/ASQ Q9001-2008

SRI Quality System Registrar, 300 Northpointe Circle, Seven Fields, Pennsylvania, 16046, USA, hereby grants to:

Globe Metallurgical, Inc.

Registration of the management system at its location:

**2401 Old Montgomery Highway
Selma, Alabama, 36701, USA**

The conditions for maintaining this certificate of registration are set forth in the SRI registration agreements R20.3 and R20.4. Further clarifications regarding the scope of this certificate and the applicability of ISO 9001:2008 requirements may be obtained by consulting the organization.

Scope of ISO 9001:2008 registration: "Manufacture and distribution of electrometallurgical products and by-products."
Exclusions: Design and Development; Service Provision; Validation of Processes for Production and Service Provision
Initial SRI Registration date: June 18, 2011
Current registration period: June 15, 2014 through June 14, 2017

Signed for SRI:


 Christopher H. Lake, President & COO

Release Date: June 15, 2014
 Certificate Number: 012697
 Registration Number: 0377-02



Certificate of Registration



This is to certify that the quality management system of

Quebec Silicon LP

Main Site: 6500 Yvon-Trudeau Street, Bécancour, Québec, G9H 2V8, Canada

has been assessed and registered by Intertek as conforming to the requirements of

ISO 9001:2008

The quality management system is applicable to

The manufacturing and sale of Silicon, chemical and electronic grades, Si96%, Si98% and Si +99%, lumpy or granulated. Silicon metal, aluminum alloying grades. FeSi all grades between 75% and 96% Si. Silica fumes and slag in a variety of specs and physical shapes.



Certificate Number: QMS-1099-1
Initial Certification Date: 30 June 1995
Certificate Issue Date: 10 January 2013
Certificate Expiry Date: 17 January 2016



Calm Moldovan, President, Business Assurance
Intertek Testing Services NA Ltd. - Lachine, QC, Canada



In the issuance of this certificate, Intertek assumes no liability to any party other than to the Client, and then only in accordance with the agreed upon Certification Agreement. The certificate's validity is subject to the organization maintaining their system in accordance with Intertek's requirements for systems certification. Validity may be confirmed via email at certificates.validation@intertek.com or by scanning the code to the right with a smartphone.

The certificate remains the property of Intertek, to whom it must be returned upon request.

CT-80 9001-2008-SCC-EN LT-L-04-Jan-12



13 APPENDIX 2 - SAMPLE FORMS

SILICA FUME CHEMICAL & PHYSICAL ANALYSIS REPORT

All Testing per ASTM C-1240


CUSTOMER: Alloy Plant 14V (Densified)

DESTINATION: Nov. to Dec. 2015 Average

DATE: 1/29/16 **QUANTITY:** N/A **SHIPPING #:** N/A

N. I. #: N/A **LOT #:** N/A

BULK: **SUPERSACK:** **BAG:**

<u>CHEMICAL TESTS</u>	<u>ANALYSIS</u>
SiO ₂	<u>95.84 %</u>
SO ₃	<u>0.18 %</u>
CL	<u>0.07 %</u>
Total Alkali	<u>0.34 %</u>
Moisture Content	<u>0.09 %</u>
Loss on Ignition	<u>2.68 %</u>
pH	<u>7.28</u>
<u>PHYSICAL TESTS</u>	
Oversize - % retained on 45 µm sieve (wet sieved)	<u>1.62 %</u>
Density - (specific gravity)	<u>2.24</u>
Bulk Density - (per ASTM) <u>763.99 kg/m³</u>	<u>47.69 lb/ft³</u>
Specific Surface Area (by BET)	<u>22.03 m²/g</u>
Accelerated Pozzolanic Activity Index - with Portland Cement	<u>127.88 %</u>
<i>Prepared by Norchem Inc.  Department</i>	
Form NL 101E	



www.norchem.com

985 SEAWAY DRIVE, STE A, FORT PIERCE, FL. 34949-2744
TEL. (772) 468-6110 · FAX (772) 468-8702

P.O. BOX5537, HAUPPAUGE, N.Y. 11788-0132
TEL. (631) 724-8639 · FAX (631) 724-8645



NORCHEM, INC.

www.norchem.com

985 Seaway Drive, Suite A • Ft. Pierce, Florida 34949-2744
Tel (772) 468-6110 • Fax (772) 468-8702

Mr. Ken Phalen
Advanced Pozzolanic Technologies
15 Rowen Street
Springfield, OH 45501

January 1st, 2016

SAMPLE

Sent via email

Re: **Statement of Compliance**

Mr. Phalen,

This letter is to certify that the Norchem, Inc. silica fume sold to Advanced Cement Technologies meets the requirements of the following Standard Specifications:

1. ASTM C 1240-14, Standard Specification for Silica Fume Used in Cementitious Mixtures.
2. AASHTO M307-13, Standard Specification for Use of Silica Fume in Cementitious Mixtures.
3. CSA A3000-13, Cementitious Materials Compendium.

Norchem supplies a Certificate of Compliance known as a Norchem Chemical & Physical Analysis (CPA) via email for each load.

I trust this information will satisfy your requirements. If you have questions, feel free to contact me at (772) 468-6110.

