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U.S. Army Toxic and Hazardous Materials Agency

BENCH-SCALE GLASSIFICATION TEST ON ROCKY MOUNTAIN ARSENAL BASIN F MATERIAL

(TASK ORDER NO. 8)

August 1988
Contract No. DAAK11-85-D-0008

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Process Development Branch
Aberdeen Proving Ground, MD 21010-5401

Final Report to
**United States Army
Toxic and Hazardous
Materials Agency
August 1988**

Bench-Scale Glassification Test on Rocky Mountain Arsenal Basin F Material

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Increasing public and regulatory attention focused on the growing problems with hazardous chemical waste disposal has prompted the examination of radioactive waste treatment methods and their applicability to confine hazardous chemical wastes. The joule-heated ceramic-lined melter (JHCM) developed by the Pacific Northwest Laboratory is a good example of radioactive waste treatment technology that has begun to show excellent promise in the hazardous chemical waste treatment field. Waste materials are blended with glass-forming minerals and processed through a high-temperature furnace to produce a highly durable glass product that immobilizes any hazardous species.

A bench-scale demonstration of this treatment technique was conducted for Arthur D. Little, Inc., to support their evaluation of innovative technologies for treating hazardous materials from the U.S. Army's Basin F at the Rocky Mountain Arsenal near Denver, Colorado. During a one-day experiment at Battelle, Pacific Northwest Laboratories, more than 50 lbs of Basin F material were converted to glass in a bench-scale melter.

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Aldrin, the organochlorine pesticide present in the Basin F samples at the highest concentration, was effectively destroyed when it was processed through the glassification system. The glassification system achieved a destruction efficiency of more than 99.9999%. Except for mercury, which largely volatilized from the melter, all metal species of concern including arsenic were immobilized in the product. High product quality was shown through testing using the U.S. Environmental Protection Agency's toxicity leaching procedure. Although mercury exhibited semivolatile behavior in the furnace and very little was immobilized in the product glass, its low concentration in the Basin F material and the ability to easily capture and concentrate it for separate disposal makes mercury release from the furnace a minor issue.

Gross particulate entrainment accounted for the release of other target metal species from the furnace. Entrainment was judged to be excessive during this test, but should not be a problem in the full-scale process with proper design and operation of the feed delivery system. The concentration of acid gases in the offgas (NO_x, SO_2, HCl, and HF), which are generated by decomposition of certain hazardous species, was low enough that the melter exhaust does not appear to require any special treatment for acid gas removal prior to release.

A preliminary process flowsheet was prepared to further examine technical feasibility and serve as the basis for a separate cost feasibility analysis. The flowsheet builds heavily on well-established, large industrial systems for processing physically similar materials. The development needs for full-scale application of the glassification technology appear modest. No insurmountable technical difficulties were identified by either the test or flowsheet development effort. Longer-term small-scale testing (lasting several weeks) is recommended to better define the process flowsheet and to substantiate engineering judgement used in preparing the flowsheet. A pilot-scale field demonstration program could follow shortly thereafter to complete engineering optimization, technology demonstration, and equipment design verification.

The glassification system performed well during all aspects of the test and successfully demonstrated the treatment approach as a suitable remediation technique for Basin F materials. Additional testing should lead to further improvements and thus, better performance as less-than-optimum conditions are corrected.

This report was prepared by Battelle Pacific Northwest Laboratories for Arthur D. Little, Inc., in fulfillment of a requirement for Task Order Number 8 under Contract DAAK11-85-D-0008.

SUMMARY

Increasing public and regulatory attention focused on the growing problems with hazardous chemical waste disposal has prompted the examination of radioactive waste treatment methods and their applicability to confine hazardous chemical wastes. The joule-heated ceramic-lined melter (JHCM) developed by the Pacific Northwest Laboratory[a] is a good example of radioactive waste treatment technology that has begun to show excellent promise in the hazardous chemical waste treatment field. Waste materials are blended with glass-forming minerals and processed through a high-temperature furnace to produce a highly durable glass product that immobilizes any hazardous species.

A bench-scale demonstration of this treatment technique was conducted for Arthur D. Little, Inc., to support their evaluation of innovative technologies for treating hazardous materials from the U.S. Army's Basin F at the Rocky Mountain Arsenal near Denver, Colorado. During a one-day experiment at Battelle, Pacific Northwest Laboratories, more than 50 lbs of Basin F material were converted to glass in a bench-scale melter.

Aldrin, the organochlorine pesticide present in the Basin F samples at the highest concentration, was effectively destroyed when it was processed through the glassification system. The glassification system achieved a destruction efficiency of more than 99.9999%. Except for mercury, which largely volatilized from the melter, all metal species of concern including arsenic, were immobilized in the product. High product quality was shown through testing using the U.S. Environmental Protection Agency's toxicity leaching procedure. Although mercury exhibited semivolatile behavior in the furnace and very little was immobilized in the product glass, its low concentration in the Basin F material and the ability to easily capture and concentrate it for separate disposal makes mercury release from the furnace a minor issue.

Gross particulate entrainment accounted for the release of other target metal species from the furnace. Entrainment was judged to be excessive during

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(a) Operated for the U.S. Department of Energy by Battelle Memorial Institute. Battelle, Pacific Northwest Laboratories is a division of Battelle Memorial Institute.

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CONTENTS

FIGURES

TABLES

1.0 INTRODUCTION

The Process Development Branch of the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) issued Task Order No. 8 entitled "Innovative Technology Development for Rocky Mountain Arsenal Environmental Control/Concept Evaluation" to Arthur D. Little, Inc. The objectives of this task order are to review the industrial database of promising hazardous material treatment technologies for application at Basin F of Rocky Mountain Arsenal (RMA); evaluate the candidate technologies (a maximum of three) through laboratory and/or bench-scale testing on RMA Basin F material; and prepare preliminary process designs and cost estimates for the most promising technologies.

An initial survey was completed, the candidate technologies were ranked, and the selected technologies for testing were identified.(a) Glassification was one of three promising technologies chosen for further evaluation. Battelle Pacific Northwest Laboratories (Battelle Northwest) was awarded a subcontract to perform a bench-scale feasibility test for this technology.

A quality control and a test plan were developed by Arthur D. Little, Inc. to control the data collection and sample analyses. Battelle Northwest, with assistance from Arthur D. Little, Inc., developed the specific test plan for glassification that established test parameters and procedures. During the test period, Arthur D. Little, Inc. personnel observed operations and assisted in sample collection.

The online exhaust gas sampling was performed for Battelle Northwest by NHS, Inc. (a subsidiary of the Hanford Environmental Health Foundation). The collected samples were analyzed in the Arthur D. Little, Inc. laboratories, which are certified by USATHAMA for the chemical analyses of concern, and the results provided to Battelle Northwest for evaluation. Test results are incorporated in this report along with a description of the test conditions and a discussion of the results.

(a) A final report entitled "Evaluation/Selection of Innovative Technologies for Testing with Basin F Material," and dated February 28, 1987, was prepared by Arthur D. Little, Inc.

The glassification system proposed by Battelle Northwest is known as a joule-heated ceramic-lined melter (JHCM). In order to evaluate this process for application to Basin F hazardous materials treatment and to predict full-scale equipment needs, it is necessary to establish the ultimate distribution of hazardous materials during processing. Samples of the Basin F material, glass additives, melter-generated glass, off gas, and off-gas scrub solutions were collected during bench-scale testing. Results of these sample analyses were then used to determine the destruction/immobilization of the hazardous materials during glassification and to identify any auxiliary treatment equipment needed to safely dispose of the Basin F materials.

Processing rates for this bench-scale system were determined in order to predict the full-scale size needed to treat the estimated 400,000 yd^3 of Basin F material in a 2.5-year period. The data from the bench-scale testing, along with engineering judgment were used in the preparation of the preliminary process flowsheet and the specification and selection of major equipment for a full-scale (950 ton/day) treatment plant. The flowsheet and equipment specified were then used to estimate the capital and operating costs for the full-scale glassification system.

The primary objectives of the bench-scale test were to determine

- the apparent organic contaminant destruction efficiencies achieved in the absence of an afterburner
- the disposition of the nonvolatile heavy metals
- the type and quantity of chemical additives required to produce glass at an acceptable temperature and rate
- the properties (leach resistance) of the product glass
- the melter processing rate
- the potential for production of hazardous gaseous emissions
- the chemical additive requirements and power consumption
- the soil pretreatment requirements.

2.0 DESCRIPTION OF TEST FACILITIES

The glassification system under study is a JHCM developed by the U.S. Department of Energy for treating highly radioactive wastes generated during the reprocessing of spent nuclear fuel. This section describes the JHCM process and facilities.

2.1 PROCESS DESCRIPTION

The melter operates by passing an alternating electric current between metallic electrodes separated by a pool of molten glass. At elevated temperatures, glass becomes a good electrical conductor and thus, serves as the resistance heating element for the furnace. The melting furnace is a refractory-lined chamber that resists corrosion by the glass and serves as an electrical isolator to permit joule heating of the glass.

The furnace is typically operated below 1250°C, although higher temperatures are possible with a change in the electrode material. Waste materials to be processed, either dry solids or liquids, are mixed with the appropriate glass formers, typically silica, borax, soda ash, and lime (or a preformulated glass frit). This mixture forms the basic glass structure that allows the inorganic waste materials to be dissolved. The mixing of glass formers and waste can be done either prior to feeding the melter or in the melting cavity itself.

As depicted in Figure 2.1 for the case where the feed is a slurry, waste materials are reacted inside the melter by heat from the glass pool. Water is evaporated and the waste materials are converted to oxides, releasing some gaseous species such as CO_2, NO_x, SO_2, and HCl. The ash from oxidation of the waste species is fluxed by the glass formers and is incorporated into the molten glass pool, which is periodically discharged to waste disposal canisters. In a full-scale system, the process is typically continuous with evaporation, decomposition, and fluxing occurring simultaneously in the furnace. The size of the molten glass pool is adjustable by design of the furnace, but is generally set to achieve a mean residence time of greater than 1 day. This

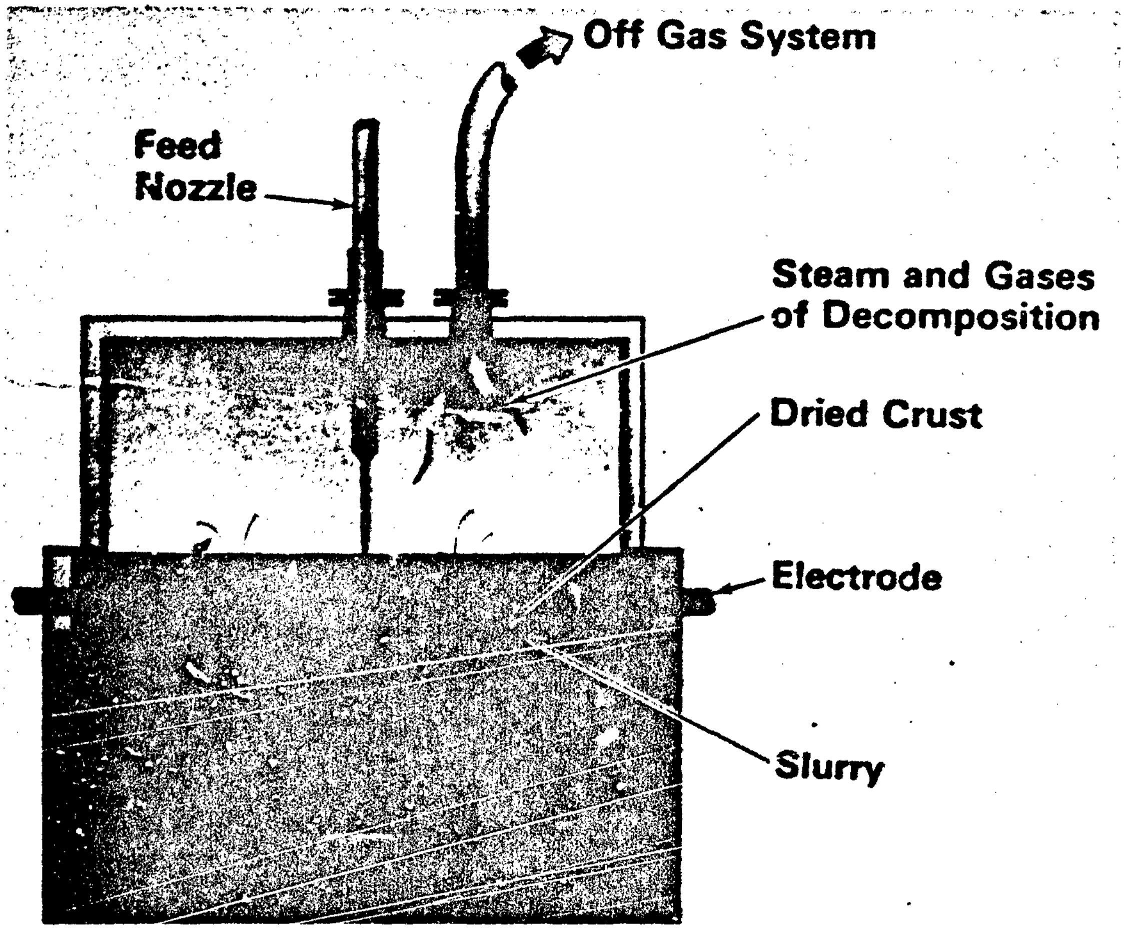

FIGURE 2.1. Glass Melting Process for Liquid Waste Treatment (Source: Battelle Northwest)

adjustment is made so a high quality homogeneous glass is consistently delivered to the disposal container and feed compositional variations are dampened out.

