R&D of a Cyclone Melting Technology for Converting SPL to Useful Glass Fiber Products

FINAL TECHNICAL REPORT

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1.0 EXECUTIVE SUMMARY

Vortec Corporation performed a research and development project for application of Vortec’s Cyclone Melting System CMS® technology to conversion of spent potliner (SPL) waste from primary aluminum smelting operations to useful glass fiber products. The project was sponsored by the United States Department of Energy (DOE), Office of Industrial Technology (OIT), as part of the DOE/Aluminum Industry Partnership Program under Cooperative Agreement No. DE-FC07-97ID13569. Industrial participants in the project were Vortec Corporation; Danieli Corus Technical Services (formerly Hoogovens Technical Services, Inc.); Alcoa, Inc.; and CertainTeed Corporation Insulation Fiberglass Group. Commercial implementation of the technology will eliminate the treatment and landfilling of up to 100,000 metric tons of SPL waste annually and result in significant energy savings in the aluminum and glass fiber manufacturing industries.

The objective of the project was to develop experimental data and evaluate the feasibility of converting SPL to commercial quality glass fiber and fluoride products using Vortec’s CMS® technology. The project was initiated on October 1, 1997, and was completed on January 31, 2002. During the first two budget years, the project focused on pilot-scale production and qualification testing of glass fibers from SPL waste and characterization of the flue gas from the process. The third and fourth budget years concentrated on pilot-scale testing to validate modifications to the process design to achieve flue gas characteristics necessary for economic viability of the process.

Glass fibers meeting CertainTeed’s criteria with respect to size distribution and dissolution performance were produced from vitrified SPL in the pilot-scale process. The average fiber diameter was between 3 and 5 microns, and only a small percentage was less than one micron in diameter. The dissolution rate, analyzed by the New York State College of Ceramics at Alfred University, was similar to that of commercial mineral fiber products. CertainTeed performed standard ASTM testing for mineral fiber loose fill insulation and determined that the fiber produced during the pilot-scale trials from SPL pass the tests.

Pilot-scale SPL vitrification testing demonstrated that HF concentrations in the flue gas of 2%, the project goal established by Alcoa, are achievable. Additionally, a large percentage (averaging about 75%) of the fluoride in the SPL partitioned to HF in the flue gas for conversion to a valuable aluminum fluoride product in the air pollution control system. The aluminum fluoride product can be recycled to the aluminum industry. Feasibility studies indicate that the recycling system should be an economically attractive alternative to the current treatment-and-landfill disposal method of handling SPL waste.

A program plan for demonstration testing on a larger scale at Ormet Primary Aluminum Corporation has been developed with Alcoa. Additionally, a plan for subsequent installation of a 20,000 ton/year or larger SPL vitrification process at an Alcoa facility has been developed. At the time of this report, Alcoa has not made the financial commitment necessary to continue the project.

Descriptions of the work performed, accomplishments, and results of testing and analysis obtained during the first three budget periods were provided in project technical progress reports issued on October 31, 1998, January 28, 2000, and May 16, 2001. This report provides a summary of results of the project for the first three budget periods and details of the work performed, accomplishments, and results of testing and analysis for the fourth budget period from January 31, 2001, to January 31, 2002.
1.1 FIRST BUDGET YEAR PERFORMANCE SUMMARY

During the first 12 months of the project, pilot-scale SPL vitrification testing was performed to produce sufficient quantities of glass product for quality analysis and to characterize the flue gas from the CMS® process. Prior to the testing, a feedstock composition was developed that would produce a glass in the CMS® having physical and thermodynamic characteristics similar to commercial insulating glass fiber products. Based on data developed under previous contractor funded projects, a conventional wheel-type fiberizer, typical of that used for the manufacture of mineral and ceramic fibers, was fabricated and installed in Vortec’s pilot-scale CMS® test facility. The pilot-scale system was also modified to incorporate equipment necessary to cool the flue gas from the CMS® and allow for accurate measurement of fluorides and particulates in the gas. SPL waste from an Alumax primary aluminum smelting plant was shipped to Vortec’s test facility, reduced in size to meet the size specifications for processing in the CMS®, and blended with additives required to meet the feedstock design criteria. A pilot-scale vitrification trial was then performed during which glass fibers were produced and flue gas sampling and analysis were performed. The test was performed under the treatability study exemption from the U. S. Environmental Protection Agency (EPA) and Pennsylvania Department of Environmental Protection (PADEP) Resource Conservation and Recovery Act (RCRA) regulations. The fibers produced were analyzed with respect to size distribution, chemical composition, and temperature-viscosity relationship. The flue gas was sampled and analyzed for fluoride concentrations.

Results of the fiber analysis from the first budget year tests show that some fibers produced were in the diameter range desired (3 to 5 microns) but the majority of the fibers were in the 10 micron to 45 micron diameter range. Vortec discussed the results with CertainTeed, a major fiberglass manufacturer in the United States and a subsidiary of Saint Gobain, the largest European glass manufacturer with extensive experience in glass fiber production with wheel type fiberizers. An agreement was negotiated between CertainTeed and Vortec in which CertainTeed would provide Vortec with proprietary glass composition and property data, provide technical support with respect to fiberizer operation, and analyze the glass and fibers produced during pilot-scale vitrification trials with respect to commercial product quality specifications. In return, CertainTeed will have first priority to the production and marketing of fibers made from vitrified SPL if commercial implementation of the process by Vortec is realized.

CertainTeed analyzed samples of the glass and fibers produced during the initial vitrification trials and Saint Gobain personnel reviewed the resulting chemical composition and viscosity data. Based on the results of their review, Saint Gobain provided viscosity/temperature criteria for fiberizing with a wheel-type fiberizer based on their proprietary data and operating experience. They also provided Vortec with proprietary mineral fiber chemical composition data to use as a guideline in designing a glass, produced from SPL, that would meet commercial fiber product specifications. Additionally, Saint Gobain reviewed the design of Vortec’s pilot-scale fiberizer and made recommendations for minor modifications to improve the fiberizer efficiency. Based on the data provided by CertainTeed and Saint Gobain, Vortec redesigned the glass and feedstock composition and made modifications to the pilot-scale fiberizer for additional pilot-scale trials during the second year of the project.

Flue gas analyses performed during the first year pilot-scale SPL vitrification trials provided data to define the ratio of gaseous-to-solid fluorides in the flue gas from the CMS®. The total particulate concentration in the flue gas was also defined. These data were used as input to the specification of a high temperature gas filter for installation into the pilot-scale test loop during the second project year, followed by additional testing to evaluate the particulate removal efficiency of the filters.
1.2 SECOND BUDGET YEAR PERFORMANCE SUMMARY

Two vitrification trials were performed during the second year of the project. One of the trials produced fiber for commercial qualification testing by CertainTeed and dissolution analysis by the New York State College of Ceramics at Alfred University. The other trial was performed to further characterize the flue gas from the CMS® when operating at the conditions required for recycling SPL into glass fiber products, and to evaluate the performance of high temperature flue gas filters constructed of stainless steel mesh. Data from the trial were used as input in the evaluation of alternative air pollution control (APC) system concepts and the preliminary design of an APC system producing aluminum fluoride.

The feedstock for the fiberizing trial contained glass produced from SPL in a commercial CMS® by Ormet Primary Aluminum Corporation. One of Saint Gobain’s comments based on their project review at the end of the first year was that the flowrate of glass to the fiberizer during the first fiberizing trial was too low. However, the flowrate was the maximum that could be processed in the pilot-scale CMS® from actual SPL waste because of the residence time requirement for effective oxidation of the carbon in the SPL. The required flow rate could be achieved when using the glass from Ormet since the carbon had already been oxidized in the Ormet process. The chemical composition of the glass from Ormet’s process differed somewhat from that required for effective fiberizing. Therefore, additives were blended with the glass prior to the pilot-scale trial that would result in the production of a glass with chemical composition and properties similar to a glass that would be produced directly from SPL for fiberizing. This second fiberizing trial was successful in that it produced sufficient quantities of glass fibers meeting the project physical properties criteria for performance evaluation by CertainTeed. CertainTeed performed standard ASTM testing for mineral fiber loose fill insulation and determined that the fiber produced during the pilot-scale trials from SPL meet the project size criteria and pass the ASTM tests.

The New York State College of Ceramics at Alfred University performed a dissolution analysis of the fibers produced during the pilot-scale trial. Results of the analysis indicate that the dissolution performance of the fibers from SPL glass is similar to the reference commercial mineral fiber product.

The feedstock for the vitrification trial to characterize the flue gas from the CMS® process consisted of SPL waste received from Alumax’s Eastalco primary aluminum smelting facility. The SPL waste was blended with glass forming additives to produce a glass suitable for fiberizing. Feedstock, glass, and flue gas particulate samples taken during the vitrification trials were analyzed for fluoride concentrations. Additionally, a near-infrared tunable diode laser absorption system (LasIR) was used for continuous measurement of HF in the flue gas. X-ray diffraction (XRD) analyses of the flue gas particulate samples were also performed. Results of the analyses indicate that about 29% of the fluoride contained in the SPL waste introduced into the process remained as HF in the flue gas after being cooled to about 700°F in the evaporative cooler. About 16% of the fluoride partitioned to the glass, and about 52% partitioned to fluoride salts, primarily sodium fluoride compounds, removed as solids in the baghouse.

Prior to the performance of the vitrification trial for flue gas characterization during the second budget period, a stainless steel baghouse filter system was installed in the test facility. The system consisted of a stainless steel housing and stainless steel mesh filters. The system is capable of operating at temperatures up to 1,050°F. The flue gas to the baghouse was maintained at 700°F during the vitrification trial, within the temperature range for optimum reaction of HF with aluminum oxide (alumina) to produce aluminum fluoride. Flue gas particulate samples were taken upstream and downstream of the baghouse in accordance with EPA methods to determine the particulate concentrations. The data collected indicate...
that the particulate concentration downstream of the baghouse will likely satisfy the criteria for producing a high purity aluminum fluoride product from the HF in the flue gas. There was no apparent deterioration of the stainless steel mesh filters at the end of the vitrification trial.

Two alternative APC system concepts were defined based on the pilot-scale flue gas characterization data. One uses lime as a reagent in a wet scrubbing process to remove HF from the CMS® flue gas and produce an acid-grade fluorspar (calcium fluoride) product. The other concept uses alumina as a reagent in a dry scrubbing process to produce aluminum fluoride from the HF in the flue gas. Both concepts use an evaporative cooler to reduce the temperature of the flue gas to a level compatible with appropriate flue gas filters. Both concepts also use a baghouse filter system for removal of particulates from the flue gas before it is introduced into the HF scrubber to minimize contamination of the fluoride products. A comparative economic analysis was performed, and the results indicate that the aluminum fluoride producing concept is the most cost effective.

Engineering studies and economic evaluations were performed for a commercial CMS® process for conversion of SPL to glass fiber products under another project supported by Reynold’s Aluminum Corporation, Alcoa, Inc., and CertainTeed Corporation. Data from the DOE supported project were used as input to the evaluations. The baseline plant concept for the evaluations consisted of an air-fired CMS® for SPL vitrification, CertainTeed proprietary glass fiberizing system, and an aluminum fluoride producing APC system based on an HTS proprietary technology. A wet chemical process for converting the sodium fluoride in the particulate from the APC system baghouse into a calcium fluoride product was initially considered, but was determined to be uneconomical. Analytical modeling studies indicated, however, that recycling of the particulate back to the CMS® to increase the HF in the flue gas to the HF scrubber, thus increasing the production of aluminum fluoride, is technically feasible and more economical. Therefore, the baseline plant included this concept. Results of the economic evaluations on the commercial plant indicated that the capital and operating cost of the process needed to be reduced to make the process economically attractive relative to current waste disposal costs. Results of additional analytical studies indicated that partial oxygen firing of the CMS® could improve the energy efficiency of the vitrification system, thus reducing the size and capital cost of the equipment and the energy costs to the extent that the commercial plant is economically attractive.

1.3 **THIRD BUDGET YEAR PERFORMANCE SUMMARY**

Approximately sixty-six hours of pilot-scale SPL vitrification testing was performed during the third budget period with the objective of validating analytical modeling results which predicted system performance improvements via oxygen enrichment and baghouse dust recycling in the Vortec CMS®. The specific goals were to verify and quantify the increase in partitioning of fluoride to HF in the flue gas, increase in HF concentration in the flue gas, and increase in waste throughput as a result of oxygen enhancement and baghouse dust recycle. Additionally, the size of the baghouse dust recycle stream was to be quantified, along with the impact of oxygen enhancement on NOx emissions and the effectiveness of staged combustion in the CMS® on NOx emissions.

To address these issues, approximately 19,000 lbs of SPL waste was vitrified in Vortec’s pilot-scale CMS® located in Harmarville, PA, and chemical composition and leaching analyses were performed on over sixty feedstock, glass, and flue gas samples taken during the testing. The SPL waste utilized during the tests was provided by Ormet Aluminum Corporation from their Hannibal, Ohio, primary aluminum smelting plant.
HF concentrations in the flue gas as high as 1.2%(v) were measured during testing, a factor of 3 higher than previously measured during testing without oxygen enhancement or baghouse dust recycling. However, problems encountered with the laser HF measuring instrumentation due to high particulate and HF loadings in the flue gas limited the operational time of the equipment. Flow rate measurements and results of chemical composition analyses on feedstock, glass, flue gas, and flue gas particulate samples taken during the trials indicate that HF concentrations of 2%(v) or more may have been achieved. Problems were also encountered with agglomeration of feedstock in the injection system, thus limiting testing time so that optimum operating conditions were not achieved with oxygen enhancement. Establishment of optimum conditions would further increase the HF concentration in the flue gas. Sufficient experimental data was obtained to define the size of the baghouse recycle stream at steady state conditions (17% of the feedstock flow) and to determine that the feedstock throughput was increased by about 40% via oxygen enhancement to 28 wt.%.

Experimental data also indicated that the fluoride concentration in the glass increased by a factor of about 3 as a result of baghouse dust recycling. The concentration of fluoride in the leachate from the U.S. EPA recommended leaching procedure on the glass samples was typically an order of magnitude lower than the 1996 EPA Land Disposal Restriction (LDR) standard and two orders of magnitudes lower than that reported by the EPA as typical for the residue from the Reynolds’ Gum Springs, Arkansas, SPL treatment process. However, the leachate concentration was slightly higher than the EPA’s July 12, 2000, proposed LDR standard. The highest fluoride concentration observed in the leachate from the pilot-scale test glass samples was 4.6 mg/l versus the EPA’s proposed standard of 2.7 mg/l. The cyanide concentrations in the pilot-scale test glass samples were typically less that 0.2 mg/kg with the maximum observed of 0.5 mg/kg. This is two orders of magnitude lower than that reported by the EPA to be observed in the treated residue from the Reynolds Gum Springs SPL treatment process.

The effect of oxygen enhancement on NO$_x$ emissions in the CMS$^\circledR$ and effect of staged combustion in reducing these emissions was not evaluated because of the limited test time resulting from the feedstock agglomeration problems.

As a result of the significant improvement observed in partitioning of fluoride in the SPL to the flue gas as HF and the concentration of HF in the flue gas as a result of baghouse dust recycling and oxygen enrichment of the CMS$^\circledR$ during the third budget period testing, Vortec and Alcoa recommended to the DOE that additional pilot-scale testing be performed to expand the experimental data base. DOE approved a one-year extension of the project for additional pilot-scale testing and analysis.

1.4 FOURTH BUDGET YEAR PERFORMANCE SUMMARY

Approximately fifty hours of pilot-scale SPL vitrification testing was performed during the fourth budget period with the objective of validating analytical modeling results that predicted system performance improvements via oxygen enrichment and baghouse dust recycling in the Vortec CMS$^\circledR$. The specific goals were to verify and quantify the increase in partitioning of fluoride to HF in the flue gas and the increase in HF concentration in the flue gas resulting from oxygen enhancement and baghouse dust recycle. Additionally, the size of the baghouse dust recycle stream was to be quantified, along with the impact of oxygen enhancement on NO$_x$ emissions and the effectiveness of staged combustion in the CMS$^\circledR$ in reducing NO$_x$ emissions.
To address these issues, approximately 19,000 lbs of SPL waste was vitrified in Vortec’s pilot-scale CMS® located in Harmarville, PA, and chemical composition and leaching analyses were performed on over sixty feedstock, glass, and flue gas samples taken during the testing. The SPL waste used during the tests was provided by Ormet Aluminum Corporation from their Hannibal, Ohio, primary aluminum smelting plant.

HF concentrations in the flue gas greater than the 2%, the goal specified by Alcoa, were achieved during the testing. Three different methods of measuring HF concentration in the flue gas were used during the tests: a tunable diode laser spectroscopy system developed by Unisearch Associates, Inc; an FTIR system; and flue gas sampling and analysis in accordance with U.S. EPA Method 13B. The concentrations measured by each method differed significantly. Based on an average of the measured values, about 75% of the fluoride from the SPL appeared to partition to the flue gas as HF. The baghouse dust recycle stream varied from 11 wt. % and 23 wt. % of the feedstock input to the process. NOx concentrations in the flue gas were about 25% lower with oxygen enhancement than without oxygen enhancement. The mass flowrate of NOx emissions were about 50% lower with oxygen enhancement, because of the reduced flue gas flowrate achieved with oxygen enhancement.

2.0 INTRODUCTION

SPL is the carbon and refractory waste generated by the primary aluminum industry during the decommissioning of electrolytic cells. It includes first cut carbon, from the bottom block and side walls, and second cut thermal insulating material, typically composed of refractory bricks, or any other arrangement of carbon and refractory material in the electrolytic cell. It may also contain tramp aluminum, iron, and silicon carbide normally associated with the decommissioning of the cells. The U. S. Environmental Protection Agency (EPA) has identified SPL as a listed hazardous waste (K088) subject to RCRA regulations due to the presence of cyanide, fluoride, aromatic hydrocarbons and heavy metals. The primary means of disposal of this waste by aluminum reduction facilities has been by landfilling in hazardous waste landfills. Land Disposal Restrictions (LDR) for K088 went into effect on October 8, 1997. The LDR identified numerical standards for hazardous constituents in SPL that must be achieved prior to land disposal. However, in 1998 the LDR standards were challenged by several aluminum companies and a federal court invalidated certain aspects of the LDR rule for SPL. The U.S. EPA published proposed revised LDR requirements for SPL in July 2000, based on new vitrification data provided by Ormet Corporation. In anticipation of more stringent SPL disposal standards, aluminum producers are seeking alternative SPL treatment and recycling technologies.

SPL Vitrification

SPL contains many of the chemical oxides typically used in the manufacture of glass products (sodium oxide, calcium oxide, silicon oxide, fluorides, etc.), but in different concentrations. Adjusting the concentration of these oxides by blending the SPL with other materials (such as sand, limestone, and waste glass) can result in a feedstock composition suitable for glass manufacture.

However, SPL also contains significant quantities of carbon (ca. 40%) and some cyanide that can be detrimental to the operation of conventional, pool type glass melting furnaces. Oxidation of these constituents before they become mixed with the pool of glass is difficult in conventional furnaces; therefore, the carbon and cyanide can “rob” oxygen from the glass making compounds, such as silica and iron oxide. This glass reduction can shorten glass-contact refractory life and can result in the formation of silicon stones that can be detrimental to glass forming operations.
The patented Vortec CMS® is a fossil fuel fired, suspension heating and melting technology developed for rapid, efficient glass melting and industrial waste vitrification. An artist rendering of the basic CMS® components is presented in Figure 2-1. The CMS® process vitrifies material in two steps. The first step consists of preheating the feedstock to its melting temperature in suspension with reaction gases in a counter-rotating-vortex (CRV) reactor. This is followed by mixing of the feedstock in a cyclone melter in which the glass forming chemical reactions occur. Preheating the SPL in suspension with oxygen rich reaction gases in the CMS® provides the ability to control the oxidation of the carbon and cyanide prior to the melting operation, thus maintaining the balance between reducing and oxidizing conditions necessary to tailor the glass composition for specific commercial products.