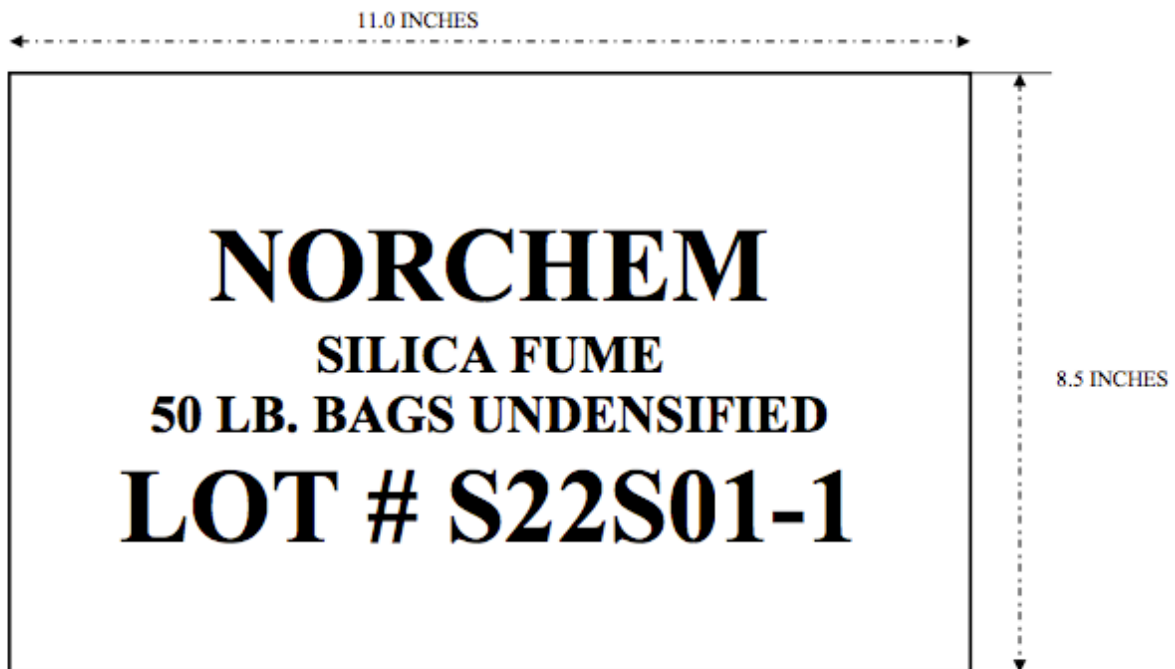
Regards

Tricia Busby
QA Administrator

PACKAGE LOT IDENTIFICATION TAG (SAMPLE)

Lot number identification must be clearly identified on a minimum two sides of each pallet and on each supersack (Section 7.0 Product Packaging and Warehouse Preparation Procedures on page 15). The label below is typically what is used for all packaged products including paper bags of all sizes (25 lb., 50lb.) and supersacks.

In the case of private labeled packaging the specific distributor's name and brand will replace the Norchem name.



14 APPENDIX 3 - TECH BULLETIN, TDS, SDS AND SRM

Technical BULLETIN



112

June 2016

Revision 9

Silo Configuration, Storage and Material Handling of Dry Densified Silica Fume

Dry densified silica fume is a higher density (42 to 48 lbs/ft³) version of undensified silica fume with all the same technical performance characteristics. The higher density allows larger payloads for economical transportation in bulk tankers. Dry densified silica fume is virtually dustless, lump less, flows readily and can be stored in ordinary cement silos and transported in bulk tankers. It can be moved into storage silos by pneumatic lines or bucket elevator and has material handling qualities similar to other dry products like cement or fly-ash that are used in concrete. The main difference, between cement and silica fume material handling is pumpability because silica fume is over one hundred times smaller than cement particles and can not be pumped-off as fast as cement. However, with proper pumping procedures, properly configured clean silos and changes to up-pipes, bulk silica fume truckloads can be pumped off in 1.5 to 2 hours.

1. PNEUMATIC BULK TANKERS

Bulk dry densified silica fume is typically delivered in bulk tankers, 1,400 cubic feet in size. These larger bulk tankers allow delivery of truck load sizes up to 46,000 pounds (23 ton), at typical densified silica fume bulk densities.

2. STORAGE SILOS

Storage silo capacity should be optimally at least 2,800 cubic feet. This allows for the storage of two bulk loads of densified silica fume, and minimizes delivery-scheduling problems. It should be noted that silica fume's typical bulk density of about (45 lb/ft³) is approximately 50% of cement bulk density (94 lb/ft³). Thus, the silica fume capacity of a cement storage silo is much less than that of the cement storage capacity of the same silo. For example, a cement storage silo that holds two loads of cement will only hold one load of silica fume.

3. DUST COLLECTION AND SILO VENTING

The silo should be vented and have an adequate dust collection system (bag house) of at least 150 square feet. The dust collection system should be clean before delivery, so that the pneumatic bulk truck trailer pumps into minimum back pressure.

Bag house media is typically specified by the bag house manufacturer, however good results have been reported using Spun Bonded Polyester Filter Media and 100% Polyester Fiber Filters. In any case the filter media should handle an average particle size of less than 1µm.

4. GROUNDING STRAPS

Every silo should have provisions on the silo for connecting a heavy grounding strap to the bulk trailers. Use of the grounding strap will minimize static charge that

could cause problems and contribute to long pump off times.

5. UP-PIPES

Since large volume (area) and low pressure are the keys to moving dry densified silica fume. The up-pipe or (input) pipe material as well as configuration plays a large role in discharging dry densified silica fume into the silo. The optimum input pipe for fast bulk tank truck discharge is a six (6) inch diameter rubber hose. The reason for the large size is obvious when comparing the cross sectional hose areas as below:

2.5" hose =	4.91 in ²
4.0" hose =	12.57 in ²
5.0" hose =	19.63 in ²
6.0" hose =	28.27 in ²

The rubber up-pipe should have smooth sides and should not be the corrugated or ridged type. We suggest fastening the hose at intervals of approximately every 12 to

15 feet, to the silo, so that the pipe can vibrate and self clean. All pipe bend radii, particularly at the top input to the silo should have a smooth large (horseshoe) radius of at minimum 5 feet.

Horizontal runs of input line and right angle bends (90°) should be avoided, especially in steel pipe, as they will drastically increase pumping times and cause clogging.