Several options are available for feeding waste and glass formers to the furnace, and the option chosen depends upon on the nature of the waste. For inorganic liquids and sludges, the best approach is to directly pump a concentrated slurry of the glass formers and waste material onto the glass surface using a feed nozzle that penetrates through the lid of the furnace. In this mode of operation a cold cap forms and covers the surface of the glass. The cold cap comprises three regions, from the top down: the boiling liquid zone, the dried solids calcination zone, and the fluxing or fusion zone. The dried solids calcination zone insulates the top layer from the fluxing or fusion zone where the melting reactions occur before the convective currents in the pool mix the material with the bulk glass. The slurry feeding mode of operation typically lowers the melter throughput by 50% for concentrated slurries since the furnace must now also serve as an evaporator.

The cold cap serves two beneficial functions. By covering the glass surface it acts as a refluxer for semivolatile species. These species have a high enough vapor pressure to escape from the glass at measurable levels. By refluxing these materials, their emissions may be reduced by as much as an order of magnitude and thus, the demand on the off-gas treatment system is significantly reduced if the species are hazardous. The cold cap also insulates the glass pool from the free air space or plenum above the pool. Lower off-gas temperatures and overall improved furnace efficiency is realized.

The same mode of operation is used for solid waste materials with the exception that the materials are delivered into the furnace through a feed tube. Since the unmelted feed is less dense than the glass product, the waste and glass formers float on top of the molten pool and also form a cold cap. This cold cap is similar to the one formed when feeding liquid except it has just the calcining and fluxing zones and lacks the liquid evaporation zone on top. Refluxing and lower heat losses are also realized with this cold cap.

The disadvantage of allowing a cold cap to form when processing solid wastes is that low-boiling-point organic compounds can be released to the off-gas treatment system without complete decomposition because of the relatively cool plenum temperatures. Consequently, in some cases, an afterburner or secondary combustion chamber may be required to complete the incineration process. Furthermore, feeding solids directly onto the glass surface for combustion could tend to increase the entrainment of fine particulates which must then be collected and possibly recycled by the off-gas treatment system.

An alternative feeding technique, specially developed for waste streams high in solid combustible materials, uses a feed tube which penetrates the glass surface and introduces the wastes within the molten glass pool. This technique allows for better material/glass contact and increased residence time at high temperatures. As the waste particles rise to the glass surface, they begin to pyrolyze and release low carbon chain gases that readily burn once exposed to the plenum air. The molten pool doubles as a first stage off-gas treatment by helping to capture the dust and ash materials like a wet scrubber. The vigorous bubbling action in the melt, primarily caused by the feed injection system, keeps the surface relatively free of a cold cap so plenum temperatures are high enough for combustion without auxiliary heating. Volatility and heat losses are also higher with this mode of feeding. Process rates are generally increased because of the more rapid heat transfer to the incoming waste feed.

The off-gas treatment system is also tailored somewhat to the specific waste stream. If the waste is a slurry or contains a large amount of moisture, the first-stage treatment is commonly a wet scrubbing operation to condense the liquid and capture the bulk of the entrained particulate. The condensing action of the water vapor in the off-gas stream as it is treated in the scrubber helps to improve the particulate removal efficiency. If necessary, the off gases will then be further filtered and treated to remove acid gases and fumes before release to the atmosphere.

2.2 TEST EQUIPMENT DESCRIPTION

The major purposes of the test program were to demonstrate feasibility and to collect sufficient data for preparing a preliminary process flowsheet and cost estimate that would compare glassification and other technology alternatives being considered to treat the RMA Basin F material. The small-scale continuously fed, joule-heated experimental melter known as the ESCM (Figure 2.2) was judged to be well suited to treat the RMA Basin F material.

This melter has a nominal glass volume of approximately 8.2 L (0.29 ft^3) with an exposed glass surface area of 0.054 m^2 (0.58 ft^2) and a typical

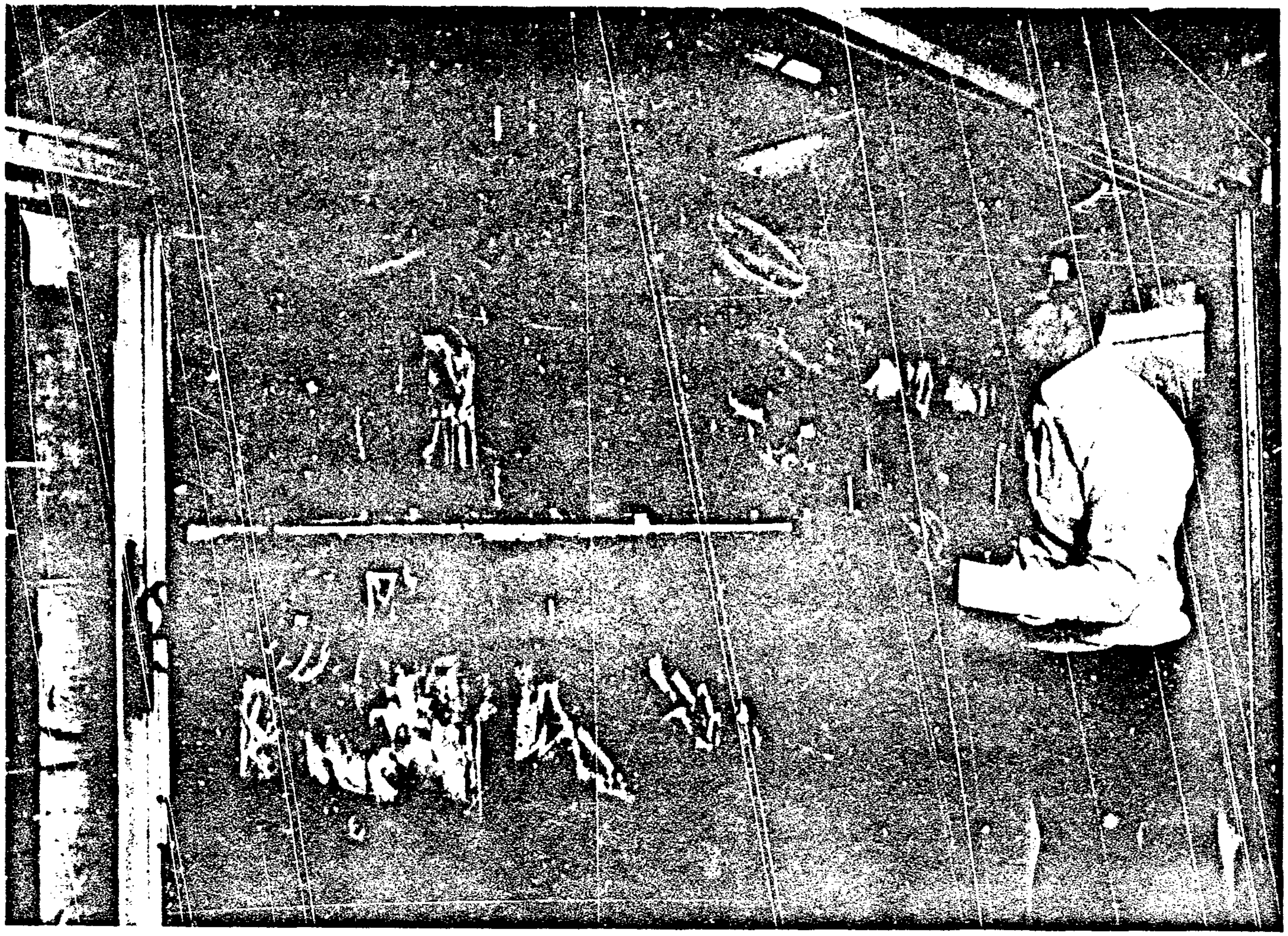

FIGURE 2.2. ESCM High-Temperature Furnace
(Source: Battelle Northwest)

operating glass depth of 15.2 cm (6.0 in.). Energy needed to convert solid material to glass, as well as to maintain operating temperatures in the melter, is supplied by alternating current passed between two Inconel 690® plate electrodes. Glass temperatures are maintained in the range of 1150° to 1250°C by manually controlling power input to the electrodes. A thermocouple located in one of the electrodes is used to monitor temperature within this melter. The electrode temperature typically operates 100° to 150°C below the glass temperatures in the center of the melting tank so the control point is 1050° to 1100°C. Two factors contribute to the lower electrode temperature: one side of each electrode is in direct contact with the cooler furnace walls; a large portion of the electrode extends above the melt level and serves as a cooling fin that radiates heat to the vapor space above the melt. Operating conditions in the melter are monitored and recorded on continuous strip chart recorders.

The inside of the furnace is lined with a high density refractory brick composed of chromium and aluminum oxides that resists corrosive attack by the molten glass. Additional layers of insulating refractory materials provide backup to prevent glass leakage and reduce heat losses. The refractories are surrounded by a sealed stainless steel shell that controls air inleakage and allows the melter to be operated under a partial vacuum to collect and control off gases generated by processing hazardous materials.

The melter is connected to an off-gas treatment system that provides the source of vacuum and removes hazardous species present in the melter exhaust. Pressure in the melting cavity is maintained at a partial vacuum of less than -2.5 cm water column (-1 in. W.C.) to contain the exhaust gases.

Prior to release to the environment, the off gas is routed through an ejector-venturi wet scrubber to remove large particulates and condensable vapors (including soil moisture). The off gas is then passed through a heat exchanger, a demister pad, a high-efficiency particulate air (HEPA) filter and an activated carbon filter prior to discharge (see Figure 2.3). The HEPA filter removes submicron particulates and aerosols while the carbon filter

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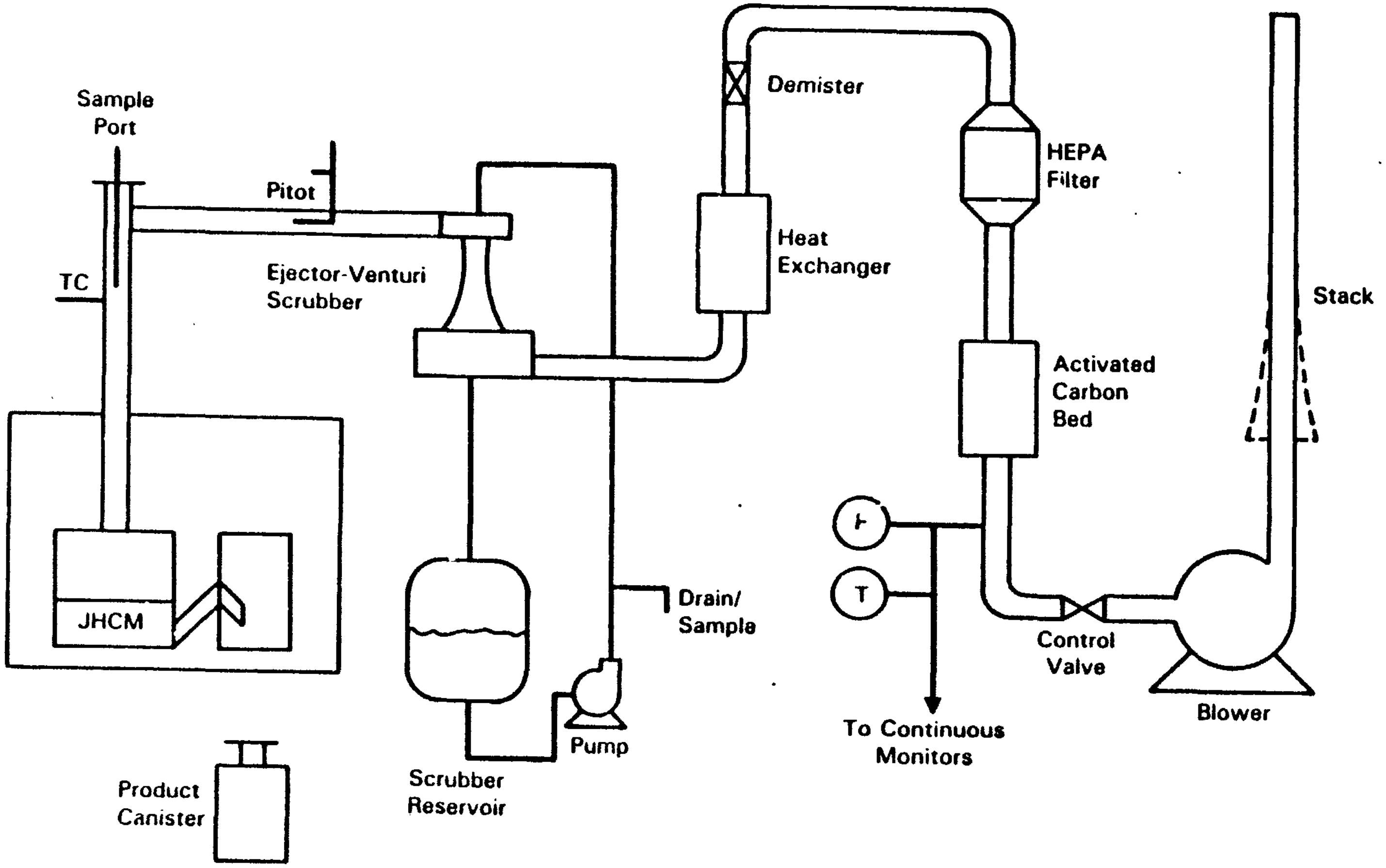

FIGURE 2.3. ESCM Off-Gas Treatment System
(Source: Battelle Northwest)

collects organic vapors. Condensate that builds up in the scrub water recirculation system is drained from the scrubber reservoir for disposal.