Prior to initiation of the DOE sponsored project, Vortec performed preliminary laboratory testing to evaluate the technical feasibility of producing fibers from glass made from feedstock containing SPL waste. During this preliminary investigation, glass frit was made from SPL in Vortec’s CMS® test facility in Harmarville, Pennsylvania, and sent to CertainTeed Corporation, a major U. S. fiberglass manufacturer, for analysis. Laboratory analyses of the frit indicated that the glass may be suitable for fiberizing with a wheel-type fiberizer typical of that used for production of mineral and ceramic fibers. Preliminary assessments of the CMS® process in this application also indicated that it could result in significant energy and cost savings relative to treatment and land disposal of the SPL waste.

Based on the results of these preliminary assessments, the next logical step in the research was pilot-scale testing to produce a sufficient quantity of fibers, from feedstock containing SPL, for product quality
analysis via standard test methods and product safety assessment. Therefore, Vortec proposed to the DOE continuation of the research via the subject pilot-scale testing project.

Flue Gas Cleanup

SPL waste (combined first and second cut) contains on the order of 10% fluoride. Up to 30% of the fluoride is incorporated into the glass produced in the CMS®, depending on the feedstock composition and operating temperature, acting as a fluxing agent to reduce the glass melting temperature. The remaining fluoride leaves the CMS® in the flue gas as HF and fluoride salts. The concentration of fluorides in the flue gas is such that some removal is required prior to discharge of the gas to the atmosphere. Since the fluoride must be removed to protect the environment, and since certain compounds, such as calcium fluoride and aluminum fluoride, have commercial value in the aluminum and steel industry, it makes sense to design an APC system that will convert the fluorides to useful products.

Calcium fluoride, referred to as fluorspar, is used by the aluminum, steel, and ceramic industries. The use depends on the purity, or grade, of the fluorspar. Acid grade, containing greater than 97% calcium fluoride, is used by the aluminum industry to make aluminum fluoride through an intermediate process that reacts the fluorspar with sulfuric acid, producing hydrofluoric (HF) acid that is then reacted with aluminum oxide (alumina). This product has a market value of about $100 per ton. Ceramic grade, containing between 90% and 97% calcium fluoride, is used in relatively small quantities by the ceramic industry as a fluxing agent. Metallurgical grade (<90% calcium fluoride) is used by the steel industry, again in small quantities, to assist with liquefying lime additives in furnaces. Market value for the lower grades of fluorspar are on the order of $10 per ton. Wet scrubbing systems would be used for producing calcium fluoride in APC systems.

Since it is anticipated that a major portion of the fluoride in the CMS® flue gas would be in the form of HF, there is the potential for producing aluminum fluoride directly in an APC system via reaction with alumina. Aluminum fluoride production would provide significant benefits relative to calcium fluoride production. First, aluminum fluoride can be used directly in the primary aluminum production process. Second, it has a relatively high economic value ($1,000 per ton at 90% purity) to the aluminum producer. In addition, the equipment needed to let this chemical conversion take place operates under dry conditions at atmospheric pressure, at temperatures around 800°F, and produces a dry powder which has good handling properties.

The lack of substantial information relative to the CMS® flue gas composition, particularly with respect to the concentrations of HF and fluoride salts, has been the limiting factor in the design of a commercial scale APC system for integration with a CMS® recycling SPL waste. The fluoride salts must be removed prior to the production of the aluminum fluoride to prevent contamination of the product which would eventually jeopardize stable operation of aluminum pot cells to which it is recycled. After determining the concentration of HF relative to sodium fluoride, the next hurdle is the selection of proper filter material for high efficiency removal of sodium fluoride particles at relatively high temperature (800°F) and verification of its performance. Therefore, Vortec Corporation proposed to the DOE that the research program include sampling and analysis of flue gas from the CMS® during pilot-scale testing and conceptual design of an effective APC system producing value-added fluoride products.

3.0 PROJECT PLAN
3.1 **PROJECT GOALS AND SCOPE**

The goal of the project was to produce sufficient experimental data to verify the technical feasibility of producing commercial quality glass fibers and aluminum fluoride from SPL using Vortec’s CMS® technology integrated with conventional glass fiberizing technology and a high efficiency fluoride scrubbing system technology. The scope of the project included:

1. Design, fabrication, and installation of pilot-scale glass fiberizing and flue gas cooling, filtration, sampling, and analysis equipment in Vortec’s existing pilot-scale CMS® test facility;
2. Pilot-scale SPL vitrification tests producing glass fibers using Vortec’s pilot-scale CMS®;
3. Sampling and analysis of flue gas produced during the pilot-scale tests to define the fluoride salt and HF concentrations in the flue gas;
4. Analysis of glass fibers produced during the pilot-scale tests to provide data for product quality and human health risk assessments; and
5. Preliminary design of an air pollution control system for aluminum fluoride production.

3.2 **STATEMENT OF OBJECTIVES**

The objectives of the project were to:

1. Produce glass fibers via pilot-scale SPL vitrification tests with Vortec’s CMS® technology and conventional wheel-type fiberizing technology;
2. Measure the HF and fluoride salt concentrations in the CMS® flue gas during pilot-scale testing;
3. Determine the efficiency of a high temperature filter in removing particulates from the CMS® flue gas;
4. Evaluate the quality of glass fibers produced during the pilot-scale tests via standard testing and analysis;
5. Evaluate the solubility characteristics of the glass fibers produced during pilot-scale tests to provide data for the assessment of the affect on human health;
6. Assess the feasibility of producing aluminum fluoride via reaction of alumina with HF in the CMS® flue gas;
7. Design a cost-effective scrubbing system for removal of fluoride from the CMS® flue gas and production of a high purity, aluminum fluoride product.

3.3 **WORK BREAKDOWN STRUCTURE AND TASK DESCRIPTIONS**

The project consisted of six technical tasks and a program management task to accomplish the project objectives. These tasks were:

1. Glass and Feedstock Composition Design
2. Pilot-Scale Component Design and Procurement
3. Pilot-Scale Test Loop Modification and Component Installation
4. Pilot-Scale SPL Vitrification/Glass Fiber Production Testing
5. Glass Fiber Qualification Testing
6. Air Pollution Control System Preliminary Design
7. Program Management and Reporting

Descriptions of the objectives and planned activities for each of these tasks follows:

3.3.1 Task 1 – Glass and Feedstock Composition Definition

The objective of this task is to establish a glass and feedstock composition which maximizes the utilization of SPL in the feedstock while producing a glass that can be fiberized using conventional wheel-type fiberizing technology. An additional objective is to define alternative raw materials which can be used as additives to the SPL to achieve the desired feedstock and glass composition.

The initial target glass composition will be one which was previously identified by Vortec and for which preliminary laboratory fiberizing analysis indicated will meet conventional fiberizing technology criteria. In identifying additives for the feedstock, consideration will be given to the availability and cost of alternative raw materials and maximizing the concentration of SPL in the feedstock. The defined feedstock composition will be used for the SPL vitrification tests performed under Task 4.

3.3.2 Task 2 – Pilot-Scale Component Design and Procurement

The objective of this task is to design, specify, and procure equipment to fiberize the glass produced by the CMS®, cool the gas from the CMS® to conditions required for sampling, remove particulates from the flue gas, and sample the flue gas during pilot-scale vitrification testing. The glass fiberizing equipment will be procured during the first budget period. The flue gas conditioning equipment will be procured in the first budget period, and the particulate removal equipment in the second budget period.

The fiberizing equipment will consist of commercially available components typically used in the manufacture of ceramic and mineral wool fibers. It will include a three wheel fiberizer, spindle, air plenum, and frame assembly, variable frequency drive motor, wheel assembly cooling (water) and lubrication (oil) systems, and air blower and motor.

The flue gas cooling system existing in the CMS® test facility at the beginning of the project consists of a quench duct with several water sprays, a water recirculation tank, and water supply pump (shown in Figure 3.3-1). With this system, the flue gas is deluged with water to cool the gas to approximately 200°F. Excess water is drained to a recirculation tank from which it is pumped back to the quench duct. This type of system makes it difficult to accurately determine the chemical composition of the flue
Figure 3.3-1  Pre-Project Flue Gas Quench System

gas because it requires the sampling and analysis of several streams: the flue gas, particulates in the flue gas, the water, and particulates in the water. Additionally, because the water is recirculated, build-up of solid species in the system can introduce error into the analysis. Therefore, to facilitate accurate flue gas chemical composition analysis, the cooling system will be modified for this project to incorporate an evaporative cooling system consisting of an evaporative cooler, water pump, and associated piping and ductwork to interface the system with the CMS® and city water supply. All the water introduced to cool the flue gas will be evaporated and exit as a vapor with the flue gas. This will require sampling only the flue gas for gaseous and solid constituents for analyzing the flue gas chemical composition. This modification will be completed during the first budget period.

Vortec will issue performance specifications for the fiberizing system components; evaluate the bids based on cost and ability to meet specifications and schedule; and issue and expedite a purchase order to the selected vendor. Vortec will also issue purchase orders for the ancillary equipment.

Hoogovens Technical Services, Inc. (HTS) will design and purchase the evaporative cooler for the pilot-scale test facility. Vortec will provide HTS with temperature and flow conditions of the flue gas from the pilot-scale CMS® based on testing previously performed.

HTS will also provide specifications for a high temperature filter for removing particulates from the flue gas. Vortec will issue the specifications to qualified vendors for bids; evaluate bids received based on cost and ability to meet specifications and schedule; and issue and expedite a purchase order to the selected fabricator. This component will be procured during the second budget period of the project.
The scope of Task 2 was expanded for the third budget period to include design and procurement of a combination feedstock/oxygen injector for integration with the CRV burner for use in oxygen enhancement testing; the installation of an air injection system for staged combustion testing; and the modification of the control system for controlling fuel, oxygen, and air injection into the process.

3.3.3 Task 3 – Pilot-Scale Test Loop Modification and Component Installation

Vortec will design modifications to its pilot-scale CMS® test loop required to install the new components. Vortec will prepare layout drawings illustrating the relocation of existing equipment and installation of new components. Vortec will design and prepare construction drawings for foundations, pads, and any structural supports, P&ID’s, electrical diagrams, and installation drawings and instructions detailing the procedures for installing the new components and integrating them with existing equipment and utility services.

Vortec personnel will make the modifications to the pilot-scale CMS® test loop and install the equipment purchased under Task 2 in the test facility. The particulate filtration equipment will not be installed in the facility until the second budget period. Vortec personnel installed all the existing components and support systems in the test facility and has maintained and made all modifications to the system since its construction. New components will be installed in accordance with the manufacturer’s installation instructions. HTS will provide on-site supervision for the installation of the flue gas cooling and filtration equipment. The components will undergo cold checkout after installation to ensure proper operation prior to initiation of the vitrification testing in Task 4.

Relining of the high temperature components in the CMS® test loop with refractory will be required prior to vitrification testing. The relining with AZS refractory will be accomplished under this task. Vortec personnel will also make any modifications or additions to foundations, pads, and structural supports required for installation of the new components.

3.3.4 Task 4 – Pilot-Scale SPL Vitrification/Glass Fiber Production Testing

SPL vitrification tests will be performed using Vortec’s pilot-scale CMS® (shown in Figure 3.3-2) to produce glass fibers for product quality and human health risk assessments, and to produce flue gas compositional data for design of a fluoride scrubbing/aluminum fluoride production system. The tests will be performed in accordance with Pennsylvania Department of Environmental Protection (PADEP) regulations regarding hazardous waste treatability studies.

Two tests will be performed, one during the first twelve month budget period and one during the second budget period. The objectives of the test during the first budget period will be to produce glass fiber for product quality and human health risk assessment and collect flue gas samples for fluoride concentration analysis. The objective of the second test, performed during the second budget period, will be to collect flue gas particulate samples upstream and downstream of particulate filters and evaluate the efficiency of solid fluoride removal. The second test will be performed over a two week period at the same feedstock flow conditions as in the first test.

Alumax will provide approximately 4,000 kg (8,800 lb) of first cut and 4,000 kg of second cut SPL for the pilot-scale vitrification testing in Task 4. Alumax will reduce the size of the SPL to approximately ½
inch in size prior to shipping the material to Vortec’s test facility. Vortec will further reduce the SPL to the size distribution specifications of the CMS® using an existing ball mill in Vortec’s test facility.

Vortec will purchase the glass-forming additives required and blend the SPL with the additives in accordance with the feedstock composition designed in Task 1. The blending will be accomplished in an existing pneumatic blend tank in Vortec’s test facility. The blended material will then be transferred into flexible intermediate bulk containers (FIBC’s) for subsequent delivery to the CMS®.

Flowrates of feedstock, natural gas, and air to the CMS®, glass and gas temperatures in the CMS® components, and pressure in the components will be measured and recorded using the facility’s existing data acquisition system. Flue gas samples will be taken in accordance with U. S. EPA methods and the samples analyzed for gaseous and solid fluoride concentrations and total dust concentrations.

During the course of the second pilot-scale testing period, particulate samples will be collected from the high temperature flue gas filter. Samples collected will be analyzed by an outside laboratory for chemical composition, particularly with respect to fluorides. These data will be used as input in assessing the feasibility of aluminum fluoride production via scrubbing of fluoride from the flue gas and preliminary design of a commercial scale APC system.

![Figure 3.3-2 Vortec Pilot-Scale CMS®](image)
The scope of this task was expanded for the third budget period to include additional pilot-scale testing to evaluate the effect of oxygen enhancement and baghouse dust recycling on fluoride partitioning to the flue gas and hydrogen fluoride concentration in the flue gas. The SPL to be used in the third budget period tests was provided by Ormet Primary Aluminum Corporation from their aluminum smelting plant in Hannibal, Ohio. Ormet provided a mixture of 1st cut SPL (carbon block) and 2nd cut refractory material. Ormet ground the material to approximately 25% minus 170 mesh in their SPL size reduction facility, and Vortec further ground the material to about 75% minus 170 mesh in a ball mill located in Vortec’s test facility.

The scope of work included chemical composition analysis of the feedstock, glass, flue gas, and flue gas particulate collected during the pilot-scale testing by Alcoa, Inc., and leaching analysis of the glass by Alcoa, Inc.

3.3.5 Task 5 – Glass Fiber Qualification Testing

Glass fibers from the first vitrification test will be analyzed with respect to thermal performance in accordance with ASTM building materials testing methods. The results of the analyses will be compared to those of existing commercial products. These tests and analyses will be performed during the first budget period. Based on the results, the compositions of the glass and feedstocks defined in Task 1 will be re-evaluated and any changes warranted will be incorporated in the second vitrification test.

The primary health concern with respect to the use of fiber products is the respiratory effects on personnel handling the material. The New York State University College of Ceramics at Alfred University will perform dissolution tests on fiber products from the pilot-scale testing and commercially available fibers. This analysis will be performed during the second budget period.

3.3.6 Task 6 – Preliminary Design of Commercial Air Pollution Control System

HTS will assess the feasibility of cost-effective production of aluminum fluoride in an APC system integrated with a CMS® recycling SPL, based on the second pilot-scale test data. HTS will develop a preliminary design of a commercial scale APC system which will include equipment necessary for cooling the flue gas, removing sodium fluoride and other particulates, aluminum fluoride reactions, and final cleanup of other acid gases and particulates. HTS will develop process descriptions, process flow diagrams with flow data, equipment list, equipment performance data sheets, P&ID’s, process control logic, and capital equipment, operating, and maintenance costs. Based on the design, HTS will develop capital cost estimates for the equipment. This task will be performed during the second budget period.

3.3.7 Task 7 – Program Management and Reporting

The objective of the project management and support task is to ensure the timely, professional, and cost effective execution of the contract. Vortec’s Program Manager will direct the technical effort of the project team, be the single point of contact, assure that the deliverables are prepared as required, and assure that the entire program remains within the schedule and budget.
Vortec will prepare quarterly technical and financial status reports, annual technical progress report, and a comprehensive final report. Vortec will also prepare interim reports as required by the U. S. DOE.

4.0 PROJECT SCHEDULE

The project was originally scheduled to be completed in two years. However, the project was extended for an additional year to perform pilot-scale testing to evaluate the effect of oxygen enhancement and baghouse dust recycling on fluoride partitioning to the flue gas and hydrogen fluoride concentration in the flue gas. The project schedule is shown in Figure 4-1.

![Figure 4-1 Project Schedule]

5.0 PROJECT PERFORMANCE SUMMARY

5.1 TASK 1 – GLASS AND FEEDSTOCK COMPOSITION DEFINITION

During the first budget period, glass and feedstock composition criteria were established based on the composition of a glass that was produced from SPL by Vortec during laboratory scale investigations under a previous Vortec/industry sponsored program. Results of the laboratory analyses indicated that the glass may be suitable for fiberizing using a wheel-type fiberizer. The target glass viscosity was 1,000 poise at a temperature less than or equal to 2,450°F. Considerations in establishing the criteria were:

1. An acceptable system operating temperature (≤ 2,700°F),
2. Maximize SPL waste loading in the feedstock, with a goal of ≥ 40%,
3. Use low cost additives in the glass formulation,
4. Maximize fluoride capture in the glass.

A feedstock consisting of 45% SPL waste from Alumax and 55% additives (waste glass, sand, and limestone) meeting the above criteria was defined. Glass produced in the vitrification trials was analyzed by CertainTeed Corporation. Results of the analyses verified that the target composition and viscosity were met. This composition was used during the first budget period SPL vitrification and glass fiberizing trial.
Based on reviews of the results of the first budget period fiberizing trial with CertainTeed, the viscosity criteria and target composition for the glass were redefined. The feedstock formulation was then modified to meet the target criteria and used as input to an additional fiberizing trial performed during the second budget period. The re-defined feedstock formulation still consists of about 45% SPL waste and 55% low-cost additives.

During the third budget period, a feedstock formulation was defined to produce a glass meeting the project specifications based on the chemical composition of the Ormet SPL waste. The formulation also took into consideration the anticipated chemical composition of the baghouse dust that would be generated and recycled back into the CMS® during the vitrification of SPL. The result was a feedstock formulation consisting of greater than 50% SPL waste.

5.2 Task 2 – Pilot-Scale Component Design and Procurement

5.2.1 Glass Fiberizing System

Vortec submitted requests for quotations to qualified vendors for the fabrication of a wheel-type fiberizer based on fabrication drawings that Vortec had purchased from Dynamic Research, Inc., a former manufacturer of fiberizers. Vortec evaluated the bids received and issued a purchase order for fabrication. Photographs of the fiberizer are shown in Figure 5.2-1.

The fiberizer consists of three stainless steel wheels. Each wheel is driven by a variable speed motor capable of providing wheel speeds up to 12,000 rpm. Each wheel is water cooled, and the drive shafts are oil lubricated. A fan, driven by a 7.5 hp, 3,600 rpm motor, delivers 2,000 cfm of air at a static pressure of 11” w.c. to a shroud of nozzles around the wheels. The fiberizing wheel assembly is mounted on a platform which can be moved in the forward, reverse, and side directions by linear actuators to allow for precise positioning of the wheels under the glass stream. The platform is mounted on casters which ride on rails to allow greater movement of the assembly in the forward and reverse directions.

A programmable logic controller (PLC) based fiberizer control system was designed by Vortec and its subcontractor, Automations Engineering, Inc. An operator provides input to the control system on a control console (shown in Figure 5.2-2). An electronic interface enables the operator to monitor operating conditions and input parameters into the control system. A joystick enables the operator to provide directional control input. A video monitor on the panel enables the operator to see the position of the fiberizer wheels in the glass stream as transmitted by a video camera located at the fiberizer.

The control system contains safety interlocks to ensure that:

1. lubricating oil is provided to the drive shafts before starting the motor drives,
2. the wheels are up to design speed and air is being delivered to the shroud before the wheels can be positioned under the glass stream,
3. the wheel speed is below the maximum safe speed at which they can be removed from the glass stream to prevent glass from being flung outside a protective enclosure,
4. the fiberizer assembly is not moved beyond safe operating position limits.

The fiberizer was used in the first fiberizing trial during the first budget period. The results of the first trial were reviewed with CertainTeed Corporation and Saint Gobain, the parent company of CertainTeed
and the largest European glass manufacturer with extensive experience in glass fiber production with wheel-type fiberizers. Saint Gobain recommended minor modifications to the fiberizer based on their experience. The modifications included a re-design of the surface of the fiberizing wheels and the air shroud, modifying the spacing between the wheels and the distance between the air shroud and the wheels, and increasing the flow of air to the wheels. Vortec re-designed the pilot-scale fiberizer and made the modifications accordingly. The modified fiberizer was used in an additional, successful fiberizing trial performed during the second budget period, as discussed in Section 5.4.4.