If the existing input pipe is steel (4 to 6 inch diameter), with a restricted input radius at the top of the silo, a vibrator should be attached to the top bend radius to prevent build up and clogging at the angled input. The vibrator should be cycled on and off while the bulk tank truck is emptying into the silo, with vibration time duration depending on the size of the pipe, bend radius and its tendency to clog. The vibrator will help prevent the silica fume from build up on the sides of the steel pipe. This build up is caused by the

Silo Configuration, Storage and Material Handling of Dry Densified Silica Fume

static charge of the silica fume particles, where in the attraction is lessened by the use of rubber pipe. To minimize this effect on existing steel input pipes, they should be grounded to the bulk tank truck trailer during discharge.

In general, if the input pipe is steel, (4 inch diameter) with sharp right angle bends, horizontal runs and restricted box type entry inside the silo it will probably cause long truck discharge times. If it is planned that an existing silo will be utilized for silica fume storage, on a long-term basis, the up-pipe should be changed to rubber.

6. SILO TOP UP-PIPE CONNECTIONS

The pipe should be fastened to the horizontal top portion of the silo, as close to the mid point as possible. This connection point can be accomplished with a pipe flange and metal clamps, so that the silica fume drops directly down into the silo without obstruction (Fig 1). However a more detailed layout is offered on the Norchem Silica Fume Up-pipe Design & Silo Modifications page.

7. CHARGING THE WEIGH HOPPER

Densified silica fume does not pump vertically as well as cement, but flows downward more freely and at a faster rate. Therefore controlling the feed rate becomes an important factor. To assist silica fume discharge from the silo to the weigh hopper the storage silo should have aerators in the cone discharge section. Running the aerators at least 15 minutes prior to discharging materials will fluff up the silica fume in the silo making discharge easier.

Dry densified silica fume flows easily in a gravity or air slide connection to the weigh hopper. On the other hand when the silo discharges into a cement screw configuration several interrelated items should be considered. The first item is the length of the screw shorter is always better. Shorter distances generally clog less and are much easier to clean. If it is impossible to cut the distance down, than distance and feed rate will most likely dictate the diameter necessary. For longer distances (over 6ft) a (10 to 14") screw diameter

size, is recommended. Since this size is less restrictive and not as likely to pack.

Lastly look at the feed rates both to the screw and the rate required at the weigh hopper. Dry densified silica fume will flow like water unless controlled. The easiest method to restrict the flow is by adjusting the gate size or opening as needed. Another method employs the use of a rotary feeder at the bottom of the silo before the screw conveyor. The rotary feeder can be adjusted to feed the screw at a required the rate.

8. OPTIONAL SILO ACCESSORIES

The items listed in this section are not required for plant operation but have been found useful in our configurations.

Level indicators take the guesswork out of how much material is in the silo at any given time. Additionally they can help prevent a blow out or overfill situation when off loading a delivery.

Aeration timers will allow the plant operator flexibility in

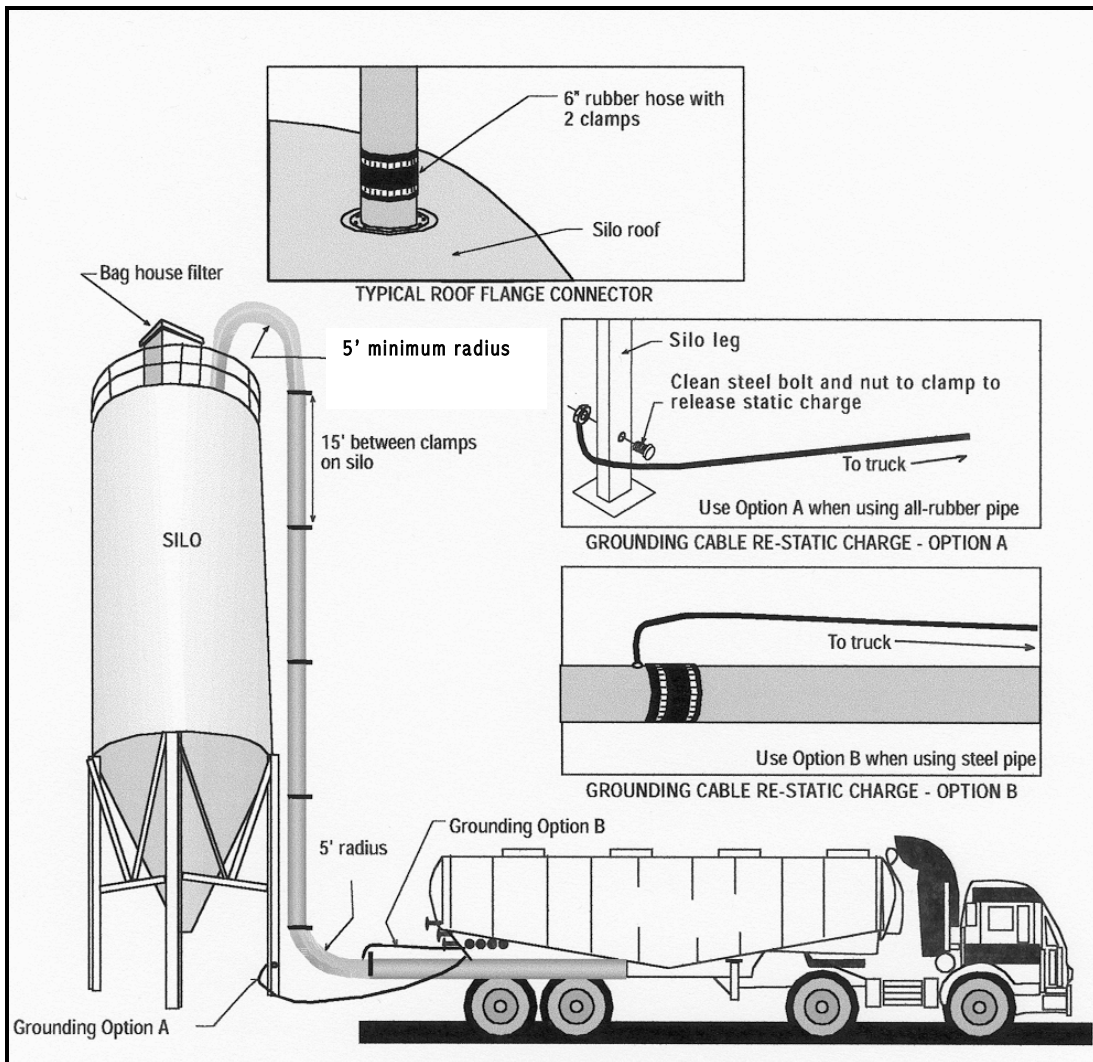
when and how long the silica fume is fluffed prior to discharging.