The exhaust line from the melter is a 7.6-cm (3-in.) schedule 40 pipe which is reduced to a 5.1-cm (2-in.) pipe after the gas is quenched through the ejector-venturi wet scrubber. From the melter exhaust port on the melter lid, the off-gas line rises vertically for about 56 cm (22 in.) to a tee in the line. The off-gas line then makes a 90° turn and runs horizontally for 132 cm (52 in.) to the inlet of the ejector. The off-gas sample probe was positioned in the center of the vertical section, 20 cm (8 in.) upstream of the tee. The Pitot tube used for flow measurement was positioned 61 cm (24 in.) downstream of the tee in the horizontal section of pipe. Positioning of the Pitot tube on the vertical centerline of the pipe was established before the test to correspond with the point where the velocity matched the measured velocity at the sample point under the expected flow conditions. This resulted in a position slightly above the pipe center.

Positioning the sample probe in the vertical section of the pipe allowed the gas stream to be sampled as close to the pipe entrance as was reasonable to minimize the loss of particulate through impaction with the pipe wall. The Pitot tube was positioned further downstream of the line entrance to more accurately measure flow rates. A thermocouple inserted through the off-gas line into the gas stream measured the gas temperature near the sample nozzle.

The sample point selected for the continuous monitoring of noncondensable gas species was located downstream from the wet scrubber and filters. This location was selected so the gas would be dry and free of contaminants which might otherwise damage the analyzers. The removal efficiency for NO_x, CO, and CO_2 gases by the water scrubber was judged to be low.

Near the floor of the melting cavity is an opening, through the refractory wall, which leads to a separate riser and pour trough for draining glass. When the level of the melter approaches its natural overflow depth of 15 cm (6 in.), the glass level is lowered a few centimeters by the transfer of the molten glass through this drain system to steel canisters. Resistance heating elements located in the vapor space surrounding the trough maintain the glass stream above 1000°C to achieve acceptable pouring behavior. A view port on the front of the melter allows direct access to the pour stream for monitoring the pour and collecting glass samples.

The feed system for delivering dry waste feed material to the melter is located on the level above the melter inside a ventilated plastic hood known as a temporary greenhouse. The greenhouse isolates the hazardous materials from the main building while they are transferred from their shipping drums to the sealed feed hopper. The feeder is a belt-driven gravimetric system which meters material from the hopper to a nominal 5.1-cm (2-in.) diameter pipe feed tube as shown in Figure 2.4. Solids fall by gravity onto the glass surface through the feed tube. The feed tube is sealed where it penetrates the lid of the melter to minimize inleakage and is adjustable in length to permit lowering it below the melt surface for submerged feeding. The capability exists for air injection through the feed tube to aid the submerged feeding mode and to introduce excess combustion air.

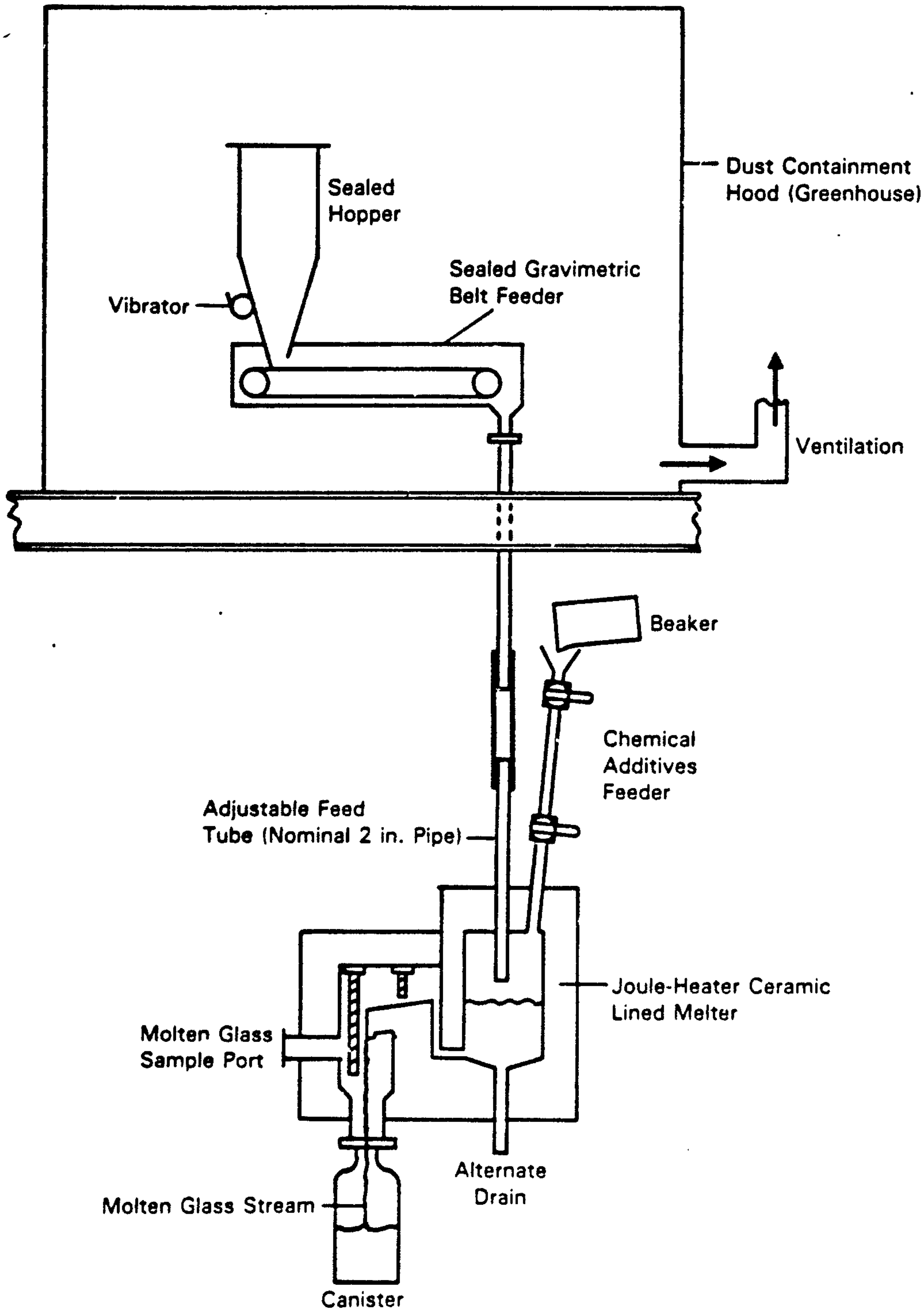

FIGURE 2.4. ESCM Feed Delivery System Schematic (Source: Battelle Northwest)

3.0 TEST PROGRAM PROCEDURE

This section discusses the test program procedure, including glass formulation, soil preparation, equipment shakedown tests, general test operation, data and sample collection, and off-gas sampling methodology.

3.1 GLASS FORMULATION

Samples of the Basin F contaminated material were provided in 4, partially filled 55-gal drums. The drums were labeled: "RMA Basin F Composite 87014 Soil," and were sequentially numbered N5883 #13 through N5886 #16. A small sample was collected from the surface of Drum #13 and analyzed for inorganic composition using ion coupled plasma spectroscopy. In addition, soil from an uncontaminated region adjacent to the Basin F was also obtained and analyzed. Both samples showed similar chemical composition for the major glass-forming constituents and so, to avoid special handling requirements, the uncontaminated soil was used for all laboratory crucible-scale melting work in order to formulate the product glass.

The main objective of this effort was to lower the melting temperature of the soil so it could be processed efficiently below 1200°C in the bench-scale furnace. Some borax, instead of soda ash alone, was added to lower the glass viscosity and improve its ability to dissolve the refractory waste materials without dramatically decreasing the electrical resistance and chemical durability of the product. Lime could also have been used to improve fluidity and product durability, however, this was viewed as an optimization step and outside the required scope of work. The glass formulation work was intended to be a minor effort at this level of study and so, only a few simple glasses were tested.

As shown in Table 3.1, the material contains mostly silica and alumina. Additional fluxes in the form of soda ash and borax were needed to lower the melting temperature and generate a suitable glass composition. Several crucible-scale melts with various combinations of glass-former additives and uncontaminated soil were made. A mixture consisting of 65 wt% dry soil, 20 wt% Na_2O, and 15 wt% B_2O_3 was selected.

TABLE 3.1. Element Composition of Background RMA Soil Sample (wt%) (Source: Battelle Northwest)

Element	wt%
Aluminum	6.60
Boron	0.06
Barium	0.08
Calcium	0.58
Copper	0.04
Iron	2.76
Potassium	1.9
Magnesium	0.58
Manganese	0.08
Sodium	1.31
Silicon	31.4
Tin	0.02
Titanium	0.30
Zirconium	0.02
Others[a]	54.3
	100.03

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[a] Includes oxygen, organics, nitrates and other trace elements.

3.2 SOIL PREPARATION

In order to test the handling, blending, and feeding characteristics, a mixture of 75 wt% local Hanford sand and 25 wt% bentonite clay was prepared to simulate the Basin F material. Based on a moisture analysis of the soil sample from one of the drums, 20 wt% water was added to the simulated soil mixture. Blending of this mixture using a barrel tumbler created a solid mass that stuck firmly to the walls of the drum. When the sample from Drum #13 (actual Basin F soil) was also mixed on the barrel tumbler, it behaved similarly.

The simulated Basin F soil was removed from the 55-gal drum and placed on plastic sheets to air dry for several days. This material was then tumbled again in the barrel until a free flowing granular material with only a few small (pea-sized) soil agglomerates was produced. This material was judged to produce desirable feeding characteristics and so was adopted as the reference pretreatment procedure.

Following the same procedure, roughly equal parts from each drum of actual Basin F soil were spread on plastic sheets in a well ventilated room and allowed to dry for 4 to 5 days. During all handling of the Basin F material, personnel wore protective clothing and fresh-air breathing apparatus. Material from Drum #13, which had previously been tumbled and appeared to have the highest moisture content, remained in large pieces when removed from the drum. The large pieces were broken up with a shovel to speed up the drying of material from Drum #13.

The air dried soil was then placed in a 55-gal drum along with several large river rocks and tumbled for 5 hours. The rocks were added to help crush any remaining soil agglomerates. The resulting soil consisted of a fine powder that was largely homogeneous. A 1-L sample jar was then collected from the top surface soil and analyzed in the Arthur D. Little, Inc., USATHAMA-certified laboratory for the target hazardous species. Results of the analysis are presented later in this report. This drum of pretreated soil served as the feed material for the bench-scale test.

3.3 EQUIPMENT SHAKEDOWN TESTS

Equipment shakedown tests were performed to establish operating parameters and the feeding technique for the controlled experiment. These tests were typically short runs of several hours duration each using the simulated Basin F material rather than the actual Basin F material to prevent contamination of the test apparatus until all major equipment and procedure problems were identified and corrected.

Testing started by feeding simulated Basin F material directly onto the glass surface with the feed tube exit point positioned about 6 in. above the melt. During this test the glass former fluxes were mixed directly with the simulated feed prior to feeding. Waste feeding onto the melt surface resulted in noticeable entrainment of fine powder type particulate by the off gases.