A glass fiber collection box (shown in Figure 5.2-3) was also designed, and fabrication drawings were prepared by Vortec. Requests for quotations were solicited from qualified fabricators and evaluated with respect to cost and ability to meet the project schedule. A fabricator was selected and purchase order issued.

The collector consists of a rectangular carbon steel framework with dimensions 6 ft in width, 15 ft in length, and 10 ft in height. The sides, bottom, and lower half of the front of the frame (portion of the frame facing the fiberizer) are covered with sheet metal. The upper portion of the front is open and accepts the fibers that are blown from the fiberizing wheels. The top and the back of the frame are covered by a screen to allow air to pass through the collector but to contain the fibers produced. The collector is supported by casters on each corner so that it can be easily moved into and out of position.

5.2.2 Flue Gas Cooling System

Vortec provided HTS with design flue gas conditions for the flue gas cooling system. HTS prepared fabrication drawings and specifications for the equipment. Vortec reviewed and approved the drawings prior to their being issued by HTS to qualified vendors for bids. HTS evaluated the bids and issued purchase orders during the first budget period for the procurement of the equipment.

The flue gas cooling system consists of an evaporative cooler, ductwork to interface the separator/reservoir component of the CMS with the evaporative cooler, water pumps to supply water to the evaporative cooler, ductwork for interfacing the evaporative cooler with the wet electrostatic precipitator that already existed in the test system, and instrumentation and control equipment. A sketch of the evaporative cooler and interfacing ductwork is presented in Figure 5.2-4. A photograph of the evaporative cooler is shown in Figure 5.2-5.

The evaporative cooler was designed to cool up to 6,186 lb/hr of flue gas from a temperature of 2,550°F to 450°F. The evaporative cooler consists of a vertical carbon steel shell with refractory lining for heat protection. The shell was fabricated in five sections for ease of installation. A single spray lance consisting of water and atomizing air inlet pipes and a nozzle (shown in the photograph in Figure 5.2-6) is installed at the top of the unit. Air is provided to the lance by a plant air system that was existing at the test facility. Water and air are supplied to the lance via quick-disconnect piping. A spare lance is provided as a backup. A separate opening is provided in the shell to allow the backup lance to be installed before removal of the operating lance.
Figure 5.2-1  Photographs of Wheel Type Fiberizer in CMS® Test Facility
Figure 5.2-2  Fiberizer Control Console

Figure 5.2-3  Fiber Collection Box
Figure 5.2-4 Sketch of Evaporative Cooler and Ductwork

Figure 5.2-5 Photograph of Evaporative Cooler
The evaporative cooler was designed for dry bottom operation. That is, all the water introduced into the flue gas stream is vaporized and leaves the cooler with the flue gas. The unit was sized based on a conservative gas retention time for operational flexibility in the test facility. An important design criteria from a cost viewpoint was that the evaporative cooler be integrated into the existing structure in the test facility with minimal modification to the structure. The evaporative cooler is supported by four legs that are mounted on pedestal foundations on the floor of the test facility.

The ductwork interfacing the separator/reservoir component of the CMS® consists of carbon steel vertical and horizontal shell sections with refractory lining for heat protection. The refractory lining consists of a high temperature, alumina-zirconia-silica (AZS) refractory backed by an insulating refractory. The insulating refractory maintains the steel shell temperature above the HF condensation temperature to avoid the formation of hydrofluoric acid on the inside shell surface. The interfacing ductwork and the evaporative cooler contains multiple openings for preventative or active maintenance. It also contains ports in the ductwork before and after the evaporative cooler for flue gas sampling in accordance with EPA methods. The interfacing ductwork is supported by a steel structure straddling the
separator/reservoir component of the CMS® and constant load hangars to allow for thermal expansion of
the ductwork. A refractory-lined expansion joint connects the ductwork with the evaporative cooler to
allow for the differential thermal expansion between the ductwork and evaporative cooler.

Water is supplied to the spray lance in the evaporative cooler via one of two vertical, multi-stage pumps,
shown in Figure 5.2-7, capable of delivering 7 gpm at 260 ft. TDH. One pump is operational while one is
on standby. The pumps are constructed of 304 stainless steel with silicon-carbide mechanical seals. Each
pump is driven by a 2 hp, 3600 rpm, 115/1/60 TEFC motor. Two 40 mesh strainers are located in the
pump inlets to remove solid material that may damage the pump or plug the spray lance nozzle. Air is
supplied to the evaporative cooler spray lance by a plant air system existing in the test facility. This
system can provide 125 scfm at 100 psig.

Controls for the evaporative cooler were integrated into the CMS® PLC based control system. Two
thermocouples are located in the exhaust duct from the evaporative cooler and transmit the measured flue
gas temperature to the control system. Orifice plates and pressure transducers measure the water and
atomizing air flow rates and pressures in the supply lines to the spray lance and transmit these data to the
control system. The control system maintains the flue gas temperature at the discharge of the evaporative
cooler by manipulating the water and atomizing air control valves.

Figure 5.2-7  Evaporative Cooler Water Supply Pumps
5.2.3 Baghouse System for Particulate Removal from the Flue Gas

During the second budget period, HTS solicited quotations for procurement of a baghouse system for removing particulates from the CMS® flue gas in the pilot-scale test facility based on flue gas condition data provided by Vortec. HTS reviewed the quotes received with Vortec and issued a purchase order for a baghouse system. The system procured was a CLINOX system designed by Biothermica International, Inc. A photograph of the system is shown in Figure 5.2-8. The system consists of 120 woven stainless steel, removable filters each 7 inches in diameter and 10 ft. in length. The total net filtration surface area is 1,760 ft². The system can filter 7,500 acfm of flue gas at a filtration velocity of 3.6 ft/min. at a temperature of 550°C (1,022°F). The filters are enclosed in a housing constructed of 316L stainless steel with external insulation to keep the outside surface temperature below 60°C (140°F). The instrumentation includes differential pressure (clean vs. dirty side) transmitter, pressure switch, clean and dirty side thermocouples, and pressure and temperature displays. Cleaning of the filters is accomplished with a pulse jet system consisting of an air tank manifold, air distributor pipes, valves, and instrumentation and controls for sequencing air pulses. The ductwork transporting the flue gas to and from the baghouse contains ports for flue gas sampling in accordance with EPA methods.

Figure 5.2-8 Baghouse Filter System

5.2.4 Cyclone Melter Air Injection System
Vortec designed and fabricated an interface with the existing site port in the cyclone melter, shown in Figure 5.2-9, for injection of air into the cyclone melter during the testing to evaluate the effect of staged combustion on NOx emissions from the CMS. An air blower contained in a burner system already existing at the test facility, shown in Figure 5.2-10, was connected to the interface for air supply.

5.3 TASK 3 – PILOT-SCALE TEST LOOP MODIFICATION AND COMPONENT INSTALLATION

Vortec and HTS prepared construction drawings for the modification of the pilot-scale CMS test loop and installation of the new components. Layout drawings were prepared showing the relocation of existing equipment required and the location of the new components. Construction drawings were prepared for foundations, pads, and structural supports in addition to P&ID’s, electrical diagrams, and installation drawings and instructions. Vortec personnel performed the modifications and component installations. A layout sketch showing the location of the major components inside the test facility building in relation to the existing CMS equipment is shown in Figure 5.3-1. A photograph of the CMS, refractory lined duct from the CMS to the evaporative cooler, and evaporative cooler is shown in Figure 5.3-2. A photograph showing the arrangement of the flue gas cleanup equipment (baghouse, wet electrostatic precipitator and induced draft fan) after the installation of the baghouse system is shown in Figure 5.3-3.

5.3.1 Glass Fiberizing System

Horizontal steel beams were welded to existing steel columns in the test facility to support the rails for the fiberizer assembly. The rails were bolted in place and the fiberizer assembly mounted on the rails via a 2-ton forklift. A photograph of the fiberizer assembly installed in the facility is shown in Figure 5.3-4. The fiberizer control panel was mounted on an existing platform in the test facility (also shown in Figure 5.3-4) in a location where the operator has a good view of the fiberizer assembly. A video camera mounted at the fiberizer transmits to a monitor located on the control panel to enable the operator to see the position of the wheels in the glass stream. All electrical and instrumentation and control cables to and from the fiberizer and control system were routed through cable trays and installed in accordance with local safety codes. The electrical system existing in the facility was sufficient for the installation of the fiberizing system; therefore, no modifications to the facility electrical system were required.

5.3.2 Flue Gas Cooling System

Modifications to the flue gas cooling system in the CMS test loop consisted of removing the existing excess water quench system and installing the evaporative cooling system. The quench duct and water supply piping associated with the existing flue gas quench system were removed. The evaporative cooler and ductwork interfacing the cooler with the existing CMS and wet electrostatic precipitator equipment were installed using a 5-ton overhead crane existing in the test facility. These components were fabricated in sections to facilitate installation. Pedestal foundations were constructed on which to set the four legs supporting the evaporative cooler. A structural steel frame was fabricated and installed to support the vertical refractory lined duct interfacing the
Figure 5.2-9  Air Injection Interface To Cyclone Melter

Figure 5.2-10  Air Supply System for Cyclone Melter Air Injection
Figure 5.3-1  Layout Sketch of Major Components Added to CMS® Test Loop

Figure 5.3-2  Major Components in CMS® Test Loop
Figure 5.3-3  Flue Gas Cleanup System

Figure 5.3-4  Photograph of Fiberizer Installed in CMS® Test Facility
separator/reservoir component of the CMS® with the evaporative cooler. A structural steel frame for hanging the refractory lined duct was existing in the facility. Steel beams were added to the structure in the facility to support the refractory lined “cross-over” duct fabricated to interface the vertical duct with the evaporative cooler.

The pumps for supplying water to the evaporative cooler were mounted on the floor of the facility (as was shown in Figure 5.2-7). The existing water recirculation tank was kept as a fresh water supply tank to the pumps, providing the necessary head of water to the pumps. The tank was plumbed to the existing city water supply system. A float valve controls the flow of water to the tank to maintain a constant water level. A recirculation loop from the water pumps to the tank was installed with a pressure relief valve to maintain the water flow rate through the pumps above the minimum manufacturer recommended rate when the control system calls for a lower rate to the evaporative cooler.

5.3.3 Baghouse System

Modifications to the flue gas cleanup system to incorporate the baghouse system in the CMS® test loop consisted of relocating the WESP and ID fan from the east end of the test building to the west end and installing the baghouse system where the WESP was previously located. This minimized the quantity of insulated ductwork required and constituted the lowest cost installation option.

A foundation and structural steel that existed at the west end of the building, that previously supported a coal storage tank, was sufficient to support the WESP. Another existing foundation and concrete pad at the west end of the building was sufficient for supporting the ID fan. The existing foundation previously supporting the WESP was supplemented by adding footers to support the baghouse.

Only the ductwork external to the building required modification for incorporating the baghouse. All the ductwork between the evaporative cooler and the baghouse was insulated with 4 in. of mineral wool pipe and tank wrap with aluminum cladding as a moisture barrier.

The baghouse housing and supporting steel were shipped in sections and erected at the test facility using a mobile 50 ton crane and manlift. The pulse jet filter cleaning system components and filters were shipped separately and installed at the facility. Electrical wiring and pulse air supply piping were run from the existing electrical supply and plant air systems to the baghouse system. All erection and installation work was performed by Vortec personnel. Biothermica personnel supervised the installation of the fabric filters in the baghouse and the installation of the control system.

5.4 Task 4 – Pilot-Scale SPL Vitrification/Glass Fiber Production Testing

During the first budget period, three vitrification trials were performed over a four day period. The first trial was a shakedown test of the new equipment installed in the test system and was performed using glass cullet (waste consumer glass) as the feedstock. The second and third trials were SPL vitrification trials during which glass fibers were produced and flue gas samples were taken for fluoride analysis.

Two additional SPL vitrification trials were performed during the second budget period based on the results of the first budget period trials. The first trial was performed over an 8 hour period and produced glass fibers with the modified glass composition and fiberizer. The objective of the second trial,
performed over a two day period, was to further characterize the flue gas from the CMS® when vitrifying SPL and evaluate the performance of the baghouse.

Eight additional SPL vitrification trials were performed during the third budget period, from September 1999 through December 2000, to evaluate the effect of oxygen enhancement and baghouse dust recycling on fluoride partitioning to the flue gas and hydrogen fluoride concentration in the flue gas from the CMS® process. Approximately 19,000 lbs of SPL was processed during these trials over a period of about 66 hours of vitrification operation. Two trials were performed with air only as the oxidant to the CMS® (no oxygen enhancement). During these trials, flue gas particulate was collected in the baghouse for mixing and re-injection into the CMS® with the SPL waste during subsequent tests. Three trials were subsequently performed with air only as the oxidant and various concentrations of baghouse dust in the feedstock to the process. Two additional trials were then performed varying oxygen enhancement up to 31 wt.% oxygen (total oxygen in the oxidant), one without baghouse dust in the feedstock and one with baghouse dust in the feedstock.

The SPL vitrification trials were conducted in accordance with the U. S. Environmental Protection Agency (EPA) and Pennsylvania Department of Environmental Protection (PADEP) regulations for treatability studies exempt from the Resource Conservation and Recovery Act regulations.

5.4.1 1st Budget Period Testing

Trial #1 – Shakedown Testing

Approximately 3,815 pounds of feedstock was vitrified over a period of approximately eleven hours to shakedown the system with the added fiberizing and flue gas cooling components. The feedstock consisted of glass cullet from Ormet Primary Aluminum Corporation’s SPL vitrification plant and waste consumer glass cullet. Glass cullet was used instead of a feedstock containing SPL so that the shakedown trial would not be restricted in time by the processing limitations of the EPA and PADEP treatability study regulations. The composition of the feedstock for the shakedown trial was defined to provide a glass from the CMS® similar in composition to that which would be obtained from the feedstock containing SPL waste in the later trials.

The glass cullet obtained from Ormet consisted of material approximately one-eighth to one-fourth of an inch in size. This material was crushed to the size criteria of the CMS® for cullet (≤ 30 mesh) using Vortec’s rotating ball mill at the test facility. The waste consumer glass cullet was purchased from a commercial cullet supplier pre-sized to the CMS® criteria.

After comminution of the Ormet cullet, it was put in FIBC’s and then loaded into Vortec’s pneumatic blend vessel using the facility’s 5-ton overhead crane. The waste consumer glass cullet was delivered to the test facility in FIBC’s and loaded into the blend vessel. The ingredients were then blended and the mixture put back into FIBC’s for loading into the CMS® feed hopper during the vitrification trial.

Hot start-up of the CMS® was initiated seventy-two hours before initiating the first vitrification trial. During this period the refractory manufacturer’s recommended schedule for drying and heating of the refractory in the evaporative cooler and interconnecting ductwork was followed. As the system was heated to operating temperature (2,450°F), the control of the water and air flow to the evaporative cooler
to maintain a specified flue gas discharge temperature was validated. Additionally, proper operation of system instrumentation installed for monitoring and controlling the operating conditions was verified.

After steady-state operating conditions were achieved, feedstock flow to the CMS® was initiated and maintained at a flow rate of approximately 1,000 lb/hr until the separator/reservoir component of the CMS® was filled and a steady state glass stream was discharging from the overflow weir (approximately 1.5 hours). The flow rate was then reduced to approximately 400 lbs/hr for the duration of the trial, with the temperature of the glass discharging from the separator/reservoir at approximately 2,450°F.

During the shakedown vitrification trial, the fiberizer was operated at various conditions to evaluate the effectiveness of fiber production. The fiberizer was moved occasionally so that the glass stream landed at various positions on the first fiberizing wheel and deflected to various positions on the second and third wheel. The speed of each wheel was independently varied up to 12,000 rpm to evaluate the effect of wheel speed on fiber production. Proper operation of the safety interlocks was also validated.

Some glass fibers were produced during the shakedown period. Preliminary micrometer measurements indicated that they were 5 microns and higher in diameter, with most being greater than 20 microns in diameter. On-line measurement of the glass viscosity, an important parameter in fiberizing efficiency was not possible. Therefore, it was not known if the glass viscosity was in the range for optimum fiberizing efficiency. The glass temperature was varied from approximately 2,450°F to 2,550°F with no apparent affect on fiber production.

During the shakedown testing, flue gas sampling equipment was installed and checked out by TNO Institute of Applied Physics, an independent laboratory experienced in the sampling and analysis of flue gas from glass melting operations. Proper operation of the equipment was validated for flue gas sampling during the SPL vitrification trials.

**Trial #2 – SPL Vitrification and Fiberizing Trial (Vortec Test #157)**

Approximately 4,190 lbs of feedstock containing SPL waste was vitrified over a two day period. The objective of this trial was to evaluate fiberizing performance and flue gas composition at an operating gas temperature of approximately 2,450°F and glass temperatures between 2,350°F and 2,450°F.

The feedstock for this trial consisted of first and second cut SPL waste from Alumax Primary Aluminum Corporation’s Eastalco aluminum smelting plant in Frederick, Maryland, and low cost additives. The feedstock contained approximately 45% SPL waste, approximately 1,090 lbs of first cut material and 800 lbs of second cut material. The composition was that defined in Task 1 of the project.

The SPL waste received from Alumax consisted of material up to 4 inches in size. This material was crushed to approximately one-half inch in a jaw crusher and then ground to less than 100 microns in a rotating ball mill at the test facility. The additives were purchased pre-sized to the CMS® criteria.

After comminution of the SPL waste, it was put in FIBC’s and then loaded into Vortec’s pneumatic blend vessel using the facility’s 5-ton overhead crane. The additives were also loaded into the blend vessel from FIBC’s. The ingredients were then blended and the mixture put back into FIBC’s for loading into the CMS® feed hopper during the vitrification trial.
Flow of feedstock to the CMS® was initiated and maintained at about 500 lbs/hr during the vitrification trial. The cyclone melter gas temperature and glass temperature discharging from the separator/reservoir were initially maintained at approximately 2,450°F, and fiberizing trials performed. The position of the first fiberizing wheel under the glass stream and the speed of each wheel were varied in an attempt to optimize fiberizing performance. A small amount of fiber that had the potential to be in the size range desired (3 to 5 microns in diameter) were produced. A position of the fiberizing wheels in the glass stream was found which produced a large quantity of fibers; however, much of the fiber appeared to be greater than 20 microns. A photograph of the fibers coming off the wheels is shown in Figure 5.4-1.

Results of consultation with a high temperature, glass fiber manufacturer during the trial led to consideration of increasing the viscosity of the glass to the fiberizer. Therefore, the fuel and air flows to the CMS® were adjusted to lower the glass temperature to approximately 2,350°F. The positioning of the first fiberizing wheel was again varied to optimize the production of fibers. The quantity of fibers produced was not as large as when the glass temperature was 2,450°F and the diameter of the fibers appeared to be larger.

TNO Institute of Applied Physics sampled the flue gas both at the discharge of the CMS® (inlet to the evaporative cooler) and after the evaporative cooler (sample locations as were shown in Figure 5.2-4) with the cyclone melter gas temperature at approximately 2,350°F. Sampling of the hot flue gas (before the evaporative cooler) was performed with a water-cooled probe. The flue gases were drawn through a gas washing bottle filled either with 1N HNO₃ (nitric acid) to absorb the cations (metals) or 3.5% H₂O₂.
(hydrogen peroxide) to absorb the anions (F, Cl, and S). The flue gas was sampled for particulates and gaseous compounds after the evaporative cooler. The gaseous compounds were sampled with a heated probe to avoid acid condensation. The gases were passed through a particulate filter and then drawn through gas washing bottles filled with 3.5% H$_2$O$_2$ to absorb the anions. The metals were assumed to be in the solid phase at the flue gas temperature (450°F to 500°F) at the exit of the evaporative cooler. The samples were later analyzed by TNO for gaseous fluorides at the inlet to the evaporative cooler and gaseous and solid fluorides and total dust concentration at the exit of the evaporative cooler, as discussed in Section 5.4.4.

Flowrates of feedstock, natural gas, and air to the CMS® and glass and gas temperatures in the CMS® components were measured and recorded for later calculation of mass and energy balances.