9. CHECK LIST - FOR OPTIMUM BULK TRUCK TRAILER PUMP OFF

- 6-inch rubber hose with no horizontal runs.
- Clean bag house, adequately vented.
- Ground truck to silo (re: static discharge)
- If the silo has a steel up-pipe, ground to the product delivery hose clamp.
- Try to maintain a minimum 5-foot radius on all directional changes
- Rubber up-pipe fastened to silo approximately every 12 to 15 feet to allow rubber pipe vibration for self-cleaning during pump off.

Silo Configuration, Storage and Material Handling of Dry Densified Silica Fume

Figure 1
 Optimum configuration for storage and discharge of Dry Densified Silica Fume



Silica Fume Up-pipe Design & Silo Modifications

- 1. Silo configuration must consider silo capacity including dust collection & vent system.**
 - 1.1 Silica Fume avg. bulk density = 45 lb/ft³, with fluctuations ranging from 36 to 44 lb/ft³.
 - 1.2 Bulk density variations can occur during material handling from air pressure (fluffing).
 - 1.3 Silo size minimum is 1650 ft³ to store (1) full bulk tanker load 45,000 lb. allowing for air
 - 1.4 Displacement and proper operation of dust collection equipment.
- 2. Expedited truck blow-off (approx. 90 minutes) is attainable with this equipment:**
 - 2.1 Use (1) single length of 6-inch nominal (152.4 mm.) I.D. hose (when possible).
 - 2.2 Hose connections made with Aluminum – Dixon “Andrews”/”Boss-Lock” Cam & Groove Adapters (as needed).
 - 2.3 Delivery truck direct connect requires (1) Dixon “Andrews” Reducing Coupler Adapter 6” x 4” model # 6040-DA.
 - 2.4 Typically delivery trucks carry (1) piece of 4-inch rubber hose (double ended female).
 - 2.5 Minimize connections each connection represents a potential build-up location.
- 3. Hose Specifications:**
 - 3.1 Hose Name: Boston Otter Water Suction & Discharge hose.
 - 3.2 Hose Size: 6-inch nominal I.D.
 - 3.3 Hose Type: EPDM tube, reinforced with 4 spiral 2 ply helical wire, black in color,
 - 3.3.1 Temperature range of -10° F to 180° F.
 - 3.3.2 Working pressure range of 75-125 PSI.
 - 3.4 Hose Dealer:
 - 3.4.1 Nova Rubber Co. Telephone: (304) 485-6491
 - 3.4.2 134 - 7th Ave. Facsimile: (304) 744-0731
 - 3.4.3 Charleston www.NovaRubber.com
 - 3.4.4 W. Virginia 25303
- 4. Starting on the top of the Silo:**
 - 4.1 Use (1) piece of 6 inch (nominal I.D.) prefabricated steel pipe 12” to 24” length is required, pipe fabrication includes:
 - 4.1.1 The pipe end (out of the silo) is male threaded to NPT (National Pipe Tapered)
 - 4.1.2 Threads to accept a Male Adapter x Female NPT cam lock fitting.
 - 4.1.3 The other pipe end (into the silo) is “saw cut” or “as produced”.
 - 4.1.4 Weld a positioning flange on the pipe to allowing for approximately 4” to 12” of the pipe to hang inside the silo and 8” to 12” to protrude out of the silo.
- 5. Cut a hole in the silo top to accommodate the pipe O.D.**
 - 5.1 Insert the prefabricated pipe (from above) cut end 1st, into the hole and weld the positioning flange into place.
 - 5.2 Fabrication of a horseshoe shaped hose support bracket is required see the attached pictures on (sheet 2). Methods used are bent reinforcing rods with welded spacers & support brackets. Or flat stock ¼” steel bent to shape with welded support brackets.
- 6. Silo side brackets are required:**
 - 6.1 Rubber coated hose clamps (similar to a muffler clamp) are mounted to the silo side as stabilization for the hose at approximately 20 to 25 ft intervals.



SILICA FUME -TECHNICAL DATA SHEET

SILICA FUME is a very fine pozzolanic material, composed of amorphous silica produced by electric arc furnaces as a byproduct of the production of elemental silicon or ferro silicon alloys.

SILICA FUME can be used in a variety of applications such as concrete, grouts, mortars, fibre cement products, refractory, oil/gas well cements, ceramics, elastomer, and polymer applications.

SILICA FUME is produced in conformance with the ASTM C-1240 specifications. The quality is controlled and monitored throughout the entire production process to ensure that it meets or exceeds specification requirements.