The off-gas wet scrubber solution quickly became clouded with entrained particulates and the presence of heavy dust in the melter plenum space was obvious shortly after the feeding operation started.

Feeding was changed to the submerged drop tube technique to reduce the dusting problem. With the glass formers and simulated Basin F material mixed together, the drop tube rapidly plugged. Examination of this plug revealed premature partial melting of the mixture and subsequent sticking to the inside of the feed tube. The batching of glass-former fluxes onto the surface through a separate feed tube largely eliminated the plugging problem but again resulted in glass former entrainment. Without the glass formers, feed rates up to about 1.5 kg/h (3.3 lb/h) of simulated Basin F material could be maintained without plugging.

With a full-scale feed system and subsequently larger diameter feed tube the noted plugging is not expected to be a problem. Material that sticks to the walls of the feed tube should drop off before it reaches a significant thickness. In this small-scale demonstration however, it became necessary to feed the glass formers as a separate stream outside the soil feed tube.

The exit point of the special glass former feed tube was nearly 61 cm (2 ft) above the melt surface on the inside of the lid. The lower temperature in this region along with the change to batch feeding helped to eliminate plugging of this feed tube. Plugging was only experienced when the fluxes were metered at a slow rate into the melter. Batch feeding every 15 min was done by quickly opening the feed hopper valve to avoid plugging problems. The entrainment of the glass former fluxes appeared to be acceptable when operated in this batch mode even though they were released relatively high above the surface. To further reduce the dusting, a small amount of water was added to the glass formers. This procedure involved spreading the dry materials on a plastic tarp and using a fine water spray gun to dampen the surface. When the surface dried, the glass formers were stirred and the procedure was repeated a few times.

For the greatest residence time and thus, best organic destruction and removal, the feed tube should be submerged as far as possible below the glass surface. In the ESCM this is equivalent to about 12.7 cm (5 in.) below the glass surface. Operation of the feed tube at this submerged level requires a pressure differential between the feeder and melter plenum of 30 to 38 cm (12 to 15 in.) W.C. Since the feeder was not intended to be pressurized, some modifications were necessary to allow internal pressures of a few inches of water to be used. Higher pressures were avoided because they resulted in significant dust release from the feeder. The thin (3 mm) plexiglass window in the door of the feeder also limited the safe operating pressure to a few inches W.C. to prevent a rupture.

Because the ESCM off-gas system could not maintain a partial vacuum of more than about -25.4 cm (-10 in.) W.C. in the melter plenum, the feed tube submergence was reduced to 5 cm (2 in.) below the melt surface. Feeding was easily controlled at this submergence by maintaining a partial vacuum of -7.6 to -12.7 cm (-3 to -5 in.) W.C. in the melter and then adjusting the feeder internal pressure using air injection to maintain active bubbling from the feed tube. The pressure of the melter and feeder required periodic manual adjustment to correct for changes in the glass level, but these were simple and easily accomplished. The low internal feeder pressure prevented excessive dusting problems.

3.4 GENERAL TEST OPERATION

Basin F material was metered into the feed tube with a belt-driven gravimetric feeder. The feeder and feed hopper were located in a well-ventilated greenhouse structure to prevent the spread of contaminated dust. While inside the greenhouse, operators wore protective clothing and fresh breathing air apparatus. Operators transferred contaminated soil from the 55-gal drum to the feed hopper by scooping it into 5-gal cans for weighing and sampling and then dumped it into the conical-shaped hopper. The hopper was subsequently sealed so the system could be pressurized.

Material in the hopper flowed by gravity onto the moving belt. A weighing system in one section of the belt provided a feedback signal to the automatic mass rate controllers. As the material fell from the end of the belt, it was funneled into the 6.35-cm (2.5-in.) -diameter feed pipe. A pneumatic-driven vibrator secured to the throat of the feed hopper maintained the steady flow of material onto the moving belt. Without the vibrator, soil occasionally stuck in the throat and left gaps on the belt. Prior to the test, the automatic feed rate control was verified by metering material into a bucket. At the end of the test all remaining material was removed from the feeder and reweighed to determine the total mass of material processed and to recalibrate the feed control set point.

The separate glass former flux feeder system consisted of a double valve air lock assembly on the lid of the melter. During operation, the glass formers were added manually every 15 min.

Technical grade chemicals were used to supply the necessary glass former fluxes. The soda ash and borax were added in the correct proportions to a steel drum and blended with the drum tumbler. A sample of this mix was collected with a scoop from the surface after blending.

During Basin F material processing through the melter, the required quantity of glass formers was weighed and added to the feed chamber with the top valve open and the lower one closed. After closing the top valve, the lower one was quickly opened to dump the batch into the melter plenum where it fell to the melt surface. When the valve was opened slowly, the fluxes tended to stick and plug on the wall of the short pipe section leading through the melter lid. A momentary loss of negative pressure in the melter was experienced during this operation as residual air in the feeding chamber was evacuated to the melter, and the glass formers rapidly decomposed in the melter, which released some CO_2 and water vapor.

This technique of feeding glass formers and Basin F feed material to the melter separately was imprecise and produced larger variations in the glass product composition than would normally occur in a glassification process. Since close control of the product glass composition was not considered necessary for this application, the simplest, most direct approach was adopted.

Adequate product quality control was judged to be possible through a simple visual assessment of the glass pouring behavior during canister filling and product sampling. When the operator observed the glass becoming obviously more or less viscous, the glass former additions were appropriately adjusted until the problem was corrected.

The glass pouring operation was controlled by injecting air in the overflow riser section of the melter. This technique of glass transfer is known as airlifting. Approximately every 2 hours during normal operation, the airlift was turned on to a set value and allowed to run for about 15 min. The basis for selecting this pouring frequency was to empty between 1.3 to 2.5 cm (0.5 to 1 in.) of glass in the melter in order to minimize system perturbations. A 2.5 cm (1-in.) level change is equivalent to about 3.5 kg (7.7 lb) of glass.

Because the glass pumping rate is strongly affected by the submergence of the air injection point and the air rate, this method of operation tends to self-regulate. If the glass level was low from the previous pouring or from not processing feed, the pour rate would automatically be reduced, and consequently, not as much glass would be transferred during the 15-min pouring period. The reverse was true in the case of a high glass level and so, the level variations were dampened out. As a backup, the operators would visually monitor for the initiation of glass pouring by gravity overflow. This condition would indicate glass had reached the high control point and airlift operation should be started. The bubbling behavior of the feed tube when it was operated in the submerged mode was a secondary glass level indicator.

The melter pressure was controlled by a valve downstream of the off-gas treatment equipment. A differential pressure gauge connected across the melter plenum space and the atmosphere served as the control point for manually adjusting the valve as required. Three sources of air contributed to the melter off-gas volume in addition to the gases generated by the decomposition and drying of the Basin F feed. Air from inleakage around the various melter flanges and the injection air used to operate the submerged feed tube accounted for part of the off gas. The major air source was however, a constant 10-standard cubic feet per minute (scfm) source injected at the entrance to the off-gas pipe. This air was added to stabilize flow fluctuations, simplify the vacuum control, and increase velocity in the off-gas pipe so it could more accurately be measured for material balance purposes. Although this had the result of reducing the concentrations of species in the off gas, sufficiently long sampling periods were used to collect an adequate sample. The dilution air also provided added assurance that flammability limits for combustible gases were not reached in the off-gas piping if incomplete combustion occurred.

The test was started using the submerged tube feeding technique. In less than 1 hour of operation the stack gas sampling filter was loaded with particulate and had to be replaced. The lack of a cold cap with this mode of operation and the vigorous bubbling action of the submerged feed tube presumably combined to strip large quantities of semivolatile salt compounds from the melt. These salts condensed in the off-gas steam to form small particulates that easily passed through the gas sampling system cyclone. Removal and recycle of these materials from the melter exhaust should be easily accomplished with conventional filtration or wet scrubbing techniques; however, for the purpose of this feasibility test, the particulate level was judged to be unacceptably high and prevented reasonable off-gas sampling. Feeding was consequently changed to the surface feeding technique and a thin cold cap of unmelted Basin F material was allowed to form on the melt surface. The pressure in the melter was reduced to about 2.5 cm (1 in.) W.C. so that inleakage and thus, feed entrainment, would be minimized. Melter power was controlled to maintain an electrode temperature above 1050°C and below 1100°C and subsequently a glass temperature between 1150° and 1250°C.

3.5 DATA AND SAMPLE COLLECTION

Operational parameters that describe the experimental conditions, such as feed rate and electrode power, and are used to control the process were recorded by continuous strip chart recorders or on data sheets. Hazardous materials present in the melter exhaust gas were sampled by a combination of two standard U.S. Environmental Protection Agency (EPA) sampling trains, Modified Method 5 (MM5) and Method 101A, and a series of continuous gas analyzers [CO, CO_2, O_2, SO_2, and NO_x compounds (nitric oxide and nitrogen dioxide)].

The process streams identified in Figure 3.1 were sampled and the total quantities processed or produced were determined for overall material balance purposes. With the exception of glass product samples and sample train

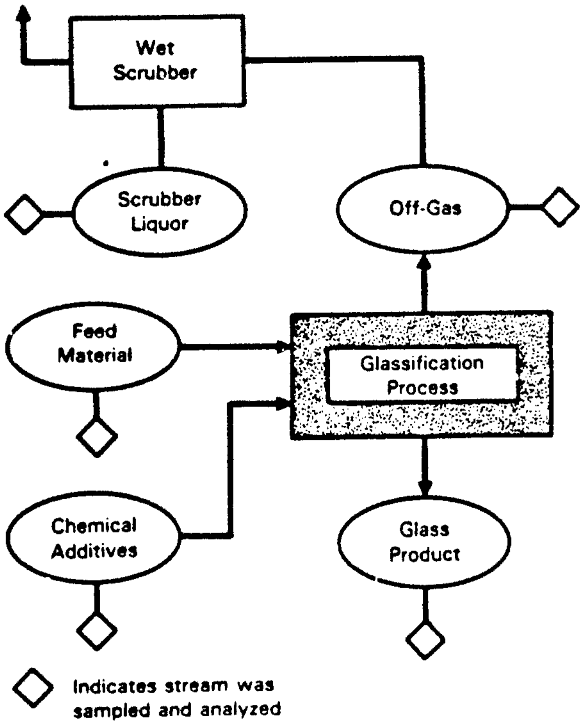

FIGURE 3.1. Sampling Plan for Glassification Test (Source: Arthur D. Little, Inc.)

filters, all solid and liquid samples were placed in glass bottles with Teflon®-lined lids. Each sample container was precleaned, using a soap-and-water wash, a distilled-water rinse, and an organic-solvent wash. The gas sampling train filters were placed in glass petri dishes and the glass product grab samples were placed in paper envelopes. The MM5 organic sorbent resin remained in the sorbent module for shipment to Arthur D. Little, Inc. analytical laboratories.

3.5.1 Basin F Material

Material received from Basin F was blended as previously described. Following blending, a 0.5-L sample was collected in a 1-L jar by simply scooping

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the surface. A second sample was collected when Basin F material was transferred to the feed hopper. Multiple samples were collected and combined to make up a composite sample.

3.5.2 Chemical Additives

A single batch of glass formers was blended with the drum tumbler and then sampled using the same method as described for the Basin F material sample.

3.5.3 Melter Glass

Glass removed from the melter using a batch pour method was sampled with a graphite sampling boat inserted directly in the pour stream. The sample, in the form of an ingot (approximately 20 g), was allowed to cool and removed from the boat. Three samples were collected during each of the seven pouring periods for the test.

3.5.4 Off-Gas Scrub Solution

A sample of the off-gas wet scrubber solution was obtained using a tap on the pipe that recycled the collection reservoir solution to the scrubber. This process stream was sampled for general comparison purposes and to obtain an approximate estimate of the wet scrubber effectiveness in removing hazardous species from the melter exhaust. The scrubber and collection reservoir were drained before starting the test, and fresh water was added. After circulating the solution for more than 5 min, a sample was collected and the quantity of solution in the reservoir was recorded. At the end of the test, a sample was again taken and the solution quantity was determined.

3.5.5 Off Gas

Sampling of the off gases generated during the processing of the Basin F material were accomplished using the equipment and methods outlined in Table 3.2.

The MM5 sampling train was used to collect semivolatile organics, nonvolatile organics (constituents with boiling points >100°C), and acid gases (hydrochloric, hydrofluoric, and phosphoric) for subsequent analysis. Particulate

TABLE 3.2. Off-Gas Sampling Equipment

Off-Gas Component	Equipment/Method
Particulate, Semivolatile and Nonvolatile Organics, Acid Gases	EPA MM5 sampling train
Semivolatile, and Nonvolatile Metals	EPA Reference Method 101A
CO	On-line analyzer, Infrared Industries Model IR-703-D-445
CO_2	On-line analyzer, Infrared Industries Model IR-703-D-355
O_2	On-line analyzer, Infrared Industries Model IR-2100
NO/NO_x	On-line analyzer, Beckman Model 953
SO_2	On-line analyzer, Beckman Model 951A

emission rates were also determined using this method. The EPA reference Method 101A system was used to collect both nonvolatile and semivolatile (e.g., mercury) metals.