**Trial #3 – SPL Vitrification and Fiberizing Trial (Vortec Test #158)**

Approximately 2,100 lbs of feedstock containing SPL waste was prepared for Trial #3 in the same manner as in Trial #2. The composition of the feedstock was the same as in Trial #2. The objective of Trial #3 was to evaluate flue gas composition at an operating gas temperature of 2,650°F. Glass fiberizing was again performed at glass temperatures ranging from 2,450°F to 2,630°F. The 2,100 lbs of feedstock, containing 545 lbs of first cut SPL waste, 379 lbs of second cut SPL waste, and 1,176 lbs of additives, was vitrified over a two day period.

Flow of feedstock to the CMS® was initiated and maintained in the range from 400 lbs/hr to 500 lbs/hr during the vitrification trial. The position of the first fiberizing wheel under the glass stream and the speed of each wheel were varied in an attempt to optimize fiberizing performance. Similar results to those obtained during Trial #2 were observed.

TNO Institute of Applied Physics sampled the flue gas both at the discharge of the CMS® (inlet to the evaporative cooler) and after the evaporative cooler with the cyclone melter gas temperature at approximately 2,650°F. The method of sampling was the same as that used during Trial #2.

Flow rates of feedstock, natural gas, and air to the CMS®, flow rates of water and air to the evaporative cooler; and glass and gas temperatures in the CMS® components were measured and recorded for later calculation of mass and energy balances.

**5.4.2 2nd Budget Period Testing**

**Trial #4 – Vitrification and Fiberizing Trial (Vortec Test #169)**

A fiberizing trial was performed during the second budget period with the feedstock composition and fiberizer modified based on recommendations by CertainTeed and Saint Gobain. Personnel from CertainTeed and Saint Gobain experienced in the operation of wheel-type fiberizers observed the trial to assist in the fiberizer operation and to perform a preliminary evaluation of the physical characteristics of the fiber produced.

The feedstock used in the trial contained glass produced from SPL in a commercial CMS® (at Ormet Aluminum Corporation’s Hannibal, Ohio, primary aluminum smelting plant) instead of raw SPL waste. The reason for using the glass from Ormet was that a higher flow rate could be achieved in the pilot-scale
CMS® because the carbon in the SPL had been previously oxidized in the process at Ormet. A higher flowrate to the fiberizer was one of the primary recommendations of Saint Gobain. Additives such as soda ash and limestone were blended with the Ormet glass so that the glass produced in the CMS® would have a composition similar to the target composition for glass produced directly from SPL.

The as-received Ormet glass consisted of material approximately 1/8” in size. This material was pulverized to less than 300 microns in Vortec’s rotating ball mill at the test facility. The additives were purchased pre-sized to the criteria of the CMS®.

After comminution of the Ormet glass, it was put in FIBC’s and then loaded into Vortec’s pneumatic blend vessel using the facility’s 5-ton overhead crane. The additives were also loaded into the blend vessel from FIBC’s. The ingredients were then blended. About 8,000 lbs of feedstock was prepared for the fiberizing trial.

Start-up of the CMS® was initiated 12 hours before the vitrification/fiberizing trial. After the system refractories were heated to about 2,600°F, flow of feedstock to the CMS® was initiated and maintained at approximately 900 lbs/hr during the vitrification trial. After a stable stream of glass was flowing from the separator/reservoir component of the system, the fiberizing wheels were brought up to design speed and the wheels were brought into position under the glass stream. Minimal stripping air was introduced to the fiberizing wheels initially to allow time for the surface of the wheels to rapidly increase in temperature. After several minutes of operation, a thin layer of molten glass appeared on the surface of the wheels and the flow of stripping air to the wheels was increased. Prior to the formation of the layer of glass, most of the glass exited the fiberizer as “shot” – small pieces of molten glass, not fibers. After formation of the glass layer and increase of the stripping air to design flowrate, a high percentage of the glass introduced to the fiberizer exited as fiber. The CertainTeed and Saint Gobain representatives at the test indicated that the fibers appeared to be in the desired size range, predominantly 3 to 5 microns in size, and considered the test a significant success. Based on their observations of the pilot-scale fiberizer performance, they predicted that a commercial scale fiberizer should be able to produce the fibers for the glass at an efficiency approaching 80%. This is greater than the project goal of 60% fiberizing efficiency.

Fiberizing operations continued until sufficient quantities of fibers were collected for chemical composition, thermal performance, and solubility analyses. Total fiberizing time was on the order of one hour; however, total melting duration was approximately 8 hours during which time additional glass samples were taken for analysis.

**Trial #5 – SPL Vitrification Trial (Vortec Test #170)**

An additional SPL vitrification trial was performed during the second budget period to collect more flue gas characterization data via pilot-scale testing and to evaluate the performance of the woven stainless steel filters in removal of particulate from the flue gas at a gas temperature of about 600°F.

Approximately 3,040 lbs of feedstock containing SPL waste was vitrified over a two day period. The feedstock contained 58 wt.% SPL waste (1,100 lbs of first cut material and 600 lbs of second cut material) from Alumax Primary Aluminum Corporation’s Eastalco aluminum smelting plant in Frederick, Maryland. The feedstock also contained 42% wt.% low cost ($5 - $15/ton) additives.
The SPL waste received from Alumax consisted of material up to 4 inches in size. This material was crushed to approximately one-half inch in a shredder at Vortec’s test facility, ground to less than 100 microns in Vortec’s rotating ball mill, and loaded into FIBC’s. The additives were purchased pre-sized to the criteria of the CMS® and shipped to the test facility in FIBC’s. Immediately prior to the vitrification trial, the ground SPL waste and additives were loaded into Vortec’s pneumatic blend and feed vessel, using the facility’s 5-ton overhead crane, and blended.

Prior to the vitrification trial, Unisearch Associates, Inc. installed their near-infrared tunable diode laser absorption system (LasIR), shown in Figures 5.4-2 and 5.4-3, for on-line continuous measurement of HF concentration in the flue gas from the CMS®. Two measurement points were set up, one at the inlet to the evaporative cooler and one at the exit of the cooler. The flanged pipe sections to which the laser components were mounted were water cooled via a copper cooling coil around the external surface to minimize conductive heat transfer to the laser component. Instrument air was also used as purge air at the penetration of the flue gas duct where the laser component was mounted to prevent solids buildup on the laser “window”. A photograph of the duct transporting flue gas to the evaporative cooler showing the interfacing flange for the laser is presented in Figure 5.4-4.
(a) Enlargement of Monitor Showing HF Concentration in the Flue Gas

Figure 5.4-3  Computer Based HF Data Acquisition System in Pilot-Scale CMS® Facility

Laser Interfacing Flange

Figure 5.4-4  Flue Gas Duct to Evaporative Cooler With Laser Interfacing Flange
Another outside laboratory, Comprehensive Safety Compliance, Inc. (CSC) installed a flue gas sampling train in the ductwork upstream and downstream of the evaporative cooler for sampling and analyzing the flue gas, in accordance with USEPA Method 13B, for total fluorides. A water-cooled sampling probe was used upstream of the evaporative cooler where the gas temperature was on the order of 2,650°F. The sampling train at the discharge of the evaporative cooler was also used to sample and analyze the flue gas for SO₂, in accordance with EPA Method 6, and collecting flue gas particulate samples in accordance with EPA Method 5. Another sampling train was installed in the baghouse discharge duct to collect particulate samples for evaluating the efficiency of the baghouse.

Start-up of the CMS was initiated approximately 24 hours prior to the vitrification trial. After the system was stabilized at 2,600°F operating temperature (CRV reactor and cyclone melter discharge gas temperature), flow of feedstock to the CMS was initiated and maintained at approximately 300 lbs/hr for the duration of the vitrification trial. The flue gas temperature at the baghouse was maintained at approximately 600°F by controlling the water flowrate to the evaporative cooler.

The concentration of HF in the flue gas upstream and downstream of the evaporative cooler was measured continuously throughout the test. After approximately 5 hours of steady-state operation, the flue gas was sampled for particulates and analyzed for total fluorides and SO₂. The pressure differential across the stainless steel filters was approximately 6 in.w.g. at the time of sampling. Samples of the glass produced were also taken during the flue gas sampling period.

After a one-hour flue gas sampling period, the filters were cleaned of particulates via the air pulse system until the pressure differential across the filters dropped to zero, and samples of the particulate were then taken from the baghouse particulate hopper. Limestone was then introduced into the flue gas duct upstream of the baghouse to precoat the filters. Five-hundred pounds of limestone was introduced and the pressure differential across the filters at the end of the precoating period was about 2 in.w.g. During the precoating period, the system temperatures and feedstock flowrate were maintained at the test design conditions. Approximately one hour after completion of the precoating, the flue gas was again sampled for particulates upstream and downstream of the baghouse for a period of one hour. The pressure differential across the filters was approximately 3 in.w.g. during the sampling period.

5.4.3 Flue Gas Analysis from 1st and 2nd Budget Period Testing

SPL waste, glass, and particulate samples collected from the flue gas during the 2nd budget period SPL vitrification trial were sent to an outside laboratory, Galbraith Laboratories, Inc., for analysis with respect to concentrations of fluorides, silicon, aluminum, calcium, sodium, iron, and other metals of interest. Results of the analyses were used, along with the measured data from the vitrification trial, to develop a fluoride mass balance. The samples were analyzed for fluoride concentration via pyrohydrolysis. Additionally, particulate samples were sent to the New York State College of Ceramics at Alfred University for x-ray diffraction (XRD) analysis to identify specific fluoride compounds present. CSC calculated the particulate concentration in the flue gas based on the results of the flue gas sampling during the vitrification trial.
A mass balance based on the average of the data measured during the flue gas sampling period for the vitrification trial is presented in Figure 5.4-5. Also shown in the Figure is the mass balance for fluoride based on the measured HF data and data from the chemical analyses on the feedstock, glass, and flue gas particulate samples. Results of XRD of the particulates are presented in Figure 5.4-6.

The data indicate a good balance between the calculated quantity of fluorides entering the CMS® and leaving the evaporative cooler. Approximately 16% of the fluorides entering the CMS® in the feedstock partitions to the glass. On-line measurement of the HF indicates that of the 84% that partitions to the flue gas, about 80% is bound in HF before the evaporative cooler. As the flue gas is cooled in the evaporative cooler, some of the fluoride combines with other elements in the flue gas to produce compounds such as sodium fluoride (NaF), calcium fluoride (CaF₂), and cryolite (Na₃AlF₆) which condense and become solids in the flue gas stream (peaks shown in Figure 5.4-6). After cooling, about 36% of the fluoride in the flue gas is bound in HF, based on on-line measurement of the HF, and the remaining 64% is in solid fluorides. Therefore, about 29% of the fluoride introduced into the CMS® in the SPL waste is in the form of HF downstream of the evaporative cooler and 52% is in the form of solid fluorides.

The fluoride balance data is also summarized in Table 5.4-1 and compared to the data obtained during the first budget period SPL vitrification trials that were discussed in detail in the first budget period report, “CMS™ Technology for Converting SPL into Useful Glass Fiber Products Technical Progress Report,” October 31, 1998. During the first budget period trials (Trials #2 and #3), flue gas and particulate samples were taken and later analyzed with respect to fluorides and metals concentrations. On-line measurement of HF in the gas was not made during those trials.

The data indicate that the percentage of the fluoride entering the system that partitions to the glass and the portion of the fluoride that is in the form of HF in the flue gas stream is a function of the operating temperature. At the operating temperature required for the insulating glass fiber product (2,600°F), more of the fluoride entering the system is vaporized and partitions to the flue gas than would be the case with a lower operating temperature product. Additionally, less of the fluoride that partitions to the flue gas is in the form of HF at the higher operating temperature. This is probably because more of the sodium entering the system vaporizes and partitions to the flue gas at higher operating temperature; therefore, more is available to react with the fluoride when the flue gas is cooled and to condense as sodium fluoride. As discussed in the first budget year technical progress report, the data collected during Trial #3 did not yield a good fluoride mass balance. Therefore, fluoride partitioning among the effluent streams was not calculated for that trial.

The concentrations of particulates in the flue gas measured upstream and downstream of the baghouse in Trial #5 (Vortec Test #170) are presented in Table 5.4-2. The particulate removal efficiencies of the baghouse filters were as expected given the short duration of the test and limited amount of pulse cleaning performed. The particulate concentration downstream of the baghouse will likely satisfy the criteria for producing a high purity aluminum fluoride product from the HF in the flue gas. There was no apparent deterioration of the stainless steel mesh filters at the end of the vitrification trial.
Figure 5.4-5  Heat and Mass Balance for Vitrification Trial #5 (Vortec Test #170)

Figure 5.4-6  XRD Pattern for Particulate from Vitrification Trial #5 (Vortec Test #170)
### Table 5.4-1  Fluoride Balance for Pilot-Scale SPL Vitrification Trials

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</tbody>
</table>

### Table 5.4-2  Particulate Concentration Before and After Baghouse Filter Vitrification Trial (#170)

<table>
<thead>
<tr>
<th></th>
<th>Particulate Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( \Delta P = 3 ) in. w.g.</td>
</tr>
<tr>
<td>Filter Inlet</td>
<td>1.55 gr/scf</td>
</tr>
<tr>
<td>Filter Outlet</td>
<td>0.034 gr/scf</td>
</tr>
<tr>
<td>Efficiency</td>
<td>97.8%</td>
</tr>
</tbody>
</table>
5.4.4 3rd Budget Period Testing

**Trial #6 – SPL Vitrification Trial (Vortec Test #171)**

Trial #6 was the first of a series of eight SPL vitrification trials performed during the 3rd budget period to evaluate the effect of oxygen enrichment and baghouse dust recycling on fluoride partitioning to the flue gas in the form of HF. The objective of Trial #6 was to establish a baseline without oxygen enrichment and without baghouse dust recycling. The feedstock consisted of SPL waste from Ormet Primary Aluminum Corporation’s Hannibal, Ohio, plant, and glass forming additives. The SPL waste comprised 60% of the feedstock. Ormet SPL waste was selected for the 3rd budget period testing because it was the only source that Vortec could identify available within the project schedule.

Twenty-two thousand pounds (10,000 kg) of a mixture of 1st cut SPL waste and 2nd cut refractory material was received from Ormet. The as-received material had been sized to about 25% minus 170 U.S. mesh in Ormet’s SPL vitrification facility. Vortec further reduced the size of the material to about 75% minus 170 mesh in a ball mill located in Vortec’s test facility. The material was then loaded into Vortec’s pneumatic blend and storage vessel and blended to ensure that a homogeneous mixture of 1st and 2nd cut material was prepared. The material was then loaded into supersacks and a representative sample of material taken from each supersack. The sample from each supersack was then combined into a single composite sample and sent to Alcoa, Inc. for chemical composition analysis.

Immediately prior to Trial #6, approximately 6,300 lbs of the mixed SPL material was loaded into the feedstock storage and blending vessel in the test facility along with about 4,060 lbs of additives. The feedstock was then blended in preparation for injection into the CMS®. An additional 9,200 lbs of SPL and additives were loaded into the storage and blend vessel after the initial feedstock mixture had been processed. Approximately 3,700 lbs of the additional feedstock was process during Trial #6.

Prior to the vitrification trial, Vortec personnel installed the Unisearch Associates, Inc. near-infrared tunable diode laser absorption system (LasIR), for on-line continuous measurement of HF concentration in the flue gas from the CMS®, in a manner similar to that described for Trial #5. CSC installed a flue gas sampling train in the ductwork downstream of the evaporative cooler for sampling and analyzing the flue gas in accordance with USEPA Methods 1 through 5 for particulate concentration and USEPA Method 13B, for total fluorides.

Start-up of the CMS® was initiated approximately 24 hours prior to the vitrification trial. After the system was stabilized at 2,650°F operating temperature (cyclone melter discharge gas temperature), flow of feedstock to the CMS® was initiated on the morning of July 10, 2000. The feedrate was slowly increased to about 400 lb/hr over a period of about 3 hours, maintaining a design glass temperature in the cyclone melter of 2,550F, while checking the system for proper operation. At this time, the thermocouple measuring the gas temperature in the CRV reactor failed. Proper operation of this thermocouple is required for safe operation of the system. Therefore, the feed input to the system was temporarily terminated to replace the thermocouple. The feed input to the system was reinitiated after about one hour, and the feedrate again increased to 400 lb/hr over a period of about 2 hours. At this time, a leak developed in the gasket sealing the flange of the orifice plate in the reactor air supply pipe. Since the reactor air at this location in the supply pipe is at a temperature of about 1,000°F, shutdown of the system
was required to replace the gasket. Shutdown of the system, replacement of the gasket, and re-heating of the system back to operating temperature delayed continuation of the test until the morning of July 12.

On the morning of July 12, the feed input to the system was again initiated and slowly increased to about 590 lb/hr, maintaining a melter glass temperature of 2,550°F and an oxygen concentration in the flue gas of about 4%(v). This feedrate was maintained for about one-half hour, and the glass product was then visually inspected for oxidation state. At this flowrate, the glass appeared to be translucent, because of the presence of precipitates, indicating that the glass was excessively reduced. The feedrate was then reduced to about 516 lb/hr and maintained for one hour. The glass discharging from the CMS® appeared to be sufficiently oxidized at this feedstock flowrate. CSC then attempted to sample the flue gas for particulates and fluoride concentrations but had difficulties with their sampling equipment. Continuation of the trial was postponed until the morning of July 13, with the system maintained in a hot-idle condition (operating gas temperature with no feedstock input) overnight.

The trial was continued on the morning of July 13 with the initiation of feed input to the system and a gradual increasing of the feedrate to about 500 lb/hr, again at a design melter glass temperature of 2,550°F. Fluctuations in feedrate between 500 lb/hr and 600 lb/hr were encountered, which were eventually associated with refills of the feed hopper. Therefore, the feedrate was reduced to about 420 lb/hr to maintain peaks in the flowrate at or below about 515 lb/hr. A feedrate averaging about 420 lb/hr was maintained for about 5 hours. During this time, CSC performed flue gas sampling for analysis with respect to particulate and fluoride concentration. The sampling period was one hour. During the flue gas sampling period, feedstock and glass grab samples were collected at 15 minute intervals. A schematic of the pilot-scale system is presented in Figure 5.4.–7 showing the sampling locations for this and all subsequent trials. The samples were packaged separately for delivery to Alcoa for chemical composition analyses and leach testing with respect to fluoride and cyanide.

The total SPL processing duration in Trial #6 was about 32 hours over four days. Approximately 14,000 lbs of feedstock containing about 8,500 lbs of SPL was processed, producing about 9,100 lbs of glass.

**Trial #7 – SPL Vitrification Trial (Vortec Test #173)**

The objectives of Trial #7 were to determine the effect of baghouse dust recycling on HF concentration in the flue gas from the CMS® and to establish the size of the baghouse dust recycle stream at steady state conditions. The baghouse dust collected in Trial #6 was used in Trial #7.

A feed hopper and screw feed system for delivering the baghouse dust to the CMS® separate from the feedstock containing the SPL waste was installed. The trial was divided into three segments. During the first segment, the baghouse dust flowrate to the system would be 5% of the combined SPL feedstock and baghouse dust flowrate. During the second segment, the baghouse dust flowrate would be increased to 10% of the combined feed streams, and the baghouse dust flowrate would be increased to 10% of the combined feed streams for the third segment of the trial.

Trial #7 was performed the day after Trial #6 was complete, so the system was maintained at design operating temperature between the two trials. On the afternoon of July 14, 2000, flow of SPL containing feedstock was initiated and gradually increased to 360 lb/hr, maintaining a melter glass temperature of
2,550°F. Flow of baghouse dust to the process was then initiated at 18 lb/hr. After approximately one hour of operation at these flowrates, a blockage occurred in the baghouse dust supply line. The blockage was cleared, and feeding of baghouse dust to the process was restarted. Intermittent blockages of the supply line continued to occur as attempts to achieve a steady state operating condition were made. Additionally, a leak developed in the WESP water supply pump necessitating shutdown of the system. The test was postponed until the WESP pump could be repaired or replaced.