PROPERTIES	
State	Amorphous - Sub-micron powder
Color	Gray to medium gray powder
Specific Gravity	2.25
Solubility	Insoluble
Bulk Density - Densified	43 to 48 lb/ft ³ (690 to 770 kg/m ³)
Bulk Density - Undensified "as produced"	14 to 20 lb/ft ³ (225 to 320 kg/m ³)

SPECIFICATIONS		
Chemical Requirements	ASTM	Typical
Silicon Dioxide (SiO ₂) %	85.0 % Minimum	93.47 %
Moisture Content %	3.0 % Maximum	0.25 %
Loss on Ignition (LOI) %	6.0 % Maximum	3.55 %
Physical Requirements	ASTM	Typical
Oversize percent retained on 45-µm (325 sieve)	10.0 % Maximum	1.73 %
Accelerated Pozzolanic Strength Activity Index with Portland cement (7 day)	105.0 % Minimum	142 %
Specific Surface	15 m ² /g Minimum	22.24 m ² /g

MECHANISM OF SILICA FUME'S POZZOLANIC REACTION IN A CEMENTITIOUS SYSTEM

SILICA FUME in contact with water goes into solution within an hour. The silica in solution forms an amorphous silica rich, Ca poor, gel on the surface of the silica fume particles and agglomerates. After time the silica rich, Ca poor, coating dissolves and the agglomerates of silica fume reacts with free lime (CaOH₂) to form calcium silicate hydrates (CSH). This reaction is called the *pozzolanic reaction*.

SILICA FUME – TECHNICAL DATA SHEET

PACKAGING, STORAGE AND HANDLING

PRODUCT FORM	PACKAGING	WEIGHT
Silica Fume – Densified	Bulk Truckload	46,000 lbs. max
	Super Sack	2,200 lbs.
	Paper Bag	50 lbs.
	Paper Bag	25 lbs.
Silica Fume - Undensified	Super Sack	1,000 lbs.
	Paper Bag	50 lbs.

STORAGE

SILICA FUME should be kept dry, out of weather and the elements.

SAFETY AND HANDLING PRECAUTIONS

SILICA FUME is generally considered a nuisance dust. Use and handling of silica fume does not represent a health risk when normal safety rules are observed. Direct contact may cause irritation of eyes. Prolonged contact may cause skin irritation. Inhalation may cause respiratory irritation resulting in coughing and shortness of breath. This product may be harmful if swallowed. Do not get in eyes and avoid prolonged skin contact. Do not take internally. Wash thoroughly with water after handling. For more detail, see our **SDS**.

WARRANTY STATEMENT

The information given here is based on our best knowledge, and we believe it to be true and accurate. Norchem assumes no responsibility for the use of these statements, recommendations or suggestions, nor are they intended as a recommendation for any use, which would infringe any patent or copyright.



Norchem, Inc – New York Office
 960 Wheeler Road, # 5537
 Hauppauge, NY 11788
 Tel: 631-724-8639



Where Ideas Are Concrete!

www.norchem.com

Norchem, Inc – Florida Office
 985 Seaway Drive
 Fort Pierce, FL 34949
 Tel: 772-468-6110



NORCHEM SAFETY DATA SHEET

1. Substance and Source Identification

Product Name: Norchem Silica Fume (Dry Powder)

Product Uses or Application: Cementitious Mixtures

Company Information:

Norchem, Inc.	Globe Metallurgical Inc.
985 Seaway Drive	P.O. Box 157, County Rd. 32
Fort Pierce, FL 34949	Beverly, OH, 45715
Telephone: 772-468-6110	740-984-2361
Fax: 772-468-8702	
Website: http:// www.norchem.com	

Emergency Telephone: CHEMTREC: 1-800-424-9300

2. Hazards Identification

Classification: Does not meet the criteria of the UN Globally Harmonized System (GHS) for hazard classification.

Physical Hazard: Not classified
Health Hazard: Not classified

Label Elements:

Symbol: No Symbol

Signal Word: No Signal Word

Hazard Statement (s): Not applicable.

Precautionary Statement(s) Not applicable.

3. Composition/Information on Ingredients

Substance: Silica Fume
Synonyms: Amorphous Silica, Silicon Dioxide, Microsilica, Corrochem, Micropoz.
CAS No: 69012-64--2
EINECS No: 273-761-1

Silica Fume may contain trace amounts (<0.05%) of crystalline silica (quartz), which has been shown to cause silicosis, and has been identified by IARC and NTP as a possible human carcinogen.

SAFETY DATA SHEET

4. First Aid Measures

Inhalation:	If inhaled to excess remove exposed person to fresh air. If necessary, seek medical attention.
Skin Contact:	Wash skin with mild soap and water.
Eye Contact:	Flush eyes with water and carefully rinse under the eyelids. If necessary, seek medical attention
Ingestion:	Obtain first aid or medical assistance immediately.
Most Important Symptoms/Effects, Acute and Delayed:	Dust may result in irritation.

5. Fire Fighting Measures

Fire and Explosion Hazards:	Silica fume is non-combustible and presents no danger of explosion		
Extinguishing Media:	N/A, Use extinguishing agents appropriate for surrounding fire		
Protective Equipment for Fire Fighters:	Wear NIOSH approved self-contained breathing apparatus (SCBA)		
NFPA Ratings:	0 = Minimal: 1 = Slight: 2 = Moderate: 3 = Serious: 4 = Severe		
	Health = 0	Fire = 0	Reactivity = 0

6. Accidental Release Measures

Personal Precautions, Protective Equipment and Emergency Procedures:	Use 42 CFR 84 NIOSH/MSHA approved respirators when airborne concentrations equal or exceed the Permissible Exposure Limit.
Methods and Materials for Containment and Cleanup:	Collect using methods that minimize creation of airborne dust. High efficiency vacuum cleaning is recommended to recover spilled material. Place in suitable container for recycling or disposal. Handle with adequate ventilation for dust.