3.6 OFF-GAS SAMPLING METHODOLOGY DETAILS

A description of the equipment and procedures used in association with these sampling trains is presented in this section.

3.6.1 Modified Method 5 Train

The MM5 sampling train was the primary extractive and concentrating sampling system used for sampling of the glass melter off gas. This system is based upon the design of units that normally are employed for EPA reference Method 5 sampling. The modified system includes a sorbent module filled with a porous polymeric resin XAD-2®, which permits trapping of semivolatile organic vapors.

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A stainless steel probe unit with a sharp, tapered leading edge was inserted into the off-gas line upstream of the ejector-venturi scrubber. The sample line was heat traced from the sample port to the cyclone unit to maintain the gas temperature above 120°C. A standard Pitot tube and thermocouple sensor installed in the off-gas line near the probe unit monitored the melter off-gas flow rate and temperature respectively. A glass cyclone was included in the train to remove large particulates and prevent plugging of the downstream filter.

The cyclone was followed by a high-efficiency glass filter to collect fine particulates passing through the cyclone. The cyclone and filter were enclosed in an electrically heated box that was thermostatically maintained at a temperature of 120°C to prevent any water condensation in this section of the train.

Before reaching the sorbent resin, the sampled gas was cooled to a temperature of 20°C (70°F) or less. This cooling operation resulted in the condensation of water vapor. Water condensation may have caused some of the organic vapor to collect with the condensate. For this reason, the condensate was allowed to percolate through the resin bed prior to being discharged into the knock-out trap located below the sorbent module. A diagram of this system is shown in Figure 3.2.

At the downstream side of the sorbent module (e.g., downstream of the knock-out trap) were four impingers connected in series and immersed in an ice bath. The first and second impingers, connected to the outlet of the knock-out module, were filled with scrubbing solution. A caustic solution of 0.5 M sodium bicarbonate was used to collect hydrochloric and other acid gas species. The third impinger remained dry to trap any entrained water droplets. The fourth impinger was filled with silica gel to absorb any residual moisture contained in the sampled gas. Moisture removal was important to ensure accurate gas flow measurements and to prevent damage to the pumping system.

3.6.2 Nonvolatile and Volatile Metal Sampling Train

The EPA reference Method 101A sampling train was required to collect both nonvolatile (copper, iron, manganese) and semivolatile (mercury, arsenic) metals. This system is essentially equivalent to the MM5 system described

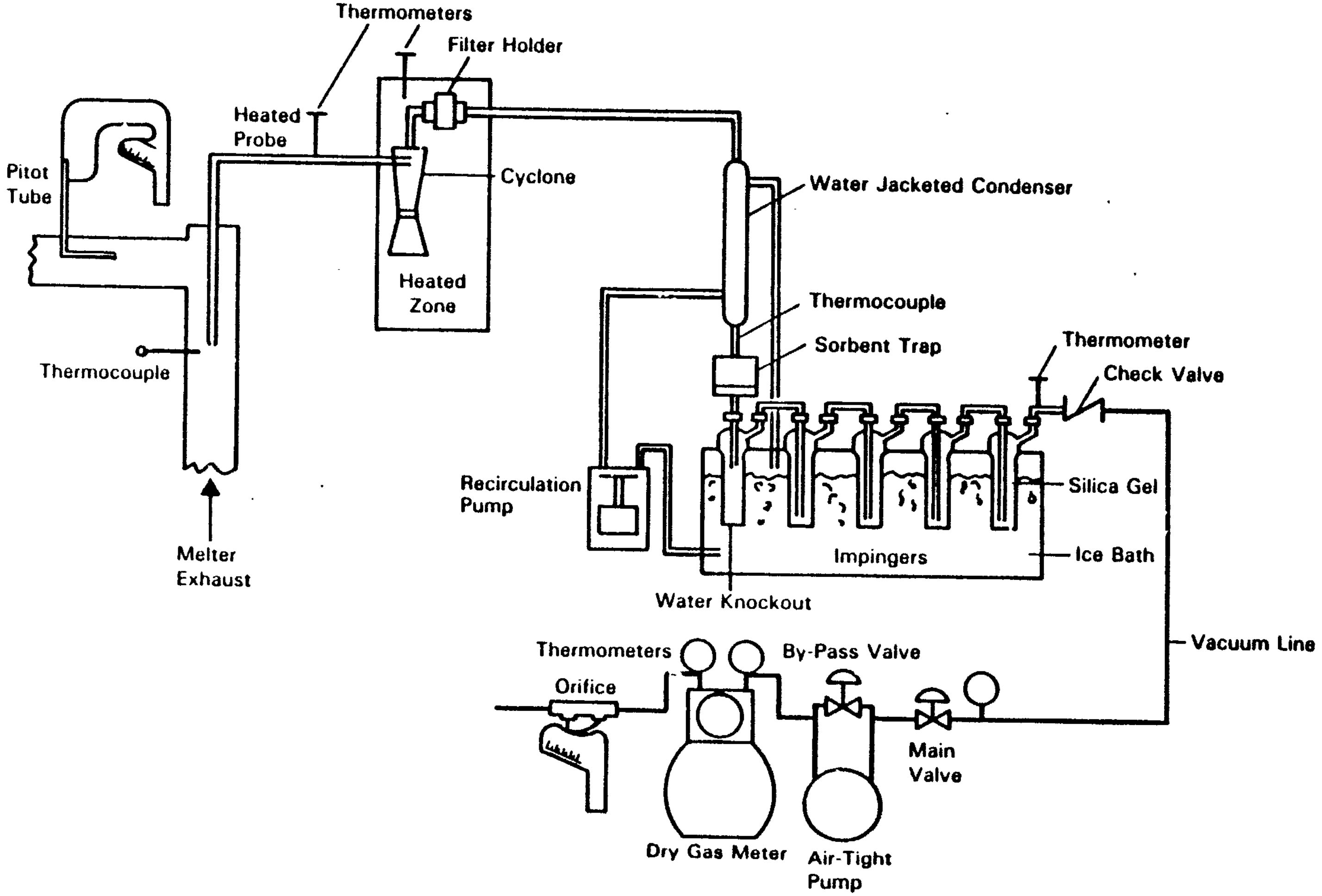

FIGURE 3.2. Modified Method 5 Sampling Train (Source: Arthur D. Little, Inc.)

above, with the principal difference being that the sorbent module (e.g., condenser, sorbent trap, and knock-out trap) was eliminated. Once the sampled gas passed through the heated high-efficiency fiber filter, where the nonvolatile metals were removed, it passed directly into the impinger that was charged with a 4% potassium permanganate in sulfuric acid solution. The second impinger was also charged with the 4% potassium permanganate solution. The third impinger was left empty again, while the fourth impinger was charged with silica gel.

3.6.3 Sampling System Operation

Normally, samples obtained using either the MM5 or Method 101A systems would be obtained in a 12-in.-diameter or larger exhaust duct by compositing sample aliquots from a number of traverse points. Each aliquot removed is obtained at a rate that is identical (within a ±10% tolerance) to the locally prevailing gas velocity, and is measured by a Pitot tube firmly affixed to the sampling probe.

As this program was undertaken to develop data that broadly assess the capabilities and efficiencies of the candidate treatment technology, rather than compliance testing, and involved bench-scale equipment with a 3-in. exhaust line, minor sampling liberties were considered acceptable during the performance of the work. The foremost of these liberties was that the sampling system was not conducted utilizing full discharge duct traverses. Instead, samples were obtained from a single point. This point was located at a position typical of the average velocity measured during preliminary velocity traverses.

The samples were obtained following procedures developed to collect a sample isokinetically. The linear velocity present at the selected sampling point was determined and periodically monitored, and the sampling system was operated to assure that the sampled gas entered the collection train at a rate that was close to isokinetic conditions. The sample system was actually operated at 75% of isokinetic conditions because of an initial error in calculating the off-gas flow rate from the Pitot tube data. This condition led to a higher proportion of large particulate entering the sample nozzle and so the particulate mass loadings were conservatively high.

3.6.4 Sample Train Precleaning

Prior to the use of either comprehensive sampling system, all sample gas wetted components were cleaned. The sample cleaning procedure required for all components of the MM5 sampling system was specially designed to reduce likely organic contaminants. The cleaning procedure for use with the Method 101A system was designed to reduce metallic species contamination. For the MM5 precleaning system all surfaces were

- washed with soap and warm water
- rinsed with tap water
- rinsed 3 times with Type I deionized, distilled reagent-grade water
- rinsed with methanol
- rinsed with methylene chloride.

For the Method 101A System all surfaces were

- rinsed with 50% HNO_3
- rinsed with tap water
- rinsed with 8M HCl
- rinsed with tap water
- rinsed 3 times with Type I deionized, distilled reagent-grade water.

4.0 TEST RESULTS

This section discusses results for the general system performance, contaminant destruction/immobilization efficiencies for target species, noncondensable gas analysis, product leach testing, acid gas evaluation, and particulate loadings.

4.1 GENERAL SYSTEM PERFORMANCE

Immediately prior to starting the test, the off-gas sampling train was connected to the melter exhaust and Basin F material was fed to determine moisture content of the off gas for use in establishing isokinetic sampling conditions. The sample train was then disassembled and cleaned. Residual material in the feed hopper was removed and a preweighed quantity was added. Feeding was restarted, and after approximately 1 hour when the sampling system leak checks were complete and melting conditions were stabilized, the sample probe was installed in the off-gas line and the MM5 train started. The MM5 train was run for a total of 377 min during which time 13.6 kg (30 lb) of air dried Basin F material was processed. Each kilogram of Basin F material resulted in about 1.5 kg of glass product being formed. The average glass production rate during this portion of the test was therefore, 3.2 kg/h (7.0 lb/h).

The MM5 sample train filter required replacement twice because of excessive pressure drop conditions. Table 4.1 summarizes the sampling conditions for each of three periods. Period 1 lasted only 25 min as a high loading of fine, light yellow particulate occurred in the melter off gas. As previously discussed, the feeding technique was changed from the submerged drop tube method to directly feeding on the melt surface for the remainder of the test.

Stack gas velocities were reduced after the first period because of the decrease in melter vacuum and the lowering of off-gas temperatures with the formation of a small cold cap. The vacuum was reduced to lower the dusting problems. With the change in feeding technique, filter life was extended to 237 min for the second period. The third filter period was terminated when the total sample volume exceeded the target level of 3 dry standard cubic meters (dscm).

TABLE 4.1. Summary of Sampling Conditions for MM5 and Method 101A (Source: Arthur D. Little, Inc.)

	MM5			
	Filter 1	Filter 2	Filter 3	101A
Nozzle Diameter (in.)		0.4976		0.4976
Barometric Pressure (in. Hg)				29.42
Net Sampling Time (min)	25	237	115	133
Sample Volume (ft^3)	10.62	71.968	31.939	34.306
Average Dry Gas Meter Temperature (°F)	80	81.3	83.9	90.4
Average Pressure Drip at Orfice (in W.C.)	0.616	0.309	0.255	0.215
Dry Gas Meter Accuracy		1.037		1.027
Volume of Water Collected (mL)		60.2		22.8
Volume of Water Vapor at STP (ft^3 at 68°F, 29.92 in. Hg)		2.838		1.075
Volume of Dry Gas at STP (ft^3)	10.561	71.094	31.463	33.251
Moisture (%)		2.45		3.13
Dry Molecular Weight of Stack Gas[a] (lb/lb-mole)		29.252		29.252
Wet Molecular Weight of Stack Gas (lb/lb-mole)		28.976		28.900
Pressure of Stack Gas (in. Hg)	29.306	29.301	29.266	29.141
Average Stack Temperature (°F)	306	205	194	178
Average Stack Gas Velocity (ft/min)	629	389	357	319.7
Isokinetic (%)	75.5	75.2	73.7	74.2

(a) Assuming 19.5% O_2, 1.2% CO_2, 0.2% CO, 80.1% N_2.

At the completion of the MM5 sampling period, the gas sampling system was removed and cleaned to recover any material coating the inside of the lines. This same system was then reinstalled without the organic sorbent trap and used for the Method 101A sampling period. Table 4.1 summarizes the test conditions during this period. Only one filter was required since the total sample time was only about one third as long (133 min). Based on the concentration of metal species in the feed stream and their expected volatility, a sample volume of about 1 dscm was considered adequate.