As a result of a post-test evaluation of the baghouse dust feeding problem, it was concluded that because of the low density of the dust (ca. 6 lb/cu.ft.), uninterrupted feeding of the dust with the existing feed system in the test facility was improbable. Results of further evaluations with respect to anticipated commercial feed system designs indicated that the baghouse dust would most likely be mixed with the SPL containing feedstock prior to introduction into the CMS® process. Therefore, a decision was made to change the baghouse dust feeding approach from separate injection to one of mixing the baghouse dust with the SPL feedstock in the test facility’s feedstock storage and blending system, and delivery of the combined material to the CMS® with the existing primary feedstock delivery system.
Trials #8 through #11 – SPL Vitrification Trial (Vortec Tests #175 through #178)

Trials 8 through 11 were performed on August 8 through 10, 2000, as follow-ups to Trial #7 to evaluate the effect of baghouse dust recycling on HF concentration in the flue gas from the CMS® and the size of the baghouse dust recycle stream at steady-state condition. For these trials, baghouse dust was mixed with SPL waste and additives in the test facility’s feedstock storage and blending vessel prior to delivery to the CMS®. Trial #8 was a repeat of the baseline with no baghouse dust recycle; that is, the feedstock to the CMS® contained only the SPL waste and additive mixture.

Baghouse dust collected from Trial #6 was used in Trial #9 and Trial #10. The dust was mixed with the SPL containing feedstock such that the baghouse dust was 5 wt.% of the total mixture in Trial #9 and 15 wt.% of the total mixture in Trial #10. Baghouse dust collected during Trial #10 was used in Trial #11 with the baghouse dust being 17 wt.% of the total feedstock delivered to the CMS®.

CSC performed flue gas sampling for analysis with respect to particulate and fluoride concentration during each of the trials. It was intended that the duration of each sampling period be one hour; however, the sampling periods for Trial #10 and Trial #11 were only 20 minutes and 25 minutes, respectively, because of rapid buildup of particulate on the filters during these two trials. Grab samples of the feedstock and glass produced during the flue gas sampling periods were collected at 15 minute intervals. The samples were packaged separately for delivery to Alcoa for analysis with respect to chemical composition. Splits of the glass samples collected during each trial were combined into a composite sample which was sent to CertainTeed Corporation for chemical composition analysis. Samples collected by CSC (particulate samples, filters used in the flue gas sampling, impinger solutions, and washdown solutions) were also sent to Alcoa for analysis with respect to fluoride and metals concentrations. A 100 lb sample of water-quenched glass collected during the trials was also sent to Alcoa for fluoride and cyanide leaching tests.

Trial #8 (Vortec Test #175)

Prior to vitrification Trial #8, Vortec personnel installed the Unisearch Associates, Inc. near-infrared tunable diode laser absorption system (LasIR), for on-line continuous measurement of HF concentration in the flue gas from the CMS®, in a manner similar to that described for Trial #5. CSC also installed a flue gas sampling train in the ductwork downstream of the evaporative cooler.

The CMS® test facility was used for a test not related to this project the morning of August 8, 2001. Therefore, the system was maintained at operating temperature following completion of the morning test until the feedstock storage and blend vessel was loaded with the feedstock ingredients for Trial #8. Approximately 1,500 lbs of SPL waste and glass forming additives were loaded into the feedstock storage and blending vessel and blended for the trial.

Feedstock to the CMS® was initiated in the afternoon of August 8 and gradually increased to approximately 360 lb/hr, maintaining a melter glass temperature of about 2,550°F and O₂ concentration in the flue gas in the range of 4%(v) to 5%(v). The design feedrate for the trial was defined based on maintaining the same throughput of SPL waste during all the trials (Trials #8 through #11) without risk of exceeding the maximum achievable throughput of feedstock based on glass oxidation state criteria. For example, the SPL waste throughput for Trial #8 with no baghouse dust in the feedstock would be about 220 lb/hr (360 lb/hr feedstock flowrate times 0.61, the fraction of SPL waste in the feedstock), and the
throughput of SPL waste for Trial #11 with 17% baghouse dust in the feedstock would also be 220 lb/hr although the total feedstock flowrate in Trial #11 would be about 435 lb/hr (220 lb/hr SPL waste + 141 lb/hr glass forming additives + 74 lb/hr baghouse dust).

After approximately 2 hours of vitrification operation, CSC initiated flue gas sampling for analysis of particulate and fluoride concentrations and to collect a particulate sample for analysis with respect to chemical composition. The flue gas and particulate sampling period was about 1 hour. All 1,500 lbs of feedstock was processed during Trial #8, producing about 1,000 lbs of glass product, over a period of about 4.5 hours. At the completion of Trial #8, the system was maintained at design operating temperature overnight in preparation for Trial #9 on the following day.

**Trial #9 (Vortec Test #176)**

Approximately 1,660 lbs of feedstock, containing about 5 wt.% baghouse dust from Trial #6, was loaded into the feedstock storage and blending vessel and blended for Trial #9. Feedstock flow to the CMS® was initiated on the morning of August 9, 2001, and gradually increased to about 380 lb/hr, maintaining the design melter glass temperature of about 2,550°F and O₂ concentration in the flue gas in the range of 4%(v) to 5%(v). After about 2 hours of operation, CSC initiated flue gas sampling for analysis with respect to particulate and fluoride concentration, and particulate sampling for analysis with respect to chemical composition. The flue gas was sampled for a period of 1 hour. All of the 1,660 lbs of feedstock was processed, producing about 1,070 lbs of glass over a period of about 4.5 hours.

**Trial #10 (Vortec Test #177)**

Approximately 1,800 lbs of feedstock, containing about 15 wt.% baghouse dust from Trial #6, was loaded into the feedstock storage and blending vessel immediately following Trial #9 and blended for Trial #10. Feedstock flow to the CMS® was initiated on the evening of August 9, 2001, and gradually increased to about 420 lb/hr, maintaining the design melter glass temperature of about 2,550°F. After about 3 hours of operation, CSC initiated flue gas sampling for analysis with respect to particulate and fluoride concentration, and particulate sampling for analysis with respect to chemical composition. Flue gas sampling was terminated after 20 minutes because the maximum collection capacity of the filters in the Method 5 and Method 13 sampling trains was reached. All of the 1,800 lbs of feedstock was processed, producing about 1,135 lbs of glass. The system was maintained at design operating temperature overnight in preparation for Trial #11 to be performed the following day.

**Trial #11 (Vortec Test #178)**

Approximately 1,280 lbs of feedstock, containing about 17 wt.% baghouse dust from Trial #10, was loaded into the feedstock storage and blending vessel and blended for Trial #11. Feedstock flow to the CMS® was initiated on the afternoon of August 10, 2001, and gradually increased to about 435 lb/hr, maintaining the design melter glass temperature of about 2,550°F and O₂ concentration in the flue gas in the range of 4%(v) to 5%(v). After about 1.5 hours of operation, CSC initiated flue gas sampling for analysis with respect to particulate and fluoride concentration, and particulate sampling for analysis with respect to chemical composition. Flue gas sampling was terminated after 25 minutes because the maximum collection capacity of the filters in the Method 5 and Method 13 sampling trains was reached. All of the 1,280 lbs of feedstock was processed, producing about 700 lbs of glass.
Trials #12 and #13 – SPL Vitrification Trials (Vortec Tests #179 and #180)

Trials #12 and #13 were performed from August 29 through September 1, 2000, to evaluate the effect of oxygen enhancement of the CMS® combined with baghouse dust recycling on HF partitioning to the flue gas and the HF concentration in the flue gas from the CMS®. For these trials, baghouse dust was mixed with SPL waste and additives in the test facility’s feedstock storage and blending vessel prior to delivery to the CMS®. Trial #12 was performed with and without oxygen enhancement with no baghouse dust recycle; that is, the feedstock to the CMS® contained only the SPL waste and additive mixture. Trial #13 was performed with oxygen enhancement and with baghouse dust recycle.

CSC performed flue gas sampling for analysis with respect to particulate and fluoride concentration during each of the trials. Two samples were taken during Trial #12, one during performance without oxygen enhancement and one during performance with oxygen enhancement. The duration of each sampling period was one hour. Two flue gas samples were also taken during Trial #13, one at a moderate feedstock flowrate and one at a high feedstock flowrate. The durations of these samples were only 25 to 30 minutes because of rapid buildup of particulate on the filters. Grab samples of the feedstock and glass produced during the flue gas sampling periods were collected at 15 minute intervals. The samples were packaged separately for delivery to Alcoa for analysis with respect to chemical composition. Samples collected by CSC (particulate samples, filters used in the flue gas sampling, impinger solutions, and washdown solutions) were also sent to Alcoa for analysis with respect to fluoride and metals concentrations. A 100 lb sample of water-quenched glass collected during the trials was also sent to Alcoa for fluoride and cyanide leaching tests.

Trial #12 (Vortec Test #179)

Prior to vitrification Trial #12, Vortec personnel installed the Unisearch Associates, Inc. near-infrared tunable diode laser absorption system (LasIR), for on-line continuous measurement of HF concentration in the flue gas from the CMS®, in a manner similar to that described for Trial #5. CSC also installed a flue gas sampling train in the ductwork downstream of the evaporative cooler.

Heat-up of the CMS® process was initiated on August 28, 2000, approximately 24 hours prior to the planned commencement of Trial #12. During the heat-up period, approximately 7,900 lbs of feedstock consisting of 63% combined 1st cut SPL and 2nd cut refractory material and 37% additives was loaded into and blended in the facility feedstock storage and pneumatically blending vessel.

Feedstock to the CMS® was initiated in the afternoon of August 29 and gradually increased to approximately 490 lb/hr, maintaining a melter glass temperature of about 2,575°F and O₂ concentration in the flue gas of about 3% (v). After approximately 2 hours of vitrification operation, CSC initiated flue gas sampling for analysis of particulate and fluoride concentrations and to collect a particulate sample for analysis with respect to chemical composition. The flue gas sampling period was about 1 hour.

After completion of the first flue gas sampling period, oxygen flow to the CMS® was initiated and slowly increased, along with a simultaneous increase in feedstock flowrate, until an oxygen enhancement level (oxygen concentration in the oxidant to the process) of 31 wt.% and a nominal feedstock flowrate of 700 lb/hr were achieved. This represents an increase in feedstock throughput of 40% over that achieved
without oxygen enhancement. The O\textsubscript{2} concentration in the flue gas at this point was about 8%(v). An attempt was made to adjust the air and oxygen flowrates to the process to reduce the O\textsubscript{2} concentration in the flue gas; however, difficulties were encountered in controlling the gas temperatures at the top of the CRV reactor and exit of the cyclone melter within acceptable limits. Therefore, the 8%(v) O\textsubscript{2} concentration condition was maintained due to the limited time remaining to complete this portion of the testing project. After about 2.5 hours of operation, CSC initiated flue gas sampling for analysis with respect to particulate and fluoride concentrations. The flue gas sampling period was about 1 hour. After the flue gas sampling was complete, feedstock flow to the system was terminated and the system maintained at operating temperature overnight for the start of Trial #13 on September 29, 2000. A total of 4,411 lbs of feedstock material was process during Trial #13.

**Trial #13 (Vortec Test #180)**

Approximately 3,600 lbs of feedstock, containing 17 wt.% baghouse dust from Trials #6, #9, and #11 was blended in the feedstock storage and blending vessel for Trial #13. Feedstock flow to the CMS\textsuperscript{®} was initiated on the morning of August 31, 2001, and gradually increased to about 500 lb/hr, maintaining the design melter glass temperature of about 2,550°F. The O\textsubscript{2} concentration in the flue gas was about 6%(v). After about 1.5 hours of operation, feedstock flow to the CMS\textsuperscript{®} was interrupted, due to blockage in the injector assembly. The blockage was cleared and the feed to the CMS\textsuperscript{®} re-initiated. Feedstock flow to the CMS\textsuperscript{®} was again interrupted by a blockage in the injector assembly after about 1.5 hours of operation. The blockage was again cleared and feedstock flow re-initiated. This sequence of events occurred periodically throughout the day of August 31. The system was shutdown early in the evening of August 31 to remove the injector from the CMS\textsuperscript{®} for an inspection to determine the cause of the blockages. The injector was found to have a large build up of solid material at the exit of the injector which blocked the flow of feedstock to the CMS\textsuperscript{®}. The injector was cleaned and reinstalled into the top of the CRV reactor for continuation of the test on September 1, 2001.

Based on the difficulties encountered on August 31, 2001, a decision was made to establish a feedstock flow rate condition initially on September 1 that could confidently be maintained without interruption for a sufficient duration to perform flue gas sampling for particulate and fluoride concentration analyses. After completion of flue gas sampling, the feedstock flow rate would be increased to the maximum achievable based on operating temperatures, glass oxidation state, or the ability to maintain feedstock flow without interruption. Another flue gas sample would then be taken at the maximum feedstock flow rate condition.

Feedstock flow to the CMS\textsuperscript{®} was initiated in the morning of September 1 and was gradually increased to about 425 lb/hr, while maintaining design operating temperature and oxidation conditions. These conditions were maintained for approximately 1 hour at which time flue gas sampling by CSC was initiated. Flue gas sampling was terminated after 30 minutes because the particulate filter in each sampling train (the Method 5 and Method 13B sampling trains) became saturated.

After completion of the flue gas sampling, the feedstock flow to the CMS\textsuperscript{®} was gradually increased to about 680 lb/hr while maintaining design operating temperature conditions. These conditions were maintained for about 1 hour, at which time flue gas sampling by CSC was again initiated. Flue gas sampling was terminated after 25 minutes because the particulate filter in each sampling train became saturated.
At the conclusion of Trial #13, there was insufficient SPL waste to continue testing. Therefore, the pilot-scale testing to evaluate the effect of staged injection of air into the CMS® process on NOₓ emissions could not be performed.

5.4.5 Flue Gas Analysis from 3rd Budget Period Testing

Over 60 SPL waste, feedstock, glass, and particulate samples collected from the flue gas during the 3rd budget period SPL vitrification trials were sent to Alcoa for analysis with respect to chemical composition and leaching characteristics. The sampling and analysis procedures are presented in Table 5.4-3. Results of the analyses were used, along with the measured data from the vitrification trial, to develop mass balances, determine the partitioning of fluoride among the effluent streams, and determine the effect of baghouse dust recycle and oxygen enhancement on HF concentration in the flue gas.

<table>
<thead>
<tr>
<th>Sampling/Analysis</th>
<th>Procedure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particulate Emissions</td>
<td>EPA Method 5</td>
</tr>
<tr>
<td>Fluoride Emissions</td>
<td>Modified EPA Method 13B &amp; Laser Spectroscopy</td>
</tr>
<tr>
<td>Flue Gas Analysis for O₂, CO₂, H₂O</td>
<td>EPA Methods 3 &amp; 4</td>
</tr>
<tr>
<td>Flue Gas Analysis for O₃, CO, and NOₓ</td>
<td>CEM – Beckman Analyzers</td>
</tr>
<tr>
<td>Metals Analysis in Solids</td>
<td>ICP Spectroscopy</td>
</tr>
<tr>
<td>Fluoride Analysis in Solids</td>
<td>Acid Distillation/ISE</td>
</tr>
<tr>
<td>Fluoride Leaching Analysis</td>
<td>EPA TCLP with De-ionized Water as Solution</td>
</tr>
</tbody>
</table>

The partitioning of the fluoride introduced into the process in the SPL to the glass and to the flue gas as HF during the 3rd budget period trials is illustrated in Figure 5.4.-8. For the trials with no baghouse dust recycle (Trials #171, 175, and #179), about 10% of the fluoride in the SPL partitioned to the glass and about 30% partitioned to the flue gas as HF. The remaining 70% partitioned to the flue gas as solid fluoride salts. As the fluoride laden particulate that was collected in the baghouse was added into the feedstock, the amount of fluoride partitioning to the flue gas as HF increased. When the concentration of baghouse dust in the feedstock was in the 16% to 17% range (Trials #178 and #180), about 76% of the fluoride in the SPL partitioned to the flue gas as HF, a factor of 2.5 greater than that observed without baghouse dust recycle.
During Trials #178 and #180, the amount of fluoride partitioning to the glass plus the amount partitioning to the flue gas as HF was about equal to the amount introduced into the process in the SPL. This indicates that the process is at or very near steady-state baghouse dust recycle conditions during these trials, as illustrated in Figure 5.4-9. At steady-state recycle conditions, all the fluoride introduced into the process from outside the process boundaries comes from the SPL, and all the fluoride escaping the process is either in the glass or in the flue gas as gaseous HF. Essentially all of the particulate with the solid phase fluoride is recycled within the process boundaries. Similarly, all the sodium introduced into the process is contained in the SPL under steady-state recycling conditions. At the temperature of the flue gas downstream of the evaporative cooler, essentially all of the sodium compounds are in the solid phase. Therefore, all the sodium either leaves the process in the glass or is recycled as particulate, and the amount of sodium in the glass equals the amount of sodium in the SPL.

The amount of sodium in the glass relative to the quantity introduced into the process in the SPL during the 3rd budget period trials is shown in Figure 5.4-10. These data show that the amount of sodium in the glass is about the same as the quantity in the SPL when 16% to 18% baghouse dust is in the feedstock. Another indication of steady-state baghouse dust recycle condition is when the amount of particulate in the flue gas relative to the feedstock flowrate is equal to the fraction of baghouse dust incorporated into the feedstock. The data presented in Figure 5.4-11 indicate that Trials #178 and #180 were close to this condition.
Figure 5.4-9  Process Schematic With Baghouse Dust Recycling

Figure 5.4-10  Sodium in Glass Relative to Sodium in SPL Versus Baghouse Dust in Feedstock
Figure 5.4-11  Particulate in Flue Gas Versus Baghouse Dust in Feedstock

The concentrations of HF in the flue gas after the evaporative cooler based on data from the flue gas sampling during the 3rd budget period trials are presented in Figures 5.4-12 and 5.4-13. The data in Figure 5.4-12 are based on the flue gas flow rate (wet) as measured via EPA Methods 2 through 4. The process was maintained at a slightly negative pressure relative to ambient during the trials to prevent fugitive gas emissions. This results in some infiltration air through openings around instrumentation and burners, and through the glass discharge tap hole when the glass does not completely fill the opening. The amount of infiltration air is estimated from the difference between the O\(_2\) concentration measured in the flue gas via flue gas sampling and the O\(_2\) concentration calculated based on the measured flowrates and composition of the air, natural gas, feedstock, and water input streams to the process. A commercial system would be designed to minimize or eliminate the air infiltration; therefore, an HF concentration in the flue gas flow without infiltration air was calculated and is presented in Figure 5.4-13. The data in Figures 5.4-12 and 5.4-13 indicate that the HF concentration increased by a factor of about 2.5 as a result of baghouse dust recycling. Additionally, they indicate that the HF concentration was increased by about 25% with enhancement of the oxidant to 31 wt.% oxygen. Adjustment of the air and oxygen to the process to decrease the O\(_2\) concentration in the flue gas to the level observed in the trials without oxygen enhancement should further increase the HF concentration in the flue gas.
Figure 5.4-12  HF Concentration in Flue Gas Based on Method 13B Data Versus Baghouse Dust in Feedstock

Figure 5.4-13  HF Concentration in Flue Gas After Adjustment for Process Infiltration Air
5.4.6 Glass Fluoride Leaching and Cyanide Analysis from 3rd Budget Period Testing

Glass samples taken during the 3rd budget period trials were subjected to a toxicity characteristic leaching procedure by Alcoa to evaluate the effect of baghouse dust recycling on fluoride leaching. The leaching procedure used was similar to the U.S. EPA TCLP (U.S. Environmental Protection Agency, 1996, Test Methods for Evaluating Solid Waste, SW-846, 3rd ed., Office of Solid Waste and Emergency Response, Washington, DC.) except that de-ionized water was used as the solution based on recommendations by the EPA (Federal Register/Vol. 65, No. 134/ Wednesday, July 12, 2000/Proposed Rules, 40 CFR Parts 260, 261, 268, and 271). Results of the leaching analyses on the glass samples are presented in Table 5.4-4. The total fluoride concentration in the feedstock and glass are also included in the table for reference.