7. Handling and Storage

Safe Handling Precautions:	Avoid generating dust. Handle with adequate ventilation for dust.
Storage:	Best in closed containers, ambient air temperature, keep dry.

SAFETY DATA SHEET

8. Exposure Controls and Personal Protection

Exposure Limits: No occupational exposure limits have been established for this material.

Components:	CAS Registry #	OSHA-PEL TWA	ACGIH-TWA
Silica, Amorphous Silica Fume	69012-64-2		TLV Withdrawn due to insufficient data
Silica – Crystalline α -Quartz	14808-60-7	0.05 mg/ m ³	0.025 mg/m ³

^R Measured as respirable fraction of the aerosol.

*Total Dust

**Respirable dust

There is no hazard classification for the amount of respirable crystalline silica in the product because when measured by X-Ray diffraction the level is below 0.1%

Engineering Controls: Provide sufficient mechanical (general and/or local exhaust) ventilation to maintain exposures below PELs or TLVs in processing areas.

Personal Protection: In accordance with OSHA 29 CFR 1910.132 subpart I, wear appropriate Personal Protective Equipment (PPE) to minimize exposure to this material.

Respiratory Protection: If workplace conditions warrant a respirator OSHA 29CFR 1910.134 must be followed. Refer to NIOSH 42 CFR 84 for approved respirators when airborne concentrations equal or exceed the Permissible Exposure Limits.

Eye/Face Protection: Wear tightly fitting safety goggles when a risk assessment indicates this is necessary.

Skin/Body Protection: Choose body protection in relation to the task being performed and the risks involved and should be approved by a specialist. Chemical-resistant gloves should be worn at all times when handling chemicals.

9. Physical And Chemical Properties

Physical State:	Amorphous sub-micron powder – dust has a tendency to agglomerate		
Color:	Light to medium gray	Odor:	None
Melting Point:	1200°C - 1300°C*	Specific Gravity:	2.2 – 2.50 Water = 1.0
pH:	6.0 to 9.0	Particle Size:	Approx. 0.4 μ m
Solubility in Water:	Insoluble		
Bulk Density:	Approx. 8 to 48 lb./ft ³ or 128-769 kg/m ³		
Solubility Solvents:	Insoluble to slightly soluble in organic solvents		

SAFETY DATA SHEET

10. Stability and Reactivity

Conditions to avoid:	See Below
Substances to avoid:	Hydrofluoric acid (HF)
Hazardous reactions:	Silica fume is soluble in hydrofluoric acid (HF) and can form toxic gas (SiF ₄).
Decomposition products:	Heating at temperatures above 500°C (930°F) for prolonged time periods will convert amorphous silica to crystalline phases.

11. Toxicological Information

Route of Exposure: Inhalation: X Skin: X Ingestion: N/A Eyes: X

Acute Toxicity:

Inhalation: Airborne Silica Fume dust generated by the use or handling of this product may result in respiratory tract irritation.

Ingestion: Silica Fume dust may irritate and dehydrate throat and mouth.

Eye Contact: Silica Fume dust may cause eye mechanical irritation and dryness.

Skin Contact: Silica Fume dust may cause exposed skin mechanical irritation.

Chronic Effects:

Silica Fume is generally considered a nuisance dust of low toxicity consequently it is considered to pose minimal risk of pulmonary fibrosis (silicosis). Avoid prolonged exposure to silica fume dust concentrations above the recommended exposure limits, unless the protective equipment is used.

It is possible for Silica Fume to contain trace amounts (<0.05%) of crystalline silica, which has been shown to cause silicosis, and has been identified by IARC and NTP as a Positive/Known human carcinogen.

Heating Silica Fume at temperatures above 500°C (930°F) for prolonged time periods will convert amorphous silica to the crystalline phases Cristobalite and Tridymite that may cause silicosis. Increased temperatures will increase the formation rate of these phases.

12. Ecological Information:

No adverse effects are expected. Silica Fume is not considered dangerous to the environment.

13. Disposal Considerations:

Dispose of waste in accordance with applicable Federal, State and Local regulations.

SAFETY DATA SHEET

14. Transport Information:

DOT Not regulated

IATA Not regulated

IMDG Not regulated

Special Precautions for user: None

Transport in bulk according to Annex II of MARPOL 73/78 and the IBC code: Not classified

15. Regulatory Information:

SARA TITLE III: Section 302/304 (extremely hazardous substances) Not regulated

Sections 311/312 Hazardous Categories (40 CFR 370.21)

Acute Health: No
 Chronic Health: No
 Fire: No
 Reactive: No
 Pressure: No

Section 313 This product contains no chemicals subject to the supplier notification requirements. Not regulated

CERCLA: Comprehensive Response Compensation and Liability Act (40 CFR 30.4) Not regulated

TSCA: CAS #69012-64-2 Listed
 There are no TSCA 12(b) chemicals in this product None

CEPA (Canadian DSL): #69012-64-2 is listed on the public Portion of the Domestic Substances List.

WHMIS: Not classified

California Proposition 65: This product may contain trace amounts < 0.05% of crystalline silica a chemical known to the State of California to cause cancer, birth defects or other reproductive harm.

SAFETY DATA SHEET

16. Other Information:

The UN Globally Harmonized System of Classification and Labeling of Chemicals (GHS) safety data sheets (SDS) are required only for substances and mixtures that meet the harmonized criteria for physical, health or environmental hazards. Based on Chapter 1.5.2 this product does not fit into these criteria.