Glass production rates were slightly lower, at approximately 2.5 kg/h (5.5 lb/h), during this sample period. Feed rates were intentionally lowered to prevent the cold cap from becoming too large. During both sampling phases of the test (MM5 and Method 101A) the feed rate was adjusted by observation of the unmelted feed pile (cold cap) conditions in the melter. Operators attempted to maintain a condition where the cold cap remained with an orange glow for about 2 to 3 cm around the perimeter. Since the melting cavity is rectangular, with dimensions of 36 cm long from the face of each electrode by 15 cm wide, the orange glow tended to be wider near the electrodes. This control condition was selected to maintain high enough vapor space temperatures for combustion of organic vapors. Under this condition flames on and just above the cold cap surface were routinely observed.

Heat stored in the plenum refractories may have helped to maintain higher processing rates initially, but as the test proceeded and the refractories cooled, the cold cap slowly increased so the feed rate had to be reduced. The gradual decrease in stack gas temperatures during the test are indicative of this condition. Processing conditions appeared to stabilize after the feed rate was reduced for the 101A sampling period.

4.2 BASIN F MATERIAL ANALYSIS

Analytical results for the two Basin F material samples collected after the material was air dried and blended with roughly equal parts from each of the four drums is presented in Table 4.2. Included for comparison are the results from two other samples (Ogden and MTARRI), that represent material collected from roughly the same area of Basin F. These samples were sent by Arthur D. Little, Inc. to other companies for use in tests with two other treatment technologies under consideration. The sample RMA 1019 more accurately describes the Basin F material used during the test since it is the composite sample collected when the material was added to the feed hopper. By comparison, the Basin F material used for the glassification tests at Battelle is significantly higher in most hazardous constituents. The higher concentrations, including the higher chloride content, which was nearly an order of magnitude higher, might suggest that more free standing liquid from Basin F was

TABLE 4.2. Analysis of Basin F Feed Material and Glass Formers (Source: Arthur D. Little, Inc.)

	Ogden	MTARRI	Battelle	RMA 1019(a)	RMA 1020(b)
Organochlorine Pesticides, µg/g					
Aldrin	1700	990	3800	3100	ND(<0.0503)
Isodrin	65	42	130	110	ND(<0.0825)
Dieldrin	620	460	1700	1300	ND(<0.726)
Endrin	82	50	260	180	ND(<0.500)
Organosulfur Compounds					
Dithiane	ND(<18.8)	ND(<18.8)	ND(<18.8)	ND(<1.88)	ND(<1.88)
Sulfone	190	74	230	670	ND(<6.24)
Sulfoxide	ND(<175)	ND(<175)	180	300	ND(<175)
Phosphorous Compounds					
DIMP	ND(<1.34)	ND(<1.34)	ND(<1.34)	ND(<1.97)	ND(<1.97)
DMMP	1.68	1.72	2.73	2.26	ND(<1.34)
Metals, µg/g					
Arsenic	9.2	6.3	6.8	7.0	ND(<4.58)
Mercury	0.473	0.216	0.405	0.674	ND(<0.0245)
Copper	720	170	750	870	ND(<1.81)
Iron	15000	13000	20000	14000	ND(<14.8)
Magnesium	3000	2600	2300	2600	ND(<184)
Anions, µg/g					
Fluoride	34	23	77	94	26
Chloride	570	460	4300	4600	470
Sulfate	170	130	260	640	150
Cations, µg/g					
Calcium	4500	2200	7500	--	--
Potassium	3300	3600	4200	--	--
Sodium	4200	5400	6600	--	--
Total Cyanide, µg/g	ND(<5.00)	ND(<5.00)	ND(<5.00)	ND(<5.00)	ND(<5.00)
Percent Moisture	21.0	24.3	3.1	--	--

(a) Basin F material used during glassification testing at Battelle Northwest.
(b) Glass formers used during glassification testing at Battelle Northwest.

added to one or more of the drums sent to Battelle Northwest. The low moisture content of the Battelle Northwest sample resulted from the air drying process that was carried out to simplify solids handling operation.

Also included in Table 4.2 is the target chemicals analysis of the glass forming additives (sample RMA 1020) composed of soda ash and borax. The only detectable impurities of interest were fluoride, chloride, and sulfate.

4.3 APPARENT DESTRUCTION/IMMOBILIZATION EFFICIENCIES FOR TARGET CONTAMINANT SPECIES

The off-gas sampling system collected about 1 part in 50 of the total melter exhaust. Target species in the sampled melter exhaust were removed by the sample system and analyzed. Table 4.3 summarizes the sampling results and provides the calculated destruction/immobilization efficiency values realized during the test for each of the major species of concern.

Most of the organic species were below analytic detection limits in the sample system so a conservative estimate using the detection limit as the assumed concentration was made. The reported destruction of these species are therefore, worst case and presumed to be much lower than the actuals in some cases.

The negative immobilization values reported for chloride, sulfate, and mercury result from more material being detected in the exhaust than was actually fed to the melter. The only plausible explanations for this are: 1) analysis error in either the feed or exhaust samples; 2) a concentration of these species in the melter glass, plenum space or off-gas line from prior processing (e.g., shakedown testing) followed by release during the sample period because of changes in the operating conditions; or 3) errors in measuring the sampled and total exhaust flow of the system. Other explanations, for example, errors from nonisokinetic sampling, were not considered plausible in explaining the magnitude of these discrepancies.

Mercury is known to readily form volatile compounds at these melting temperatures and past work at Battelle Northwest with simulated radioactive wastes shows essentially all the mercury evolved during melting. Analysis of the

TABLE 4.3. Summary of Contaminant Destruction/Immobilization Efficiencies (Source: Arthur D. Little, Inc.)

Pipe Size = 3 in. schedule 40 (3.068 in. ID) 0.00477 m^2

Run Parameters	Modified Method 5	Method 101A
Total Run Time (min)	377	133
Total Waste Feed (kg)	13.6	3.68
Volumetric Exhaust Rate (m^3/min at 70°F and 29.92 in. Hg)	0.44634	0.37616
Total Sampled Volume (m^3 at 70°F and 29.92 in. Hg)	3.284	0.972

	Concentration In Feed, µg/g	Total Mass In Feed, µg	Amount Found In MM5 System, µg	Total Mass Exhausted During Test, µg	Destruction/ Immobilization Values, %	Corrected[(a)] Destruction/ Immobilization Values, %
Aldrin	3100	42,160,000	0.505	25.890	99.99994	99.99998
Isodrin	110	1,496,000	<0.063	<3.229	>99.9998	99.9993
Dieldrin	1300	17,680,000	<1.64	<84.077	>99.9995	99.9998
Endrin	180	2,448,000	<1.12	<57.418	>99.998	99.992
Dithiane	<1.88	<25,568	<2.00	<102.533	>99.6	99.87
Sulfoxide	300	4,080,000	<8.00	<410.132	>99.99	99.997
Sulfone	670	9,112,000	<8.00	<410.132	>99.995	99.998
DIMP	<1.97	<26,792	<1.23	<63.057	>99.8	99.92
DMMP	2.26	30,736	<1.26	<64.595	>99.8	99.93
Fluoride						
(In feed)	94	1,278,400	--	--	--	--
(In additive)	26	238,160	--	--	--	--
(Total)		1,516,560	16,750	859,000	43.4	81.1
Chloride						
(In feed)	4,600	62,560,000	--	--	--	--
(In additive)	470	4,305,200	--	--	--	--
(Total)		66,865,200	1,312,506	67,288,000	-0.6	66.3
Sulfate						
(In feed)	640	8,704,000	--	--	--	--
(In additive)	150	1,374,000	--	--	--	--
(Total)		10,078,000	586,902	30,088,000	-199	0.0
Mercury	0.674	2480.32	132	6800	-174.2	0.0
Arsenic	17	62,560	90	4643	92.6	97.3
Copper	870	3,201,600	5,508	283,601	91.1	96.8
Iron	14,000	51,520,000	91,999	4,736,960	90.8	96.6
Magnesium	2,600	9,568,000	15,176	781,410	91.8	97.0

(a) Corrected by setting mercury and sulfate values equal to 0%.

product glass from this test shows only a very small amount of mercury, in the parts per billion range, which represents over 90% volatilization from the melt. If we assume an error in measuring the total melter exhaust flow was made and that 100% of the mercury was actually lost, a new total exhaust volume can be derived and used to adjust the immobilization values reported for the other metals. Likewise sulfate, which has a relatively low solubility in the glass product and would be evolved as SO_2 gas under reducing conditions, could be assumed to have a 100% loss from the melter and back calculated to adjust the organic and halide species. The adjusted destruction/immobilization values based on these assumptions are also presented in Table 4.3. All values increase as a result of this adjustment.

4.4 NONCONDENSABLE GAS ANALYSIS

Noncondensable gases were continuously monitored with a series of on-line instruments. Since the samples were taken from a point downstream of the wet scrubber, any removal of the gas in the scrubber must also be included. Results are summarized in Table 4.4 for each of the gases monitored. The low CO/CO_2 ratio indicates adequate oxygen present for completing combustion in the melter plenum. Decomposition of the soda ash glass additive certainly contributed to the high carbon dioxide levels recorded. Nitrate and sulfate

TABLE 4.4. Summary of Continuous Gas Analyzer Data (Source: Battelle Northwest)

Gas	Scale	Typical Operating Range, % Scale	Maximum
CO	0% to 0.5% FS[a]	1 to 5	Occasional spikes to 10%
CO_2	0% to 5% FS	20 to 50	A few spikes between 60% and 100%
O_2	0% to 100% FS	20	Constant at 20%
NO_x	0% to 25 pm FS	40 to 60	Frequent spikes at ~100%
SO_2	0% to 2 ppm FS changed to 0 to 4 ppm FS at 8:36 on 4/2/87	90 to 100	Frequently >100%

(a) FS = full scale.

analysis of the scrub solution recycled through the ejector-venturi scrubber shows minimal removal of the NO_x but a substantial scrubbing of sulfate. If all of the sulfate in solution were assumed to have come from SO_2 gas scrubbing, then the melter exhaust concentration for SO_2 would increase from approximately 5 to 25 ppm.

4.5 PRODUCT LEACH TESTING

A sample of the glass product was subjected to toxicity tests outlined in the EPA Toxicity Characteristics Leaching Procedure (TCLP).[a] All hazardous organic species present in the feed were destroyed or volatilized in the melter and would not exist in the product glass. Consequently, only fluoride, sulfate, and certain metal species are of concern.

Leachate analysis revealed below detection limit levels of mercury, arsenic, fluoride, and sulfate. Copper, iron, and magnesium were present in detectable quantities as shown in Table 4.5. If the detection limits are assumed to be the actual concentration for mercury and arsenic, the leachate values are still roughly 2 orders of magnitude below the established limits. Since acceptance limits are not in place for copper, iron, and magnesium, 100 times the drinking water standard was used as a general guideline. Leachate concentrations of these metals are also well below the assumed limits.

4.6 ACID GAS EVALUATION

Analysis of the condensate and impinger solutions from the MM5 sampling system were used to determine the generation of acidic gaseous species. These species include halides, phosphorus compounds, and cyanide. Gaseous NO_x and SO_2, which react with water to form acid, were measured by the continuous gas monitors.

Semivolatile metallic halides generated during the glassification will exist as particulate in the melter exhaust and be captured on the sample system filter. Gaseous species that penetrate the filter are assumed to exist in the

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[a] "Toxicity Characteristics Leaching Procedure" and "Development of Leaching Procedure" 51 Federal Register 21653-21657 (June 13, 1986).

TABLE 4.5. Glass Product TCLP Results
(Source: Arthur D. Little, Inc.)

Element	Leachate Analysis µg/L	Leachate Analysis PPM(a)	Acceptance Limits PPM
Hg	ND(<0.828)	<0.001	0.2[(b)]
As	ND(<5.44)	<0.005	5[(b)]
Cu	34.3[(c)]	0.034[(c)]	100[(d)]
Fe	664.3[(c)]	0.664[(c)]	30[(d)]
Mg	332	0.332	14[(d)]
F^-	ND(<171)	0.171	NA
Cl^-	NA	NA	1[(d)]
$SO_4^=$	ND(<12900)	12.9	NA

(a) Assumes leachate spg = 1.
(b) Established EPA TCLP limits.
(c) Corrected for blank.
(d) 100 times the drinking water standard.
ND = Not Detectable.
NA = None Available.

form of hydrogen halides (acid gases) which will be collected with water in the condensate trap or with sodium bicarbonate in the impingers. Efforts to differentiate between other gaseous forms, for example F_2 or Cl_2 gas, were not considered necessary for the purpose of this study.

Results of the acid gas analysis is presented in Table 4.6. Phosphorus and cyanide were below detection limits in all samples and therefore, not included.

4.7 PARTICULATE LOADINGS

Particulate is generated from the glassification system by two main mechanisms: gross entrainment and volatilization/condensation. With the surface feeding and formation of a cold cap, gross entrainment is the predominant mode. The fine dust created by drying and blending Basin F material in a barrel

TABLE 4.6. Summary of Acid Gas Analysis
(Source: Arthur D. Little, Inc.)