Table 5.4-4 Results of Fluoride Leaching Tests Performed on Glass Samples

<table>
<thead>
<tr>
<th>Trial Number</th>
<th>Baghouse Dust In Feedstock</th>
<th>Fluoride in Leachate from Leaching Procedure(1), mg/L</th>
<th>Total Fluoride in Glass, mg/kg</th>
<th>Total Fluoride in Feedstock to Process, mg/kg</th>
</tr>
</thead>
<tbody>
<tr>
<td>171</td>
<td>0</td>
<td>0.370</td>
<td>7,900</td>
<td>55,000</td>
</tr>
<tr>
<td>175</td>
<td>0</td>
<td>1.25</td>
<td>8,600</td>
<td>ND(2)</td>
</tr>
<tr>
<td>176</td>
<td>5%</td>
<td>1.02</td>
<td>11,200</td>
<td>ND</td>
</tr>
<tr>
<td>177</td>
<td>13%</td>
<td>0.770</td>
<td>15,500</td>
<td>86,000</td>
</tr>
<tr>
<td>178</td>
<td>17%</td>
<td>0.740</td>
<td>17,100</td>
<td>99,000</td>
</tr>
<tr>
<td>179</td>
<td>0</td>
<td>0.777 – 2.61</td>
<td>11,000 – 13,000</td>
<td>63,000 – 64,000</td>
</tr>
<tr>
<td>180</td>
<td>17%</td>
<td>1.53 – 4.20</td>
<td>22,000 – 26,000</td>
<td>76,000 – 93,000</td>
</tr>
</tbody>
</table>

(1) U.S. EPA TCLP with de-ionized water as solution  
(2) ND = Not Determined

The total concentration of the fluoride in the glass product was a factor of 3.5 to 7 lower than the fluoride concentration in the feedstock to the process. The fluoride concentration in the glass increased with increased concentration of baghouse dust in the feedstock. The fluoride concentration in the leachate from the leaching procedure also tended to increase with the fluoride concentration in the glass and baghouse dust concentration in the feedstock. The highest concentration of fluoride in the leachate observed of 4.20 mg/L is an order of magnitude lower than the 48 mg/L limit specified in the 1996 EPA Land Disposal Restriction (LDR) standard, but higher than the 2.7 mg/L proposed by the EPA in Federal Register/Vol. 65, No. 134/ Wednesday, July 12, 2000/Proposed Rules, 40 CFR Parts 260, 261, 268, and 271. The 4.20 mg/L fluoride concentration in the leachate is also two orders of magnitude lower than that reported by the EPA in Federal Register/Vol. 65, No. 134/ Wednesday, July 12, 2000/Proposed Rules, 40 CFR Parts 260, 261, 268, and 271, Table 1, as typical of the residue from the Reynold’s SPL treatment facility.

Samples of the glass and feedstock taken during the 3rd budget period trials were also analyzed by Alcoa for total cyanide concentration. The results are presented in Table 5.4-5. The total cyanide concentrations in the glass samples were typically more than an order of magnitude lower than the 1996 EPA Land Disposal Restriction (LDR) standard and 2000 proposed standard of 1.4 mg/kg. They were

Table 5.4-5 Results of Glass Analysis for Total Cyanide Concentration
also more than a factor of three lower than that reported by the EPA in Federal Register/Vol. 65, No. 134/ Wednesday, July 12, 2000/Proposed Rules, 40 CFR Parts 260, 261, 268, and 271, Table 1, as typical of the residue from the Reymold’s SPL treatment facility.

5.4.7 4th Budget Period Testing

Trials #14 - #17 – SPL Vitrification Trials (Vortec Tests #184 and #186 to #188)

Trials #14, #15, #16, and #17 were performed on October 4, 2001 (Trial #14) and from December 11 through 13, 2001. The objectives were to correct problems encountered in Test #180 and obtain additional experimental data necessary to evaluate the effect of oxygen enhancement combined with baghouse dust recycling on HF partitioning to the flue gas and the HF concentration in the flue gas.

The CMS® was reconfigured for Trial #14 to eliminate the CRV reactor lid burner and close couple the top of the reactor with the inlet arm section (main burner section). The purpose of this modification was to reduce the structural heat loss in the top of the reactor, allowing for removal of the natural gas/air burner at the top and reducing the flue gas volume flowrate. Insulation was also added between the internal refractory liner and steel shell of the CRV reactor and cyclone melter based on results of refractory optimization investigations on the Vortec CMS® at Ormet Primary Aluminum Corporation.

During Trial #14, oxygen was injected separately into the system to increase the oxygen concentration of the total oxidant stream to 40 wt.%. The change in configuration eliminated the buildup of feedstock material in the top of the reactor that resulted in limited testing during Trial #13; although the temperatures measured by thermocouples in the reactor lid were about 500°F lower than those measured during previous tests. It is unknown if the lower measured temperatures were indicative of an actual reduction in gas temperature in the top of the reactor or the result of glass formation and coating of the surface of the thermocouples, thus masking the true gas temperature, because of a higher gas temperature than the previous test.

HF concentrations in the flue gas as high as 1.2% were observed until the high dust loading and HF concentration in the flue gas (due to the reduced flue gas flowrate made possible by the oxygen enhancement) inhibited measurement by the tunable-diode laser (TDL) spectroscopy system leased from Unisearch Associates, Inc (LasIR system). Results of analyses on glass samples taken during the trial indicated that the fluoride concentration was higher than observed during previous testing. There was concern that this may have resulted from reduced volatization of fluoride in the feedstock due to a lower gas temperature in the top of the CRV reactor. As a result, the top of the CRV reactor was modified to
incorporate an oxy-fuel burner to provide the ability to increase the temperature at the top of the reactor without increasing flue gas flowrate. A portion of the natural gas introduced into the CRV reactor inlet arms and some of the oxygen separately injected into the process would be diverted to the oxy-fuel burner. Modifications were also made to the location and installation of the LasIR instrumentation to mitigate the problems caused by high dust loading and HF concentration in Test #14, and Alcoa installed an FTIR analyzer to measure HF concentration in the flue gas.

Tests #15, #16, and #17 were performed with the modifications identified above to provide additional experimental data at design conditions and to investigate the impact of gas temperature at the top of the CRV reactor on fluoride partitioning to the glass and flue gas. For these trials, baghouse dust was mixed with SPL waste and additives in the test facility’s feedstock storage and blending vessel prior to delivery to the CMS®. CSC performed flue gas sampling for analysis with respect to particulate and fluoride concentration during each of the trials.

**Trial #14 (Vortec Test #184)**

Prior to Trial #14, the upper section of the CRV reactor was removed and the lid close coupled to the inlet arm section. The natural gas/air burner in the lid was also removed and replaced with a combined feedstock and oxygen injector. The LasIR instrumentation was installed in the flue gas ductwork downstream of the evaporative cooler (before the baghouse). CSC also installed a flue gas sampling train in the ductwork for sampling in accordance with EPA Methods 1 through 5 (for particulate concentration measurement) and modified EPA Method 13B for fluoride concentration measurement. The Method 13B was modified to use a 0.1N sodium hydroxide solution instead of deionized water in the impinger. This modification was specified because of the anticipated high concentration of HF in the flue gas.

Feedstock to the CMS® was initiated on October 4, 2001, and gradually increased to approximately 600 lb/hr. The flue gas flowrate observed was about one-fourth that of previous tests. About one-half of the reduction was attributed to the addition of insulation in the CRV reactor and cyclone melter components and one-half to oxygen enhancement.

The laser spectrometry system for analyzing the HF concentration in the flue gas at the exit of the evaporative cooler was operating satisfactorily for the first 2.5 hours of the test, before the feedstock flowrate reached its maximum, steady-state condition. After this time, the high concentration of particulate and HF in the flue gas resulting from the higher feedstock flowrate, at the reduced flue gas flowrate, decreased the laser signal power at the detector leading to a low signal error at the analyzer. Attempts were made to increase the signal at the analyzer by narrowing the focus of the laser beam. However, with the narrower focus, the laser beam could not be aligned at the detector. Therefore, analysis of the HF concentration at the steady-state feedstock flowrate conditions using the laser spectrometry system was not possible. HF concentrations as high as 1.2% (v) were observed, but the concentration was still increasing at the time the laser signal was too low for accurate HF measurement. After approximately 4 hours of vitrification operation, CSC initiated flue gas sampling for analysis of particulate and fluoride concentrations and to collect a particulate sample for analysis with respect to chemical composition. The flue gas sampling period was about 45 minutes. During this time, feedstock and glass samples were taken at 15 minute intervals for later analysis with respect to chemical composition and leaching characteristics. O₂, CO, and NOₓ concentrations were measured continuously throughout the test using on-line Beckman analyzers in the facility.
A total of about 9.5 hours of vitrification operation was performed on October 4 processing about 5,100 lbs of feedstock containing 2,850 lbs of SPL. An inspection of the refractory surface at the top of the CRV reactor after the test showed a light coating of glassy material but no agglomeration of material on the injector as was observed at the end of Trial #13 in September 2000. Therefore, the change in component configuration made for this test appeared to have eliminated the agglomeration problem.

CSC provided Vortec with the results of the particulate analysis data on the morning of October 5. The measured flue gas particulate flowrate was lower than anticipated (45 lb/hr versus 79 lb/hr), but it was suspected that a significant amount of particulate could be “dropping out” in the ductwork and evaporative cooler prior to the flue gas sampling point due to the low velocities in the duct resulting from operation with oxygen enhancement. An inspection of the duct at the discharge of the evaporative cooler after the system was shut down showed a significant amount of solid material in the duct. This material appeared to be predominantly a white powder, with some glassy phase material and rust.

The gas temperatures in the CMS® were typically on the order of 1,900°F at the very top of the CRV reactor, 2,300°F about midway between the top of the CRV reactor and the cyclone melter, and 2,500°F at the exit of the cyclone melter. This type of temperature distribution in the CMS® is to be expected since a major portion of the energy input to the system is from the carbon in the feedstock (about 60%), requiring more residence time to oxidize than the natural gas. However, the temperature measured by the thermocouples located at the top of the CRV reactor was about 500°F lower than that measured during previous testing. It is unknown if the lower measured temperatures were indicative of an actual reduction in gas temperature in the top of the reactor caused by the change in configuration, or if the gas temperature was actually the same or higher than in previous testing and the lower measured temperature was the result of glass formation and coating of the surface of the thermocouple. However, subsequent analysis of glass samples from the test showed a higher than anticipated fluoride concentration in the glass. This raised some concerns that the temperature may be lower resulting in less volatilization of fluoride and partitioning of more fluoride to the glass. Therefore, Vortec recommended the addition of an oxy-fuel burner in the top of the CRV reactor to provide additional heat input to this area. A portion of the natural gas introduced into the reactor through the inlet arms and a portion of the oxygen injected into the system could be diverted to the oxy-fuel burner. This would provide additional heat to the top of the reactor without increasing flue gas flowrate, which would decrease HF concentration in the flue gas. Vortec also recommended that the following modifications be made prior to continuing testing:

1. Move the LasIR instrumentation downstream of the baghouse to reduce the particulate concentration in the flue gas stream at the point of HF concentration measurement;
2. Decrease the diameter of the flue gas ductwork in which the LasIR instrumentation would be installed to reduce the laser path length;
3. Decrease the diameter of the refractory-lined flue gas duct between the CMS® and the evaporative cooler to increase the velocity of the flue gas at the low flue gas flowrates to ensure that the particulate remain entrained in the flue gas.

Alcoa approved of the above modifications and additional pilot-scale testing.

Trial #15 (Vortec Test #186)

The modifications recommended based on results of Trial #14 were made and Alcoa also installed an FTIR analyzer downstream of the evaporative cooler to measure HF concentration in the flue gas.
Trial #15 was initiated on December 11, 2001, and was approximately 11 hours in duration. Approximately 4,990 lbs of feedstock was processed. The feedstock included 55% combined 1st and 2nd cut SPL and 16% baghouse dust from the Ormet SPL vitrification system. Plots of the HF concentration measured by the LaSiR instrumentation and FTIR analyzer versus time are shown in Figure 5.4.7-1, along with the feedstock flowrate. The FTIR measured HF concentration before the baghouse (after the evaporative cooler), and the LaSiR measured HF concentration after the baghouse (but before the WESP). Alcoa had some difficulties with the FTIR instrumentation setup during Trial #15, so data were collected via this instrumentation for only about 4 hours of the test.

Feedstock flow was initiated at a rate of about 200 lb/hr. The feedstock flowrate to the system was gradually increased while natural gas, oxygen, and air flows to the system were adjusted to maintain the system temperatures generally in the range from 2,400°F to 2,500°F with an oxygen enhancement level of about 40 wt.%. The oxygen flowrate to the system was established based on maintaining 20% excess oxygen by weight over that required to theoretically convert all of the carbon introduced into the system with the SPL to CO$_2$. The air flowrate was initially established based on an amount 20% in excess over that theoretically required for complete combustion of the natural gas introduced into the system. After achieving the test design feedstock flowrate (about 500 lb/hr), the air flow to the system was reduced to a level that would maintain a minimum oxygen concentration in the flue gas of about 3%(v) at the exit of the separator/reservoir. The average oxygen concentration over the test was significantly higher [ca. 10%(v)] in order to maintain the minimum concentration during fluctuations in the feedstock flow. The existing screw feeder in the feedstock delivery subsystem is optimum for higher feedstock flowrates.
(1,000 to 2,000 lb/hr) and denser material typical of most tests in the facility. As a result, at the low flowrates (400 to 500 lb/hr) required when processing high carbon containing feedstocks like SPL, feedstock flow fluctuations of ±100 lb/hr are common.

The rate of reaction between the carbon (in the SPL) and the oxygen introduced in the top of the CRV reactor was not rapid enough to maintain the temperature in the top section of the CRV reactor at the desired level (ca. 2,400°F) without exceeding the upper temperature limit criteria at the exit of the cyclone melter (ca. 2,600°F), Figure 5.4.7-2. Therefore, supplemental natural gas was introduced into the CRV reactor inlet arms and oxy-fuel burner to maintain the gas temperature at the top of the CRV reactor at the desired level. In order to maintain as high an oxygen-to-carbon ratio as possible at the top of the CRV reactor where the feedstock was injected, the amount of natural gas introduced into the oxy-fuel burner was the minimum required to maintain the top of the CRV reactor at the desired temperature.

![Figure 5.4.7-2 Trial #15 System Gas Temperatures Versus Time](image-url)

The amount of feedstock introduced into the system was established based on the maximum that could be introduced and still maintain the gas temperature at the exit of the cyclone melter and separator/reservoir below the upper limit criteria. The feedrate was about 500 lb/hr on average. With an optimized feedstock
delivery system reducing the fluctuations in feedstock flowrate, a higher feedstock flowrate may have been achievable, possibly on the order of 600 lb/hr.

Once the test design conditions were established, the conditions were generally maintained for the entire test. After about 3.5 hours of steady state operation, flue gas sampling in accordance with EPA Methods was initiated by CSC. The flue gas was sampled between the evaporative cooler and baghouse for analysis with respect to particulate, in accordance with EPA Method 5, and total fluoride concentrations in accordance with a modified EPA Method 13B. Method 13B was modified in that the impinger solution was a 1N sodium hydroxide solution instead of deionized water. The concentration of sodium hydroxide was increased relative to Trial #14 because it was found at the end of Trial #14 that the solution was saturated with HF. The flue gas was also sampled at the exit of the baghouse for analysis with respect to total fluoride concentrations. Two flue gas samples were taken at each location, about 1.5 hours apart, each with a duration of about 1 hour. Typically, the quantity of particulate captured on the filter of the Method 5 sampling train is not sufficient to perform chemical composition analyses. Therefore, a “thimble” capable of capturing 1 to 2 grams of particulate was inserted into the gas stream at the same time and location (between the evaporative cooler and baghouse) as the Method 5 sampling probe to capture enough particulate for chemical composition analyses.

Just prior to each sampling period, automatic control of the gas temperature at the exit of the evaporative cooler was terminated and water introduction into the evaporative cooler manually prohibited. There is always some atomizing air introduced into the evaporative cooler through the lance (about 150 lb/hr) even when no water is introduced. The structural heat losses from the system components between the separator/reservoir and the evaporative cooler and the heat absorbing capacity of the atomizing air were sufficient to cool the gas to close to 700°F, the test design condition, by the time the gas reached the evaporative cooler exit. This resulted in significant fluctuations in flue gas flow and HF concentration in the flue gas. Therefore, water introduction into the evaporative cooler was suspended to minimize flue gas flow, particulate concentration, and fluoride concentration fluctuations during the gas sampling periods. The increase in flue gas temperature at the exit of the evaporative cooler was less than 50°F during each one-hour sampling period.

Samples of the feedstock were taken from the feed hopper via a thief at one-hour intervals throughout the vitrification period, except during the two one-hour flue gas sampling periods. Samples of the molten glass discharging from the separator/reservoir and glass frit from the glass quench tank were taken at one-half hour intervals throughout the vitrification period, except during the flue gas sampling periods. During the flue gas sampling periods, feedstock and glass samples were taken at 15 minute intervals. All samples, including those from the EPA Methods flue gas sampling, were given to Alcoa at the end of the three days of testing for analyses.

At the end of the test, particulate removed from the flue gas in the baghouse was collected for incorporation into the feedstock for Trial #16.

Trial #16 (Vortec Test #187)  

Vitrification Trial #16 was performed on December 12, 2002, and was approximately 9 hours in duration. Approximately 3,950 lbs of feedstock was processed. The feedstock formulation was the same as used in Trial #15 except that the baghouse dust used in Trial #16 was that generated during Trial #15.
Plots of system gas temperatures versus time are presented in Figure 5.4.7-3, and plots of feedstock flowrate and HF concentrations before and after the baghouse are shown in Figure 5.4.7-4. Feedstock flow was initiated at a rate of about 200 lb/hr. The feedstock flow to the system was gradually increased while natural gas, oxygen, and air flows to the system were adjusted to maintain the system temperatures generally in the range from 2,400°F to 2,500°F with an oxygen enhancement level of about 40 wt.%. The procedures for establishing the flow rates and temperatures were the same as those described for Trial #15. The steady-state conditions were generally the same as those maintained in Trial #15, as was the HF concentration in the flue gas. The structural heat losses from the system components between the separator/reservoir and the evaporative cooler and the heat absorbing capacity of the minimum flow of atomizing air were sufficient to cool the flue gas to less than 700°F, the test design condition, by the time the gas reached the evaporative cooler exit. Therefore, no water was introduced into the evaporative cooler during the vitrification period.

Once the test design conditions were established, the conditions were generally maintained for the entire test. After about 2 hours of steady state operation, flue gas sampling in accordance with EPA Methods was intitiated by CSC. The flue gas was sampled between the evaporative cooler and baghouse for analysis with respect to particulate and total fluoride concentrations. The flue gas was also sampled at the exit of the baghouse for analysis with respect to total fluoride concentrations. Two flue gas samples
were taken about one hour apart, each with a duration of about 1 hour. A “thimble” catch of particulate was also performed at the same time as the EPA Methods sampling after the baghouse.

Samples of the feedstock were taken from the feed hopper via a thief at one-hour intervals throughout the vitrification period, except during the two one-hour flue gas sampling periods. Samples of the molten glass discharging from the separator/reservoir and glass frit from the glass quench tank were taken at one-half hour intervals throughout the vitrification period, except during the flue gas sampling periods. During the flue gas sampling periods, feedstock and glass samples were taken at 15 minute intervals.

At the end of the test, particulate removed from the flue gas in the baghouse was collected for incorporation into the feedstock for Trial #16.

**Trial #17 (Vortec Test #188)**

Trial #17 was performed on December 13, 2002, to investigate the effect of increasing the silica content of the feedstock and reducing feedstock flowrate to the system on partitioning of fluoride to HF and HF concentration in the flue gas. About 8 hours of vitrification operation was performed processing about 3,430 lbs of feedstock. The feedstock contained 43% combined 1<sup>st</sup> and 2<sup>nd</sup> cut SPL and 11% baghouse dust from Trial #16.