National Fire Protection Association (NFPA) Rating:

HEALTH HAZARD 4 DEADLY 3 EXTREME DANGER 2 HAZARDOUS 1 SLIGHTLY HAZARDOUS 0 NORMAL MATERIAL	FIRE HAZARD FLASH POINT 4 BELOW 73°F 3 BELOW 100°F 2 BELOW 200°F 1 ABOVE 200°F 0 WILL NOT BURN
0	0
SPECIFIC HAZARD OXIDIZER OX ACID ACID ALKALINE ALK CORROSIVE COR USE NO WATER W RADIOACTIVE	INSTABILITY 4 MAY DETONATE 3 SHOCK+HEAT MAY DETONATE 2 VIOLENT CHEM. CHANGE 1 UNSTABLE IF HEATED 0 STABLE
0	0
—	

HAZARD RATING SYSTEM:

Hazardous Material Identification System (HMIS)

HEALTH = 1
FLAMMABILITY = 0
REACTIVITY = 0
PERSONAL PROTECTION = E – (See section 8)

All information, recommendations, and suggestions in this SDS, concerning our products are based on tests and data believed to be reliable, it cannot be guaranteed. Since the actual use by others is beyond our control it is the user's responsibility to determine the safety, toxicity and suitability for their own use of the product described herein.

NIST CERTIFICATE OF ANALYSIS - SRM 2696 - SILICA FUME



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 2696

Silica Fume

This Standard Reference Material (SRM) is intended primarily for use in evaluating chemical and instrumental methods of analysis of silica fume used in conjunction with product specifications [1,2]. A unit of SRM 2696 consists of a single bottle containing approximately 70 g of powder.

Certified Mass Fraction Values: Certified values for constituents in SRM 2696 are listed in Table 1 on a dry-mass basis [3]. Value assignment categories are based on the definitions of terms and modes used at NIST for certification of chemical reference materials [4]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy, in that all known or suspected sources of bias have been investigated or taken into account. A certified value is the present best estimate of the true value based on the results of analyses performed at NIST and collaborating laboratories.

Reference Mass Fraction Values: Reference values for constituents on a dry-mass basis [3], loss on ignition at 750 °C, and specific surface area of SRM 2696 are listed in Table 2. Reference values are noncertified values that represent the best estimates of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [4].

Expiration of Certification: The certification of SRM 2696 is valid, within the measurement uncertainty specified, until **01 May 2023**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Storage and Use"). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Coordination of the technical measurements for certification was accomplished under the direction of J.R. Sieber of the NIST Chemical Sciences Division.

Analytical measurements (including homogeneity testing) for certification of this SRM were performed at NIST by J.R. Sieber and A.F. Marlow of the NIST Chemical Sciences Division.

The material was blended and bottled at NIST under the supervision of M.P. Cronise of the NIST Office of Reference Materials.

Statistical consultation for this SRM was provided by S.D. Leigh of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Robert L. Watters, Jr., Director
Office of Reference Materials

Gaithersburg, MD 20899
Certificate Issue Date: 19 December 2013
Certificate Revision History on Last Page
SRM 2696

Page 1 of 5

INSTRUCTIONS FOR STORAGE AND USE

To relate analytical determinations to the certified values on this Certificate of Analysis, a minimum sample quantity of 500 mg is recommended. The material must be dried at 110 °C for at least 1 h, prior to analysis for chemical constituents. The bottle should be recapped immediately after removal of a sample. Store SRM 2696 in its original container in a cool, dry location, preferably in a desiccator. When a sample is taken from a stored container, it is recommended to determine the loss on ignition and to correct for loss in excess of the reference value, if any. For evaluation of test methods for moisture content and loss on ignition, use only samples taken from a freshly opened pouch.

Warning to Users: In some laboratories, it may be accepted practice to return material to its original container after testing it for Specific Surface Area. Because this SRM is intended for testing of other properties and constituents, the material should be discarded after use. Returning it to the original container may result in contamination of the material and nullification of the certification.

PREPARATION AND ANALYSIS⁽¹⁾

Stability: The silica fume material for SRM 2696 was obtained from Elkem Materials, Inc. from a typical production batch of silica fume. SRM 2696 is considered to be stable during the period of certification when stored in its original bottle in a sealed foil pouch. Once the foil pouch has been opened, the moisture content and loss on ignition may be subject to change (see "Instructions for Storage and Use").

Moisture Content and Loss on Ignition: The reference values were determined by the cooperating laboratories using the methods of test in ASTM C 311-02 [7]. The use of alternative methods may result in different values for these properties. The determinations were made using material from freshly opened units of the SRM.

Specific Surface Area: The reference value for specific surface area provided in Table 2 was determined by the cooperating laboratories using the Brunauer-Emmet-Teller (BET) method of test in ASTM C 1069-86(1997) [8]. The determinations were performed using both single-point and multi-point analyses. The suitability of this SRM for use with different methods and measurement dynamics has not been determined.

Certified Mass Fraction Values: The measurands are the mass fractions of selected constituents in silica fume. The certified values are metrologically traceable to the SI unit of gram per 100 grams. The constituents listed in Table 1 represent consensus assignments of chemical forms by the cement industry and the analytical test methods used by NIST and collaborating laboratories are listed in Table 3.

Table 1. Certified Mass Fraction Values (Dry-Mass basis) for SRM 2696

Constituent	Mass Fraction ^(a) (%)
SiO ₂	95.61 ± 0.37
Al ₂ O ₃	0.2080 ± 0.0071
CaO	0.426 ± 0.016
MgO	0.235 ± 0.024 ^(b)
K ₂ O	0.652 ± 0.028
Mn ₂ O ₃	0.032 ± 0.004 ^(b)
ZnO	0.051 ± 0.005 ^(b)

^(a) The certified values, unless otherwise footnoted, are weighted means of the mass fractions from two to seven analytical methods [10]. The uncertainty listed with each value is an expanded uncertainty about the mean [10,11], with coverage factor, $k = 2$ (approximately 95 % confidence), calculated by combining a between-source variance incorporating inter-method bias with a pooled within-source variance following the ISO/JCGM Guide [5].