Anion	F^-	Cl^-	$SO_4^=$
Total Wt. Collected	16.8 mg	1312.5 mg	586.9 mg
Total mg-Moles Collected	0.882	37.0	6.11

Acid	HF	HCl	H_2SO_4
Mass Collected	17.6 mg	1349 mg	599 mg
Concentration in Exhaust (g-moles/m^3)	$0.268(10)^{-3}$ (6 ppm)	$11.3(10)^{-3}$ (250 ppm)	$1.86(10)^{-3}$ (42 ppm)

tumbler with large rocks was intended for use with the submerged feeding technique. Its use with the surface feeding method resulted in excessive entrainment. As much as 10% of the waste feed material was carried with the exhaust gas stream as a result of this dusting. Particulate from gross entrainment tends to be relatively large material >10 μm and is easily removed from the gas stream for recycle by simple filtration systems. It is therefore, of no special concern.

Volatilization followed by condensation is the mechanism responsible for producing small, submicron-size particles from semivolatile species like sodium, boron, and mercury. Because of its size, this material is much more difficult to capture and behaves like a gas in many wet scrubbing and filtration systems.

5.0 DISCUSSION OF RESULTS

Examination of the organic destruction and removal efficiencies suggests the organic species of concern are readily decomposed in the glassification process. Even with the large feed entrainment we observed for the selected operating conditions and the surface feeding approach, temperatures and residence time in the melter plenum were apparently adequate for good destruction.

Aldrin, the only compound in sufficient concentration to measure actual organic contaminant destruction values, had a destruction efficiency in excess of 99.9999%. Use of the submerged feeding technique or an afterburner may not be required if this level of destruction can be achieved on a large scale for all the organic species. Further optimization of the operating conditions and furnace design may lead to even higher destruction efficiency values for added safety.

The data collected on arsenic suggest it behaves as a nonvolatile metal and is released from the melter by gross entrainment only. Provided good organic destruction can be maintained with wet or only partially dried feed material, it should be possible to reduce the entrainment by as much as a factor of 10 and achieve 99% or better retention of the arsenic and other nonvolatile metals without recycle.

Mercury is an exception. Its semivolatile nature makes it necessary to concentrate mercury as a secondary waste stream for separate disposal. Previous Battelle Northwest studies found that mercury readily combined with halides during the glassification process. Continued recycling of mercury to the furnace will lead to a buildup in the off gas with possible solids deposition in the piping, materials problems from the corrosive nature of mercuric halides, and safety concerns resulting from the very toxic nature of these materials. If it were necessary to separate mercury species from the rest of the entrained waste materials it may be feasible to use a dual-stage filtration system. The first stage roughing filter could remove only the relatively large dust particles that make up the bulk of the entrained particulate. A cyclone or baghouse is well suited for this application. Mercury, which should be largely present as <10 μm particles, could then be removed in the next stage high-efficiency particulate removal system such as an absolute HEPA filter or an electrostatic precipitator. The total amount of mercury in the Basin F appears to be quite small when compared with the quantity of glass frit that will be generated. Blending of any dust collected in the off-gas systems with the frit may prove to be acceptable.

Sulfur solubility in glass is generally in the range of about 0.1 to 0.2 wt% and decreases under reducing conditions. If levels in the feed exceed the solubility, a separate sulfate salt phase will form in the melter. The presence of organic materials however, serve as reductants in the melter feed that reduce sulfate to sulfur dioxide gas so it escapes with the exhaust gas. Off-gas monitoring showed measurable levels of SO_2 and a negative immobilization value for sulfate, which suggests that all the sulfur volatilized. No separate salt phases in the melter were observed after the test, so phase separation does not appear to be a concern.

To determine the maximum concentration of SO_2 in the untreated melter exhaust, the sulfate collected in the off-gas scrubber was assumed to have evolved from the melter as SO_2 (a worst-case estimate). For this case, the SO_2 concentration in the melter exhaust is increased from the measured value of 5 ppm downstream from the scrubber to roughly 25 ppm. Even so, this level is still within normally accepted concentrations for release to the atmosphere. Nitric oxide and nitrogen dioxide concentrations on the other hand are too high for direct release at ground level. When measured in the exhaust gas from the scrubber they approach the 25-ppm level. Very little of the NO_x was removed by the scrubber solution because neither the type of scrubber used nor the scrubbing solution (tap water) were designed for NO_x removal. Dispersion through a stack or treatment will be needed.

Wet scrubbing systems have been used for NO_x removal but tend to be very inefficient with these relatively low concentrations and generate secondary liquid waste to dispose of. A catalytic reduction system that operates dry and converts the NO_x to elemental nitrogen and oxygen is a more complex and costly alternative that could be considered. The concentrations of hydrogen halides also appear to be too high for untreated release at ground level. The release through a stack should be adequate. If necessary a properly designed wet scrubbing system could easily reduce the HF and HCl to acceptable levels. The wet scrubber system could be placed downstream of the filtration equipment so the scrub solution would be free of heavy metal contaminants that create additional disposal problems.

Leach testing of the product glass has confirmed that contaminants of concern are firmly fixed in the glass matrix. With the TCLP leachate concentration of regulated hazardous species well below the threshold values, it should be possible to dispose of the product as a nonhazardous waste. Directly pouring the molten glass into water will fracture it sufficiently for easy transportation to a landfill, if so desired, or it can be poured into disposal drums directly.

6.0 PROCESS FLOWSHEET AND MATERIAL BALANCE DEVELOPMENT

The bench-scale test program demonstrated feasibility and provided data for evaluating the effectiveness of glassification as a treatment technology for Basin F materials. Using data from this test in combination with engineering judgement and experience in developing waste treatment flowsheets for a variety of radioactive wastes, a material balance (Table 6.1) and process flowsheet (Figure 6.1) were prepared for the production-scale system. This flowsheet and material balance served as the basis for preparing a preliminary cost analysis of the technology which was assembled by Arthur D. Little, Inc. Development of the flowsheet also provided insight into the state of available technology and helped to define the system configuration and data needs for any subsequent development work.

For purposes of the flowsheet development, it was assumed that the estimated 400,000 yd^3 of soil, overburden and asphalt liner, and the 4 million gal of liquid in Basin F would be processed in 2.5 years. Using an annual on-stream factor of 75%, the design process rate of 950 tons of Basin F material per day was established.

A low melting temperature borosilicate glass formulation was used during the bench-scale test program. The boron in this glass improved the fluidity and ability of the melt to flux waste materials. Improved melting and fluidity could also have been achieved by increasing the furnace temperatures, but because of a temperature limitation associated with some components of the bench-scale furnace, this was not a feasible option. Any production-scale furnace would, however, be designed for higher temperature operation so a lower cost soda-lime silicate glass formulation could be used. The reference soda-lime glass composition which was selected contained 15 wt% alkali (sodium and potassium oxides), 15 wt% alkaline earth (calcium and magnesium oxides) and 70 wt% other elements (primarily silica, alumina, and iron oxide). The soil analysis previously presented in Table 3.1 established the chemical glass former addition requirements to meet this reference glass. Soda ash and lime are assumed to provide the required sodium and calcium. Table 6.2 presents the assumed glass product composition.

TABLE 6.1. Material Balance for Glassification, Basis: Tons Per Day (Source: Battelle Northwest)

	1 + 1A Basin F Material	2 Soda Ash	3 Lime	4 In-Leakage	5 Booster Gas	6 Combustion Air	7 Melter Exhaust	8 Glass Quench Water	9 Exhaust Cooling Water	10 Glass Frit	11 Dust	12 Fritter Vessel Vent	13 Process Water	14 Odor Destructor Exhaust[(a)]	15 Stack Gas
Waste Solids	827						1			1100	1				
Water	123						162.5	416	320	30		386	736		868.5
Lime			265												
Soda Ash		220													
Natural Gas					17.5										
Nitrogen				203.5		287	490.5					555			1045.5
Oxygen				62		87.5	79.5					168.5			248
Carbon Dioxide							259.5								259.5

(a) Off-normal operation only.