Plots of system gas temperatures versus time are presented in Figure 5.4.7-5, and plots of feedstock flowrate and HF concentrations before and after the baghouse are shown in Figure 5.4.7-6.
Feedstock flow was initiated at a rate of about 200 lb/hr. The feedstock flowrate to the system was gradually increased to about 500 lb/hr, the average feedrate during Trials #15 and #16. Natural gas, oxygen, and air flows to the system were adjusted to maintain the system temperatures generally in the range from 2,400°F to 2,500°F with an oxygen enhancement level of about 40 wt.%. The procedures for establishing the flow rates and temperatures were the same as those described for Trial #15.
The HF concentration was allowed to stabilize for about 2 hours after the feedstock flowrate reached nominally 500 lb/hr. The feedstock flowrate was then reduced to about 350 lb/hr. The HF concentration in the flue gas decreased when feedstock flow was reduced; therefore, the feedstock flow was held constant at nominally 350 lb/hr for about 1.5 hours to allow the HF concentration to stabilize. After the HF concentration stabilized, the natural gas flow to the oxy-fuel burner in the lid of the CRV reactor was reduced to reduce the temperature in the top of the reactor where the feedstock was injected. The temperature in the top of the reactor, as measured by the two thermocouples protruding about 1” beyond the refractory in the lid, was decreased from 2,500°F to about 2,000°F. There was no apparent change in HF concentration in the flue gas as a result of the decrease in temperature. The temperature was not decreased any further, and the 2,000°F condition was not held very long, because the flame safety system closed the natural gas supply valve intermittently at high frequency at this condition. It is suspected that the feedstock being injected into the top of the reactor obscured the ultraviolet light sensor at the low firing rate of the burner. A very sensitive sensor must be used to detect the flame with the high solids loading in the top of the reactor even under normal firing conditions.

The feedstock was sampled at one-half hour intervals throughout the test, and the molten glass and glass frit were sampled at 15 minute intervals. EPA Method flue gas sampling was not performed in order to minimize the costs associated with the additional test. However, a “thimble” catch of particulate was collected from the flue gas after the evaporative cooler for chemical composition analysis.

5.4.8 Results of 4th Budget Period Testing

HF Concentration and Fluoride Partitioning

Feedstock, glass, and flue gas samples from the tests were sent to Alcoa Technical Service’s laboratory for analysis with respect to metals, carbon, and fluoride concentrations. Samples of the glass were also sent to Alcoa for analysis with respect to fluoride leaching characteristics.

An accurate measurement of the maximum HF concentration in the flue gas during Trial #14 was not possible because of the high particulate and HF concentration in the flue gas at the measurement location. The high concentrations occurred because of the reduction in flue gas flowrate made possible by insulating the CMS® components and oxygen enhancement of the process. The maximum recorded HF concentration was about 1.2%(v); however, the concentration was still increasing at the time that the instrumentation ceased functioning. Based on the measured HF concentrations and flue gas flowrate during Trial #14, and experimental data from previous tests, an HF concentration at steady-state feedstock flowrate was estimated to be on the order of 2%(v).

The HF concentration in the flue gas after the evaporative cooler based on the modified EPA Method 13B procedure at steady-state feedstock flowrate was about 1.3%(v). However, the pH of the impinger solution indicated that it was saturated with HF. Therefore, it is anticipated that not all the HF was captured for analysis. Based on this result, the impinger solution for Trials #15 through #17 was a 1N sodium hydroxide solution instead of the 0.1N sodium hydroxide solution used in Trial #14.

NOx concentrations in the flue gas were about 25% lower with oxygen enhancement than without oxygen enhancement. The mass flowrate of NOx emissions were about 50% lower with oxygen enhancement, because of the reduced flue gas flowrate achieved with oxygen enhancement.
Results of chemical analyses on the glass samples taken during Trial #14 revealed a fluoride concentration in the glass of about 6%. This was higher than concentrations observed during previous tests, which were on the order of 1% to 4%. There was some concern that this was caused by a lower gas temperature in the top of the CRV reactor, as indicated by thermocouple measurements.

During Trials #15 and #16, HF concentrations on the order of 2%(v) were measured; however, the differences in the concentrations measured by the FTIR analyzer, LasIR system, and flue gas sampling and analyses in accordance with modified EPA Method 13B were significant. The differences in the HF concentrations measured via the FTIR instrumentation and the LasIR were between 31% and 35% during Trials #15 and #16, and about 15% during Trial #17. The total fluoride concentrations measured after the baghouse via EPA Method 13B were consistently lower than either the FTIR or LasIR measured concentrations. One would expect the EPA Method 13B data to slightly higher since the baghouse doesn’t remove all the particulate from the flue gas. The data indicate that HF concentrations in the range of 2%(v), the goal established by Alcoa, are achievable with the CMS® process.

The data with respect to percent of the fluoride from the SPL partitioning to HF in the flue gas varied widely depending on the method of HF concentration measurement. The data based on the FTIR measurement ranged from 59% to 89% with the Trial #15 average being 76%. The data based on the LasIR measurement ranged from 86% to 130% with the Trial #15 average being 91% and the Trial #16 average being 110%. The data based on the EPA Method 13B fluoride concentration and flue gas flow rate ranged from 47% to 105% with averages of 60% for Trial #15 and 76% for Trial #16. The data based on the EPA Method 13B fluoride concentration and the mass balance flue gas flow rate ranged from 40% to 85% with averages of 48% for Trial #15 and 63% for Trial #16. Based on an average of all the data, about 75% of the fluoride in the SPL partitions to the flue gas as HF.

An analysis of the data from Trial #17 indicates that neither reducing the SPL concentration in the feedstock nor reducing the feedstock flowrate have a beneficial impact on HF concentration or partitioning of fluoride from the SPL to HF in the flue gas. The HF concentration and partitioning appear to have decreased relative to Trials #15 and #16. The data also indicate that reducing the gas temperature at the top of the CRV reactor from 2,500°F to 2,000°F (based on measurements by thermocouples protruding 1” beyond the refractory in the lid of the reactor) did not appear to impact HF concentration or partitioning of fluoride to HF. However, post-test inspection of the CRV reactor refractory surface indicated that the feedstock was very rapidly impacting the wall on one side at the top of the reactor, and therefore, not remaining in suspension in the gas stream for any significant distance.

The data from Trial #17 also indicate that reducing the gas temperature at the top of the CRV reactor from 2,500°F to 2,000°F (based on measurements by thermocouples protruding 1” beyond the refractory in the lid of the reactor) did not impact HF concentration or partitioning of fluoride to HF. However, post-test inspection of the CRV reactor refractory surface indicated that the feedstock was very rapidly impacting the wall on one side at the top of the reactor, and therefore, not remaining in suspension in the gas stream for any significant distance. This was not observed at the end of previous tests, and it is suspected that it resulted from a change in the feedstock injection system necessitated by the installation of the oxy-fuel burner in the test system.

Results of analyses on the glass revealed fluoride concentrations ranging from 3% to 6%. The effect of the feedstock impacting the wall at the top of the CRV reactor is not known. However, it is suspected that
the limited suspension time in the hot gas stream would limit volatilization of fluoride, thus increasing fluoride partitioning to the glass and decreasing partitioning to HF.

**Glass Fluoride Leaching Results**

Leach tests were performed on the glass cullet samples to characterize the chemical durability of the vitrified product with respect to the metals of interest and fluoride. The leach tests were performed with standard TCLP leach conditions using various extraction fluids. The leachate concentration of the metals were significantly below regulatory limits and are not a concern due to the low concentrations in the SPL glasses.

The species of primary concern in the SPL glasses is the fluoride. Based on the results of the leaching tests, two observations can be made: (1) the leachate fluoride concentration increases significantly as the final pH of the solution decreases; and (2) for a given pH, the leachate fluoride concentration increases as the glass fluoride concentration increases.

With the test method proposed by the EPA using deionized water, the maximum fluoride leach concentration was on the order of 9 mg/l. The data indicate that glass fluoride concentrations greater than 7% would be required to exceed a fluoride leachate concentration of 10 mg/l, the level recommended by Vortec to the U.S. EPA to be used as the standard. Fluoride concentrations this high have not been observed in any glass produced during the operation of the CMS® and this level is not anticipated.

**5.5 TASK 5 – GLASS FIBER QUALIFICATION TESTING**

Samples of the glass fibers produced during the fiberizing trial (Trial #4) in the second budget period were analyzed for physical and thermal characteristics by CertainTeed Corporation. The New York State College of Ceramics at Alfred University performed a dissolution analysis on the fiber. Its performance was compared to a commercially available mineral fiber.

**5.5.1 Fiber Size Analysis**

The size distribution of the fibers produced from SPL glass during the pilot-scale fiberizing trials and a commercial mineral fiber product were analyzed by CertainTeed Corporation. The results are shown in the histogram in Figure 5.5-1. The data indicate that the average diameter of the fiber produced from SPL glass meets the goals of the project; that is, between 3 to 5 microns. The data also indicate that a large percentage of the fibers are finer than the reference commercial mineral fiber product, which should result in lower density and improved thermal performance of an insulating layer using the fibers. It will also decrease the bio-persistence of the fibers which is desirable from a health and safety viewpoint. A very small percentage of the fibers are one micron or less in diameter, which is desirable because finer fibers tend to create more “dusting.”
Figure 5.5-1 Fiber Size Distribution - Analysis by CertainTeed Corporation

Figure 5.5-2 shows the results of fiber diameter distribution analysis performed at Alfred University. Although the size distribution of the commercially available mineral fiber as analyzed by Alfred University is different than the results from CertainTeed’s analysis, the data for the fiber produced from SPL glass in the pilot-scale trial is similar to the results of the analysis performed by CertainTeed. Both analyses were performed using a scanning electron microscope (SEM).

### 5.5.1 Fiber Performance Analysis

CertainTeed Corporation performed tests on the fiber produced from SPL glass in the pilot-scale trial to determine if it meets the requirements of ASTM C764 Specification for Mineral Fiber Loose Fill Insulation. The tests performed were as follows:

1. Corrosion Testing (under ASTM C764),

#### Corrosion Testing

The fiber produced from SPL glass in the pilot-scale trial and a commercial mineral fiber product were evaluated for corrosiveness to aluminum, copper, and steel in accordance with the method outlined in section 12.8 of ASTM C764, *Standard Specification for Mineral Fiber Loose-Fill Thermal Insulation*. 
For this test procedure, five each of specially cleaned steel, aluminum, and copper test coupons are “sandwiched” between two ½” layers of loose-fill insulation. The insulation is held uniformly against each side of the coupon with wire screens and rubber bands. For each metal, five control specimens are prepared by placing two layers of sterile cotton against the metal coupon in the same manner as the insulation specimens. The test and control specimens are then suspended in a humidity chamber controlled at 95% relative humidity and 120°F for a period of time determined by the type of metal being tested. Steel is exposed for 96 hours; aluminum and copper are exposed for 720 hours. After the exposure period, the coupons 5 test and 5 control coupons for each metal are examined by a panel of four judges. Each judge independently ranks the coupons in order from least severe to most severe corrosion. The arithmetic sum of all rankings for each coupon is then calculated.

These sums are then ranked from 1 (lowest total) to 10 (highest total). The new rankings established are then totaled for the control coupons only. If this sum is less than 21, the control coupons are judged to be significantly less corroded than the test coupons, and the insulation is considered to have failed the test. Any sum of the rankings greater than or equal to 21 indicates that the control coupons are not significantly less corroded than the test coupons, and the insulation is considered to pass the test.

The results of the corrosion testing are shown in Table 5.5-1. Since the sum of the rankings are all greater than or equal to 21, both the fiber produced from SPL glass in the pilot-scale trial and the commercially available mineral fiber passed the corrosiveness test for all three metals.

Table 5.5-1 Corrosiveness Test Results
The results of the testing on four specimens of the fiber produced from SPL glass in the pilot-scale trial are shown in Table 5.5-2. The data indicate that all the specimens met the pass criteria.

### Table 5.5-2 Behavior in a Vertical Tube Furnace

<table>
<thead>
<tr>
<th>Replicate</th>
<th>Density, lb/ft³</th>
<th>Temperature Rise, °C</th>
<th>Flame Time, sec.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Interior</td>
<td>Surface</td>
</tr>
<tr>
<td>Specimen a</td>
<td>2.35</td>
<td>7</td>
<td>8</td>
</tr>
<tr>
<td>Specimen b</td>
<td>2.46</td>
<td>0</td>
<td>9</td>
</tr>
<tr>
<td>Specimen c</td>
<td>1.92</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Specimen d</td>
<td>2.40</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

Critical Radiant Flux

Testing was performed in accordance with ASTM E970, *Standard Test Method for Critical Radiant Flux of Exposed Attic floor Insulation Using a Radiant Energy Heat Source*. Testing was performed on three specimens of fiber produced from SPL glass in the pilot-scale trial. The results of the testing are shown in Table 5.5-3.

### Table 5.5-3 Critical Radiant Flux
ASTM Specification C764 for Mineral Fiber Loose-fill Insulation specifies that the average critical radiant flux, when determined by ASTM E970 shall be greater than or equal to 0.12 watt/cm$^2$. The specimens did not ignite during the two-minute pilot burner application. The critical radiant flux at the 10cm mark was measured at 0.85 watt/cm$^2$. Therefore, a specimen that burns less than 10cm has critical radiant flux that is greater than 0.85 watt/cm$^2$. The fiber sample met the ASTM C764 requirement for critical radiant flux.

Fiber Dissolution Analysis

Dissolution analyses were performed on the fiber produced from SPL glass in the pilot-scale trial and a commercial mineral fiber, provided to Vortec by CertainTeed Corporation, at the New York State College of Ceramics at Alfred University.

The analysis method chosen was as identical as possible to that of R.M. Potter and S.M. Mattson in "Glass Fiber Dissolution in a Physiological Saline Solution" Glasstech. Ber 64 16-28 (1991). Liquid is flowed through a cassette in which the glass fibers are trapped. The experiment is maintained at 36.5°C in a constant temperature water bath. The flow rate for the liquid is 15 ml/hr maintained by a multiport pump. The liquid used was 0.85% sodium chloride solution. The fibers were dried at 110°C for 10 hours before weighing. The density was calculated from the measured BET surface areas assuming continuous fiber and neglecting end effects. Fiber diameters for Fiber A (commercially available mineral fiber provided by Vortec) and Fiber B (fiber produced from SPL glass during pilot-scale testing) were measured using the SEM (see section 5.5.1).

Results of analysis are shown in Table 5.5-4. The dissolution rate ($k_{\text{dis}}$) values are reported in terms of the average and the range given the range of fiber diameters measured. The results indicate that the rate of dissolution of the fiber produced during the pilot-scale trial from SPL glass is comparable with that of the commercial mineral fiber.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Average $k_{\text{dis}}$ (ng/cm$^2$/hr)</th>
<th>$k_{\text{dis}}$ Range (ng/cm$^2$/hr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A (commercial mineral fiber)</td>
<td>11.44</td>
<td>5.04 – 17.85</td>
</tr>
<tr>
<td>B (fiber from SPL glass)</td>
<td>7.00</td>
<td>3.63 – 10.37</td>
</tr>
</tbody>
</table>

5.6 TASK 6 – PRELIMINARY DESIGN OF COMMERCIAL AIR POLLUTION CONTROL SYSTEM
Techno-economic evaluations of APC system concepts for removing fluorides from the CMS® flue gas and producing marketable fluoride products were performed during the 2nd budget period by HTS (now Danieli Corus Technical Services) and Vortec. Flue gas characterization data from the 2nd budget period pilot-scale SPL vitrification trials were used in the evaluations. Based on the results of the techno-economic evaluations, a preliminary layout design was developed for an aluminum fluoride producing APC system.

5.6.1 Concept Descriptions

A process schematic for a generic APC system to remove fluoride from the CMS® flue gas is shown in Figure 5.6-1. The system consists of an evaporative cooler, a baghouse, and an acid-gas scrubber in which a reagent is introduced to react with HF in the flue gas to produce a fluoride product. Flue gas from the CMS® is cooled in the evaporative cooler to a temperature compatible with the use of a baghouse filter system for removal of particulates from the gas. The fluoride in the flue gas as it discharges from the CMS® at a temperature of about 2,600°F is primarily in the form of HF gas. However, as the flue gas is cooled in the evaporative cooler, a significant portion of the fluoride reacts with primarily sodium to form sodium fluoride compounds that condense at the lower temperatures. These compounds, along with non-volatile particulates carried over from the CMS® in the flue gas, must be removed prior to the HF scrubber to prevent excessive contamination of the fluoride product.

Figure 5.6-1  Process Schematic of Generic APC System
Two HF scrubbing system concepts were evaluated. One concept was a wet scrubbing process in which lime is used as the reagent to react with HF to produce a 97% pure calcium fluoride product, acid-grade fluorspar. The basic chemical reaction for this process is:

\[ 2\text{HF} + \text{CaO} \rightarrow \text{CaF}_2 + \text{H}_2\text{O} \]

The acid-grade fluorspar product can be used as feedstock in a conventional process for the manufacture of aluminum fluoride, which is used in primary aluminum smelting operations. The market value for acid-grade fluorspar is in the range from $70/ton to $100/ton.

The alternative HF scrubbing system concept evaluated was a dry scrubbing process in which aluminum oxide (alumina) is used as the reagent to produce a 70% aluminum fluoride product directly from HF in the CMS® flue gas. The basic chemical reaction for this process is:

\[ 6\text{HF} + \text{Al}_2\text{O}_3 \rightarrow 2\text{AlF}_3 + 3\text{H}_2\text{O} \]

Because the intermediate process for converting the calcium fluoride to aluminum fluoride is eliminated in this concept, less energy is needed to come to the same end. Therefore, the added value to the aluminum fluoride approach is much higher than the calcium fluoride approach. The market value for the 70% aluminum fluoride product is $500/ton to $600/ton. However, this process is less developed technically than a calcium fluoride process.

Results of the pilot-scale vitrification trials indicated that a significant portion (about 50%) of the fluoride in the SPL waste introduced into the CMS® ends up in the baghouse particulate as sodium fluoride compounds. Therefore, a wet chemistry process was initially considered to convert the sodium fluoride in the particulate to an acid-grade fluorspar product. This is accomplished by first dissolving the sodium fluoride in water, and then introducing lime to react with the sodium fluoride to produce calcium fluoride and sodium hydroxide as follows:

\[ 2\text{NaF} + \text{CaO} + \text{H}_2\text{O} \rightarrow \text{CaF}_2 + 2\text{NaOH} \]

A flow schematic of this process is shown in Figure 5.6-2. However, this process significantly increases the complexity and cost of the APC system. As a result, an analytical modeling study was performed under another Vortec funded project to evaluate the effect of recycling all of the particulates from the baghouse back to the CMS® on system performance and cost. Results of the study indicate that recycling the particulates back to the CMS® will be beneficial in that it will increase the amount of sodium that partitions to the glass and increase the amount of fluoride that is retained as HF in the flue gas downstream of the evaporative cooler. Results indicate that as much as 86% of the fluoride in the SPL may be retained as HF in the flue gas downstream of the evaporative cooler with particulate recycling [An increase in HF concentration in the flue gas via baghouse dust recycling was verified experimentally during the 3rd budget period trials]. The elimination of the wet chemistry process for converting the sodium fluoride to calcium fluoride significantly reduces the cost of the APC system. Additionally, particulate recycling will increase the quantity of aluminum fluoride that can be produced in the HF scrubber, thus significantly increasing the cost effectiveness of that process. Therefore, the comparative
economic evaluation of the calcium fluoride and aluminum fluoride producing APC system concepts assumed the recycling of baghouse particulate to the CMS®.

Calcium Fluoride APC Concept

A process schematic of the calcium fluoride producing APC system concept is shown in Figure 5.6-3. Flue gas from the CMS® is first cooled via an evaporative cooler to a temperature compatible with the use of conventional fabric filters in a baghouse (ca. 400°F). HF laden flue gas from the baghouse is transported to the HF scrubber where lime is introduced to react with the HF, producing calcium fluoride. There is a small amount of sulfur that is present in SPL waste, and this sulfur partitions to the flue gas as $\text{SO}_2$. Some of the lime that is introduced into the scrubber reacts with the $\text{SO}_2$ to produce calcium sulfate. The basic chemical equation is:

$$\text{SO}_2 + 2\text{CaO} + 2\text{H}_2\text{O} \rightarrow \text{Ca}_2\text{SO}_4 \cdot 2\text{H}_2\text{O}$$

The amount of sulfur in the SPL waste is typically so small that the calcium sulfate content in the fluorspar will be below the limits established for acid-grade fluorspar. If the sulfur content in the SPL waste is too high for producing acid-grade fluorspar with a single scrubber, separate scrubbing of the HF and $\text{SO}_2$ in the flue gas can be accomplished with two scrubbers, by maintaining the scrubber solutions at the appropriate pH levels.
Figure 5.6-3  Calcium Fluoride Producing APC Concept

Lime scrubbing with calcium fluoride production is very similar to the well-known limestone/gypsum process that is probably the most widely used scrubbing technology in the world serving thousands of megawatts of fossil fuel power generating units. Based on the experience of HTS within the field of limestone scrubbing, the lime/calcium fluoride technology is the most readily available technology to scrub the HF from the CMS® flue gas. Compiling this technology from existing systems has led to a concept that is the most developed for this particular application and is used in the economic evaluations in this study.