^(b) The certified values are unweighted means of the mass fractions from two to five analytical methods [10]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [11] with a pooled, within-method variance following the ISO/JCGM Guide [5].

⁽¹⁾ Certain commercial instruments, materials, or processes are identified in this report to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the instruments, materials, or processes identified are necessarily the best available for the purpose.

Reference Mass Fraction Values: The measurands are the mass fractions of selected constituents in silica fume determined using the test methods listed in Table 3. The measurand for moisture content and loss on ignition is the mass lost at the corresponding temperature using the test methods in ASTM C 311 [7]. The measurand for specific surface area is defined in ASTM method C 1069 [8]. The reference values are metrologically traceable to the SI unit of gram per 100 grams. The constituents listed in Table 2 represent consensus assignments of chemical forms by the cement industry.

Table 2. Reference Values (Dry-Mass Basis) for SRM 2696^(a)

Constituent	Mass Fraction (%)
Na ₂ O	0.129 ± 0.019
P ₂ O ₅	0.0863 ± 0.0057 ^(b)
Fe ₂ O ₃	0.055 ± 0.011
Moisture Content	0.251 ± 0.034
Loss on Ignition at 750 °C	2.11 ± 0.10
	Unit (m ² /g)
Specific Surface Area (S)	22.92 ± 0.36

^(a) The reference values, unless otherwise footnoted, are weighted means from two to seven analytical methods [10]. The uncertainty listed with each value is an expanded uncertainty about the mean [10,11], with coverage factor, $k=2$ (approximately 95 % confidence), calculated by combining a between-source variance incorporating between-method bias with a pooled within-source variance following the ISO/JCGM Guide [5].

^(b) The reference value is an unweighted mean of the mass fractions from three analytical methods [10]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [11] with a pooled, within-method variance following the ISO/JCGM Guide [5].

Table 3. Analytical Methods^(a)

Constituent	Methods
Na ₂ O	XRF, ICP-OES, FAAS, C 114-99
MgO	XRF, ICP-OES, FAAS, C 114-99
Al ₂ O ₃	XRF, ICP-OES, C 114-99
SiO ₂	XRF, C 114-99
P ₂ O ₅	XRF, ICP-OES, C 114-99,
K ₂ O	XRF, ICP-OES, FAAS, C 114-99
CaO	XRF, FAAS, C 114-99
Mn ₂ O ₃	XRF, ICP-OES, C 114-99
Fe ₂ O ₃	XRF, ICP-OES
ZnO	XRF, ICP-OES, C 114-99
Moisture Content	C 311-02
Loss on Ignition at 750 °C	C 311-02
Specific Surface Area (m ² /g)	C 1069-86(1997), Single-point and Multi-point analyses, N ₂ gas

^(a)Key:

- XRF: X-ray fluorescence spectrometry with various sample preparation and calibration procedures performed at NIST and collaborating laboratories
- ICP-OES: Inductively-Coupled Plasma Optical Emission Spectrometry performed at collaborating laboratories
- FAAS: Flame Atomic Absorption Spectrophotometry performed at collaborating laboratories
- C 114-99: Classical (wet) chemical methods in this ASTM standard performed at collaborating laboratories

Note: Although C 114-99 is expected to give reliable results for the analysis of silica fume, its application to this matrix has not been validated by ASTM Committee C01.

The Cooperating Laboratories tested SRM 2696 for oversize in accordance with ASTM C 430. The data generated had a wide variance that did not meet NIST criteria for value assignment. In the minutes of the December 5, 2003, meeting of ASTM International Subcommittee C09.24 Task Group 4 on Silica Fume Specification C 1240, the data variance was attributed to the self-agglomerating characteristics of this material and that the measurement of oversize in silica fume requires a modification of ASTM C 430-96(2003) [12].

Cooperating Laboratories: SRM 2696 was produced in cooperation with the Silica Fume Association as Task 11 under Cooperative Agreement DTFH61-99-X00063 between the Federal Highway Administration and the Silica Fume Association. Participation by collaborating laboratories was coordinated by G.M. Gapinski of Norchem, Inc., Ft. Pierce, Florida. Analytical determinations for certification of this SRM were performed by the following laboratories:

G. Gapinski and R. Plotria; Norchem, Inc.; Ft. Pierce, Florida (USA)
 K. Groff; New York, Dept. Of Transportation, Materials Bureau; Albany, New York (USA)
 J. Beilman; Kansas Dept. of Transportation; Topeka, Kansas (USA)
 S. Carlock; Utah Dept. of Transportation, Materials QA Section; Salt Lake City, Utah (USA)
 D. Broton; Construction Technology Laboratories; Skokie, Illinois (USA)
 R. Martin; Globe Metallurgical, Inc.; Beverly, Ohio (USA)
 K. Kasprzak; Globe Metallurgical, Inc.; Niagara Falls, New York (USA)
 R. Chevrier; CANMET Materials Technology Laboratory; Ottawa, Ontario (Canada)
 R. Hageman; Elkem Materials Inc.; Alloy, West Virginia (USA)
 S. Schlorholtz; Iowa State University, Material Analysis Research Laboratory; Ames, Iowa (USA)
 R. Karuhn; Particle Technology Labs, Inc.; Downers Grove, Illinois (USA)
 M. Thomas; Quantachrome Corp.; Boynton Beach, Florida (USA)
 M. Pohl; Horiba Instruments, Inc.; Irvine, California (USA)
 R. Xu; Beckman Coulter, Inc., Particle Characterization Operation; Miami, Florida (USA)

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Certificate Revision History: 19 December 2013 (Extension of certification period; additional storage information added; editorial changes); 17 September 2007 (Additional information provided about analytical methods; editorial changes); 06 May 2004 (Original certificate date).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet <http://www.nist.gov/srm>.