Aluminum Fluoride APC Concept

A process schematic of the aluminum fluoride producing APC concept is shown in Figure 5.6-4. The optimum temperature range for reacting alumina with HF in the flue gas to produce aluminum fluoride is from 600°F to 750°F. Therefore, the flue gas from the CMS® is first cooled to within this temperature range in the evaporative cooler. This temperature range is too high for conventional fabric filters; however, filters developed by Biothermica International, Inc. that are manufactured from stainless steel mesh, capable of operating at temperatures as high as 1,050°F, may be used in a baghouse also constructed of stainless steel. As discussed in previous sections of this report, such a baghouse filter system was installed and operated in Vortec’s pilot-scale CMS® test facility. The tests demonstrated that this type of filter could be applied to this APC system concept.
Since SO$_2$ will not be removed in the HF scrubber using alumina as the reagent, a separate acid-gas scrubber is used in this APC system concept for removing SO$_2$ to concentrations satisfying emissions regulations. Caustic soda is used as the reagent in the SO$_2$ scrubber. Because of the nature of the process, it also provides final cleanup of residual HF before the gases are discharged to the atmosphere.

Technologies for producing high concentrations of aluminum fluoride (>60%) in products from HF scrubbers operating with low HF concentrations in the gas typical of those expected from the CMS® (ca. 16,000 mg/m$^3$) may not be readily available. Conventional aluminum fluoride production facilities use technologies which produce aluminum fluoride from concentrated HF gases (150,000 to 300,000 mg HF/m$^3$ gas) based on the Kvearners-Busch process. This process uses a fluid bed reactor in which the alumina is brought into close contact with the concentrated HF gases. Applying this technology to an SPL vitrification/recycling plant is very expensive. In addition, operating with lower HF concentrations can lead to different results when using smelter grade alumina. The exact mass transfer mechanism is unknown, and it can very well be that the lower HF concentrations increase mass transfer resistance. This could lead to less effective aluminum fluoride production and/or excessive reactor/scrubber sizes.

In order to have a better understanding of the mass transfer during the conversion of alumina into aluminum fluoride, preliminary tests under another research program were conducted by HTS using a simulated gas with HF concentrations similar to those expected from the CMS® when vitrifying SPL waste. The overall result was that it is possible to achieve aluminum oxide conversion, but only at values up to 60%. However, this depends on the reactor design, and, therefore, it is believed that higher values can be achieved in better reactor designs. Another outcome of the tests was that the use of a high specific surface area alumina instead of a smelter grade alumina did not result in a higher aluminum
fluoride purity. This led to the conclusion that mass transfer to the alumina does not influence the overall
conversion, and that most probably the chemical reaction is the controlling factor in the mass transfer
process.

HTS has a patented, proprietary technology, referred to as the Pleno IV technology, that is currently
successfully used downstream of aluminum smelters for dry adsorption of HF on alumina. The HF
concentrations in the gas to the technology are in the range from 100 to 350 mg/m3. Based on the results
of their preliminary research, HTS believes that the Pleno IV technology may be used as a basis for
design of an APC system for the SPL vitrification process. However, in current applications, the
technology operates at temperatures in the range from 140°F to 200°F. At these temperatures, it has been
found that only adsorption of HF occurs, not chemical reaction between the alumina and HF. This means
that the process is limited by the adsorption capacity of alumina, and typically the alumina weight
increases by up to 2.2 percent; that is, the product from the scrubber is alumina with only up to 2.2%
fluoride. However, by increasing the operating temperature above 240°F, a chemical reaction between
the HF and alumina can be effected which can significantly increase the fluoride recovery. Like most
chemical reactions, the temperature drives the reaction rate exponentially, and that can lead to high
conversions. Nevertheless, a side effect comes into play at temperatures close to 1,100°F; the aluminum
fluoride degrades to alumina (reverse reaction). Data from industry and experiments performed by HTS
prior to this research program indicate that the optimum temperature range is 600°F to 750°F.

Alcoa, Inc. has a scrubbing technology producing aluminum fluoride from HF contained in a flue gas
from an SPL treatment process in operation in Portland, Australia. The conditions under which this
technology is operating are not known at this time. Agreements with Alcoa and the owners of the facility
in Portland are currently being pursued by Vortec and HTS so that technical and cost information on that
technology can be obtained. This would provide data for an assessment of the technical and economic
feasibility of applying the technology to an SPL vitrification/glass fiber manufacturing process using the
Vortec CMS® technology.

For the techno-economic evaluations performed in this R&D program, the design of the aluminum
fluoride reactor (HF scrubber) is based on HTS’s Pleno IV technology. The applied reactor technology
consists of the following:

1. An injector nozzle (called Vertical Radial Injector – VRI)
2. Reactor duct
3. Filter module for separation of gas and solids, and for recycling material.

Using the Pleno IV technology, it is possible to meet the main criteria for this reactor design; that is:

1. The reactor must be able to handle alumina. Alumina is very abrasive and needs proper handling
   for reliable operation. The Pleno IV system has been specifically designed to handle alumina.
2. The alumina must be dispersed very quickly and very widely in order to maximize alumina
   conversion in the quickest possible contact time. The patented VRI injector provides such
   dispersion. A photograph of the dispersion in a commercially operating system is shown in
   Figure 5.6-5.
3. Based on industry experience, it is known that it is necessary to have considerable recycling of
   material from the reactor (scrubber) in order to obtain both the required product quality and low
   emissions. The Pleno IV technology is capable of providing high recycling ratios.
The reactor (scrubber) consists of a number of modules that each handles a part of the off gases. A main header divides the off gases over the modules. The off gases enter a reactor duct that is vertically positioned. The VRI injector that disperses the alumina into the gases is installed in the duct. The physical dimensions are such that the gas velocity is high enough to carry all the solids to the filter module. The reactor duct has a certain length to provide for adequate contact time.

Once inside the module, the gases rapidly expand so that the gas velocity drops considerably. Initial separation of solids takes place, and most of the solids are collected in the hopper of the module. The fines are carried over to the bags where they are filtered out. Some final scrubbing takes place within the cake on the bags. A low pressure/high volume pulse system provides for proper bag cleaning/cake renewal.

The hopper bottom of the module is specially designed to process the alumina/aluminum fluoride material. It has a flat bottom with fluidization cloth incorporated. The material is fluidized in order to transport it. An overflow baffle controls free inlet to the recirculation outlet and an overflow pipe regulates automatically the discharge of excess material. The overflow pipe maintains a constant level of material in the hopper. The recirculation is established by making use of a circular orifice. The constant level of material provides constant material flow rate through the orifice. This ensures that the required recycle ratio is maintained. Perhaps in future installations, a mechanism can be used for dynamic recycle control, but for now no risk is taken with respect to abrasion by the material. The orifice has proven to be reliable.

Both the baghouse filter and the HF scrubber operate under negative pressure to ensure that none of the HF loaded gases leak into the surrounding environment. The off gases from the HF scrubber go through an induced draft fan, that overcomes the pressure drop of the system, and are directed to the acid-gas scrubber.

General arrangement drawings for an aluminum fluoride producing APC system removing HF from the flue gas of a CMS® based SPL recycling facility processing 60,000 tons of SPL waste annually are shown in Figures 5.6-6 through 5.6-8.
5.6.2 Economic Evaluation

An economic evaluation of the two alternative APC system concepts was performed based on the process descriptions provided in the previous section. A heat and mass balance developed for a commercial SPL recycling/glass fiber manufacturing plant under another study funded by Vortec and Reynolds Metals Company was used as input to the APC system process design. The following steps were then followed to completion of the economic evaluation:

1. Process design,
2. Equipment sizing,
3. Plant layout development,
4. Equipment cost estimating,
5. Plant cost estimating, and
6. Plant economics development.

This procedure was used for both the aluminum fluoride and calcium fluoride APC system concepts.

Technical Basis

The technical parameters used in the evaluation are as follows:

1. The SPL Recycling/Glass Fiber Manufacturing plant processes 60,000 tons/yr. of SPL waste.
2. There are two evaporative cooling units each receiving flue gas from a single Vortec CMS®
3. The two evaporative cooling units feed flue gas to a single baghouse/HF scrubbing system.
4. The baghouse in the calcium fluoride APC system concept uses conventional fabric filters with material compatible with the flue gas constituents.
5. The baghouse and reactor filters in the aluminum fluoride APC system concept are fabricated from stainless steel mesh.
6. The baghouse/HF scrubbing systems consist of one baghouse, one HF reactor, and one acid-gas scrubber (in the case of the aluminum fluoride APC system). These technologies have built-in redundancy.
7. The reagent for the aluminum fluoride producing system is smelter grade alumina with specific surface of 60 to 80 m² per gram.
8. Raw and product material is stored in silos with three (3) days capacity.

Economic Basis

The following financial parameters were used for the economic evaluations:

1. Plant economic lifetime is 10 years.
2. The plant operates 7,200 hours per year based on 24 hrs/day x 300 day/year.
3. Long term debt interest rate is 8% per year.
4. Labor rates for supervisors, operators, and maintenance personnel are $35/hr, $25/hr, and $28/hr, respectively.
5. Electricity costs are $0.03 per kWh.
6. Process water costs are $6 per 1,000 gallons.
7. Wastewater discharge costs are estimated to be $8 per 1,000 gallons.
8. Smelter grade alumina cost is $200 per metric ton ($181 per short ton).
9. Caustic soda cost is $400 per metric ton ($362 per short ton).
10. Lime cost is $80 per metric ton ($72 per short ton).
11. Calcium fluoride sale price is $100 per metric ton ($90 per short ton).
12. Aluminum fluoride sale price is $550 per metric ton ($498 per short ton) at 70% purity.

An important part of the cost development is an estimate of the costs for the process system equipment. Some quotations were used to develop the costs of major equipment. Most costs were derived from recent quotations with the use of scaling factors based on equipment sizes and capacities. Other parameters used were taken from industry standard cost estimating sources and tools.

Results of Economic Evaluation

A summary of the results of the economic evaluation of the alternative APC system concepts is presented in Table 5.6-1. The data relates to the construction and operation of the APC system alone, not the entire SPL recycling/glass fiber production facility. The data show that not only is the capital investment lower for the aluminum fluoride producing APC system, but the aluminum fluoride producing system has a positive cash flow; that is, the revenue from the sale of the aluminum fluoride product is greater than the cost of operating the system. As a result, the aluminum fluoride producing concept has a positive return on investment as opposed to a negative return on investment for the calcium fluoride producing concept.

<table>
<thead>
<tr>
<th>Table 5.6-1</th>
<th>Results of Economic Comparison of Alternative APC System Concepts</th>
</tr>
</thead>
<tbody>
<tr>
<td>Units</td>
<td>Aluminum Fluoride Producing Concept</td>
</tr>
<tr>
<td>Total Capital Investment (TCI)</td>
<td>$10^6 US$</td>
</tr>
<tr>
<td>Annual Operating Costs (AOC)</td>
<td>$10^6 US$</td>
</tr>
<tr>
<td>Total Operating Revenues (TOR)</td>
<td>$10^6 US$</td>
</tr>
<tr>
<td>Cash Flow (CF=TOR-AOC)</td>
<td>$10^6 US$</td>
</tr>
<tr>
<td>Payback Time (PT=TCI/CF)</td>
<td>Years</td>
</tr>
<tr>
<td>Return on Investment (ROI=(CF-TCI/n)/TCI x 100</td>
<td>%</td>
</tr>
</tbody>
</table>

5.7 SUMMARY OF RESULTS OF OTHER RELATED STUDIES

An engineering study was performed by Vortec under another project supported by Reynolds Metals Company, Alcoa, Inc., and CertainTeed Corporation during the 2nd budget period and updated at the end of the 4th budget period. The objectives of the study were to develop a conceptual design of a commercial facility for conversion of SPL waste into an insulating glass fiber product and aluminum fluoride, and perform an economic evaluation of the commercial facility based on the conceptual design data. Experimental data from the DOE supported project were used as input to the engineering study. HTS assisted Vortec in the conceptual design of the APC system.
The initial step in the study was the definition of baseline plant concept and design criteria. The potential plant site selected for the study was an existing Reynolds Metals Company facility in Gum Springs, Arkansas. The SPL waste processing capacity criteria for the baseline plant was 60,000 short tons per year of combined first and second cut SPL waste. A process schematic illustrating the baseline plant configuration is shown in Figure 5.7-1.

![Figure 5.7-1 Schematic of Baseline Plant for Conversion of SPL to Glass Fiber Products](image)

The baseline plant consisted of an air-fired CMS® for SPL vitrification, CertainTeed glass fiberizing system, and an aluminum fluoride producing APC system. A wet chemical process for converting the sodium fluoride in the particulate from the APC system baghouse into a calcium fluoride product was initially considered, but was determined to be uneconomical. Analytical modeling studies indicated, however, that recycling of the particulate back to the CMS® to increase the HF in the flue gas to the HF scrubber, thus increasing the production of aluminum fluoride, is technically feasible and more economical. Therefore, the baseline plant included this concept.

A heat and mass balance was developed for the baseline plant based on pilot-scale experimental data and analytical modeling studies. The heat and mass balance data were used to estimate the size of the process equipment and utility requirements. Conceptual layout drawings were then developed for the plant. These data were then used to estimate the capital and operating costs for the plant, which were used as input to a Vortec proprietary economic model to estimate the internal rate of return on the capital investment required for the plant and the rate of return on equity provided by financial investors. Reynolds Metals Co. and Alcoa, Inc. evaluated the plant economics relative to the current cost of SPL waste disposal via treatment and landfill.
Results of the economic evaluation of the baseline plant indicated that it would be marginally attractive relative to the current cost of SPL waste disposal via treatment and landfill. Therefore, Vortec performed additional analytical modeling studies to identify methods of decreasing the capital and operating costs of the plant.

One of the methods investigated for decreasing the costs was to partially oxygen fire the CMS® technology, based on results of some very preliminary pilot-scale investigations previously performed by Vortec. Results of the analytical studies indicated that partial oxygen firing of the CMS® could significantly improve the energy efficiency of the vitrification system, thus reducing the operating costs. This occurs because the increased oxygen concentration in the oxidant to the CMS® increases the oxidation rate of the carbon and reduces the flue gas volume flow. This allows the use of smaller components for the same waste throughput, thus decreasing structural heat losses and capital equipment costs. [The projected increase in feedstock throughput for the same size components via oxygen enhancement was experimentally verified during the 3rd budget period pilot-scale trials]. Additionally, the decrease in flue gas flowrate per unit of waste input will increase the HF concentration in the flue gas, resulting in an improvement in HF scrubber efficiency.

A revised heat and mass balance was developed for the partially oxygen-fired (oxygen-enriched) CMS® concept along with revised equipment size estimates, equipment capital costs, and system operating costs. The revised data were then input to the economic model. The results indicated that with an oxygen-enriched CMS® with 85% of the fluoride in the SPL partitioning to the flue gas as HF and an HF concentration in the flue gas of about 1%(v), a commercial plant for conversion of SPL waste to an insulating fiber product and aluminum fluoride would be economically attractive relative to current treatment and disposal options. Additionally, when applied to the approximate 100,000 tons per year of SPL waste generated annually in the United States, commercial implementation of the SPL conversion technology could save over 1.3 trillion Btu of energy annually relative to treatment and landfill the SPL waste, production of an equivalent amount of insulating fiber product using conventional glass melters, and production of an equivalent amount of aluminum fluoride using conventional technology. A breakdown of the energy utilization by category is summarized in Table 5.7-1.

### Table 5.7-1  Comparison of Technologies’ Energy Utilization and Energy Savings Summary

<table>
<thead>
<tr>
<th></th>
<th>Energy Utilization, trillion Btu/yr (1)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>O₂ Enriched CMS® (2)</td>
</tr>
<tr>
<td>SPL Processing</td>
<td>0.5(3)</td>
</tr>
<tr>
<td>Aluminum Fluoride Production</td>
<td>0.04</td>
</tr>
<tr>
<td>Melting for Fiber Manufacturing</td>
<td>0</td>
</tr>
<tr>
<td>Total</td>
<td>0.54</td>
</tr>
<tr>
<td>Total Savings</td>
<td>1.36</td>
</tr>
</tbody>
</table>

(1) Based on 100,000 tons per year SPL  
(2) Oxidant is air enriched to 40 wt.% O₂  
(3) Includes O₂ generation energy requirement at 2.73 million Btu/ton O₂
A commercial scale CMS® with single unit throughput of about 30,000 tons per year of SPL is expected to have a natural gas energy requirement of about 3 million Btu/ton of SPL. Oxygen generation energy requirements are estimated to total about 1.6 million Btu/ton of SPL based on an electrical energy requirement of 260 kW-hr/ton O₂ and an electrical to thermal energy conversion constant of 10,500 Btu/kW-hr. Therefore, the thermal energy requirement of a commercial CMS® process is anticipated to be about 5 million Btu/ton of SPL. Natural gas utilization of existing thermal treatment of SPL using a rotating kiln is estimated to be about 9 million Btu/ton of SPL. Therefore, the annual energy savings for the thermal treatment process alone is about 0.4 trillion Btu annually. The savings in energy realized by producing aluminum fluoride in the air pollution control system from the HF in the flue gas from the CMS® process relative to conventional technology for aluminum fluoride production is estimated to be about 0.16 trillion Btu annually. The energy savings associated with producing an insulating fiber product from the glass resulting from the thermal treatment (vitrification) of the SPL waste relative to the production of the same amount of fiber using conventional glass melting furnaces is estimated to be about 0.8 million Btu annually. Therefore, the total energy savings that can be realized by commercial implementation of the CMS® technology for converting SPL waste to useful glass fiber and aluminum fluoride products is about 1.36 trillion Btu annually.

6.0 CONCLUSIONS AND RECOMMENDATIONS

Vortec Corporation has completed a four-year research and development project for application of Vortec’s CMS® technology to conversion of spent potliner (SPL) waste from primary aluminum smelting operations to useful glass fiber products. Seventeen pilot-scale SPL vitrification trials, including two fiberizing trials, were performed in Vortec’s test facility in Harmarville, Pennsylvania. Additionally, techno-economic analyses were performed evaluating alternative concepts for removal of fluoride from the flue gas and production of value-added fluoride co-products.

Data developed during the project indicate thatconversion of SPL to commercial quality glass fiber and aluminum fluoride products is technically feasible. Glass fibers produced during the pilot-scale testing met all the project performance objectives and passed ASTM tests for mineral fiber loose fill insulation. The flue gas from the pilot-scale CMS® when vitrifying SPL waste was characterized with respect to HF and fluoride salts. The experimental data indicate that through oxygen enhancement of the CMS® and recycling of particulate removed from the flue gas in the baghouse back into the process, 75% or more of the fluoride in the SPL will partition to the flue gas as HF, and HF concentrations on the order of 2% can be achieved. The HF can be converted to a valuable aluminum fluoride product in an air pollution control system. The aluminum fluoride product can be recycled to the aluminum industry.

Techno-economic evaluations of a commercial plant for converting SPL to commercial quality glass fiber and aluminum fluoride products were performed under another project. The pilot-scale test data from the DOE funded project were used as input to the evaluation. Results of the evaluation indicate that a CMS® based process with oxygen enhancement and flue gas particulate recycling capability is an economically attractive alternative to current treatment and landfill disposal options. Results of the techno-economic evaluation also indicate that commercialization of the CMS® technology for conversion of SPL to useful products could save over 1 trillion Btu of energy annually when applied to the approximate 100,000 tons of SPL waste generated annually in the United States.

Alcoa, Inc. feels that a demonstration at a scale in between the pilot-scale CMS® (1,000 tons/yr SPL throughput) and a single commercial CMS® unit (20,000 – 30,000 tons/yr SPL throughput) is necessary.
to provide consistent experimental data from which to scale the process design. A program plan for demonstration testing on a larger scale at Ormet Primary Aluminum Corporation (ca. 5,000 tons/yr SPL) has been developed with Alcoa. Additionally, a plan for subsequent installation of a 20,000 ton/year or larger SPL vitrification process at an Alcoa facility has been developed. At the time of this report, Alcoa has not made the financial commitment necessary to continue the project into these phases.