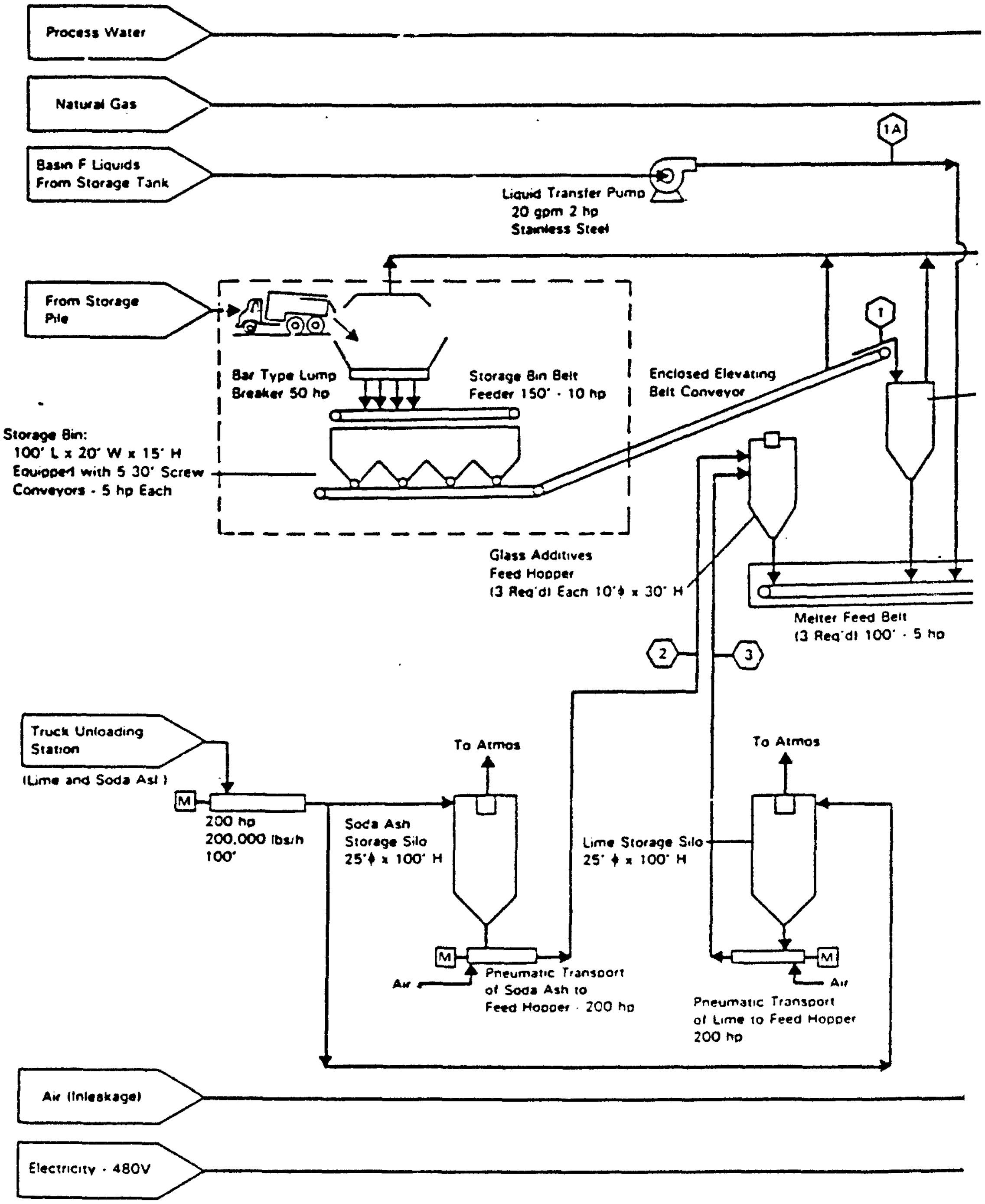


FIGURE 6.1. Preliminary Pro
(Source: Batte

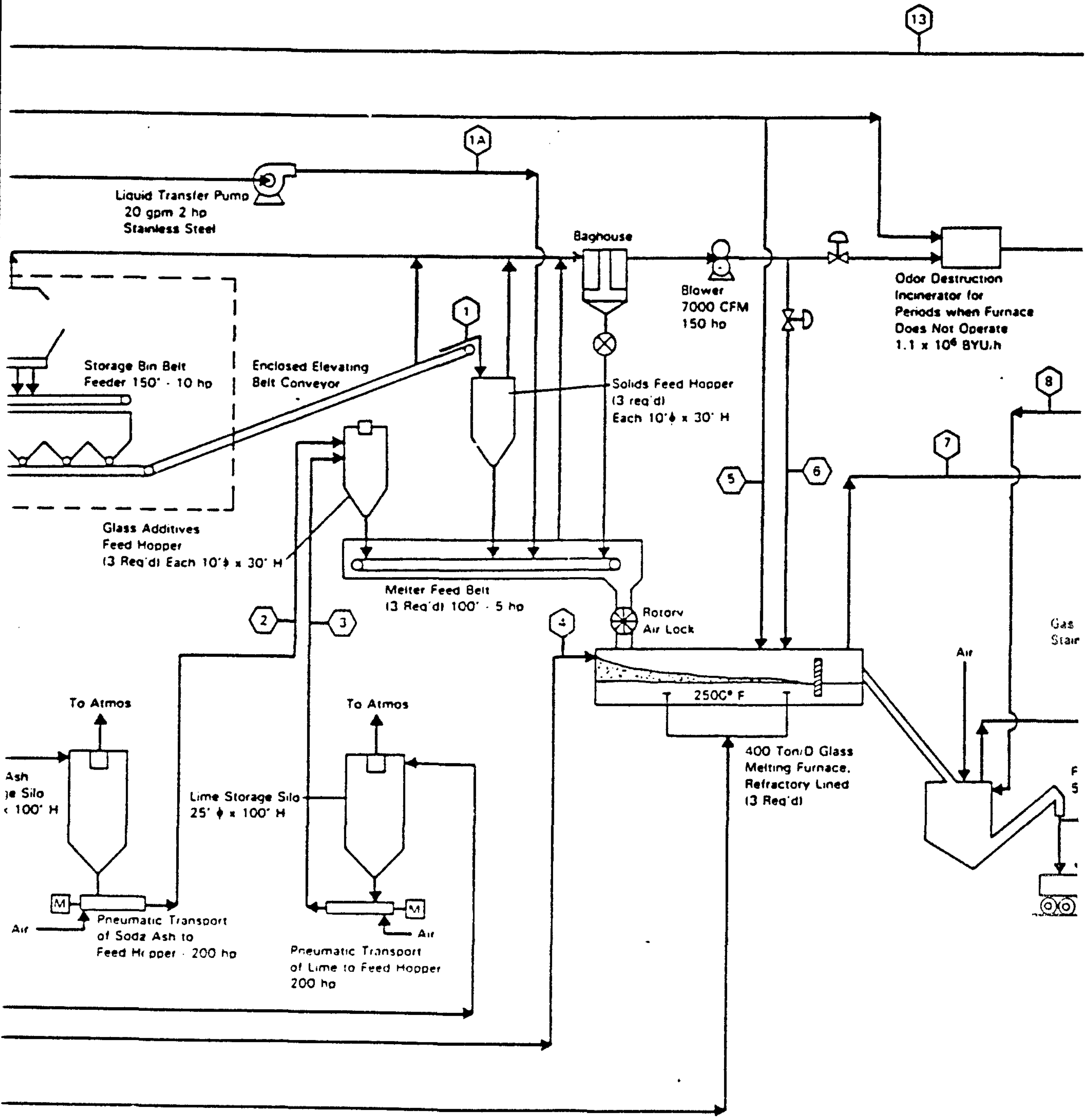
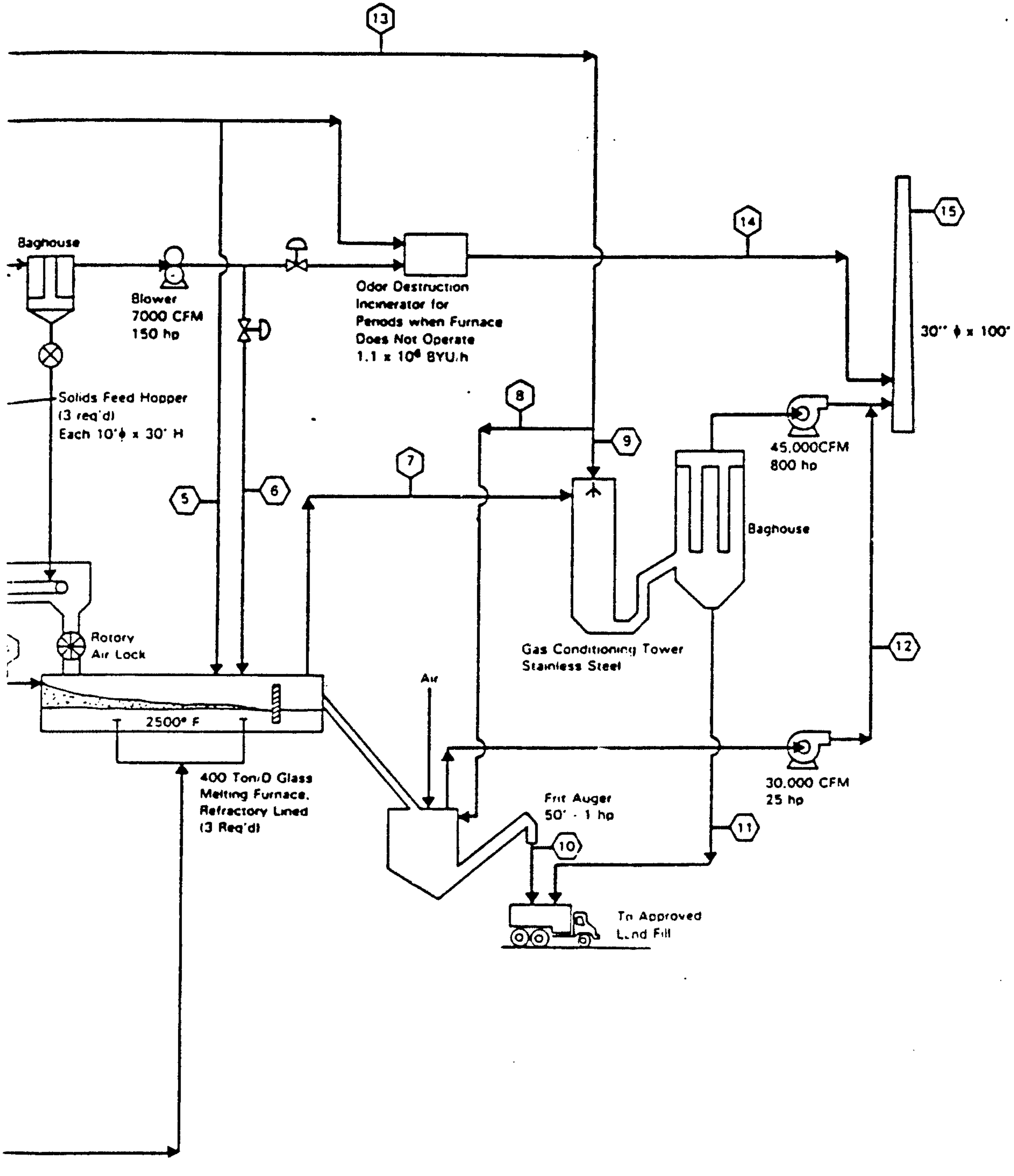


FIGURE 6.1. Preliminary Process Flowsheet for Glassification
(Source: Battelle Northwest and Arthur D. Little)



ss Flowsheet for Glassification
e Northwest and Arthur D. Little)

TABLE 6.2. Assumed Glass Formulation for Solidifying Basin F Materials (Source: Battelle Northwest)

Oxide Form	Wt% From Basin F	Wt% From Additions	Total
Al_2O_3	10.5	--	10.5
Fe_2O_3	3.3	--	3.3
Na_2O	1.5	11.5	13.0
SiO_2	55.1	--	55.1
K_2O	2.0	--	2.0
CaO	0.7	13.5	14.2
MgO	0.8	--	0.8
Others	1.1	--	1.1
Total	75	25	100

Basin F solids must initially pass through a rough size reduction step so they can be easily handled in conventional solids transfer systems. The proposed melting furnaces are quite large by commercial glass making standards and should easily accommodate reasonable variations in both particle size and composition. For this reason a simple approach to blending the additional chemical additives with the Basin F solids was selected.

The Basin F solids and glass additives will be metered from two separate feeders onto a common belt conveyor. It was assumed that the Basin F liquids could be sprayed onto the dry feed materials as they passed along the conveyor in an effort to reduce dusting problems in the feeding system and melter. Some blending will also occur when the feed is passed through a rotary air lock feeder on its way to the melting furnace. The rotary air lock helps reduce dusting associated with air inleakage to the furnace, and prevents flames from moving into the feed system.

The Basin F materials handling systems are enclosed and vented to control the release of toxins in the working place. The vent gas passes through a baghouse filter to collect any dust generated by the handling operations and is then sent to the melting furnace for destruction in the high-temperature plenum space. Alternatively the gas can be routed to a separate odor destruction incinerator for limiting the volatile organic vapors whenever the furnace is not operational. Dusts from the baghouse are periodically added back to the feed conveyor through a special air lock valve. A separate feed system is used for each of the three melting furnaces.

Glass-former additives are delivered in bulk by rail car or truck. At the unloading station these chemicals will be pneumatically transferred into two storage silos with the capacity for about a 1-week supply of glass additives. Simultaneous pneumatic transfer to the feed hoppers will be used to blend the chemicals. For the same reasons as previously discussed, adequate mixing is expected from this simple operation and so, an elaborate blending and metering system was not proposed.

Three melting furnaces that process 400 tons of glass per day are required. The furnace is an electric melter design using direct joule heating between metallic or graphite electrodes submerged in the molten glass pool. The all-electric melter was selected because of its characteristically low emission rates and high throughput per unit area. Alternating electric current is passed between pairs of electrodes with the glass acting as a resistance heater.

Glass must be molten to conduct electricity. The glass is heated to a molten state by providing an auxiliary heat source such as hot combustion gases in the vapor space above the melter. This glass heating or startup, as it is commonly called, usually occurs only a few times during the life of a furnace. Once the furnace is hot, power will be maintained between the electrodes during most nonoperational periods such as general equipment maintenance.

Electric furnaces typically process less than 100 tons of glass per day for commercial glass making. Therefore, a 400-ton per-day furnace is considered very large, but not unreasonable. Overhead-combustion-fired furnaces of this size using oil or natural gas are common. The lower energy costs associated with combustion firing is the primary reason why large glass furnaces are combustion-fired instead of electric-heated.

The Basin F feed and glass formers are introduced at one end of the furnace and the molten glass product and off gas is removed from the opposite.

With this arrangement, feed material such as decomposition gases and dust that are released at the batching end will remain in the furnace long enough for complete reaction. The vapor space near the off-gas end of the furnace serves as a secondary combustion chamber to complete destruction of any organic species present in the exhaust gas. Natural gas burners in this vapor space are provided in order to ensure sufficient heat for complete combustion. During furnace operation, ventilation air from soil handling and transfer systems will be used as a source of combustion air to the burners. This method of operation will eliminate the need to operate the organic odor incinerator continuously. These burners will operate to maintain the off gases exiting the furnace at about 1500°F, which should be more than adequate to oxidize the hazardous organic species present. The natural gas burners will also serve as the heat source for furnace startup.

From the glass melters the off gases are passed through a gas conditioning tower where a very fine water spray is introduced to achieve evaporative cooling. The temperature of the exhaust gas is lowered in this cooler to reduce flow rates through the remainder of the off-gas treatment system and to permit the use of fabric filters in the baghouse for removing entrained particulates.

The baghouse dust, a relatively small quantity in comparison to the amount of materials treated, will be sent for disposal with the glass product from the melting furnace. After filtration in the baghouse, the gases are then passed to the stack and released to the air. The required stack height is estimated to be between 50 and 100 ft in order to ensure that NO_x, SO_2, HF and HCl fumes generated by the destruction of certain Basin F materials will be dispersed to ambient air quality concentrations at ground level. Regulatory limits for air discharges from this glassification process are not established but data from the bench-scale tests suggest that the level of pollutants could be easily reduced below typical ambient air standards for breathing air quality without the need for a special wet scrubbing system.

The molten glass from the furnace will be discharged into a water bath to produce frit. The frit is a sand-like material that can then be easily transported to a land fill. Fracturing glass in this manner is a well-established, common procedure in the glass making industry. Steam generated by this process will also be sent to the stack and mixed with the process off gases.

7.0 RECOMMENDATIONS FOR FUTURE WORK

All unit operations proposed for the glassification process to treat Basin F materials are well-established industrial practices and should not require major long-term development programs for full-scale application of the technology. However, the flowsheet development work relied on engineering judgement and limited data, and some basic design issues remain as to whether the proposed materials handling and effluent treatment system are fully adequate. Larger-scale tests with prototypic equipment are needed to address these issues.

The recommended development program involves additional laboratory studies to refine the glass formulation, followed by extended duration (2 to 4 weeks) tests to examine organic destruction, study melting phenomena and establish operating specifications. These latter tests could initially be done in small engineering-scale equipment with a glass throughput capacity of a few tons per day. The data from the small-scale tests would then allow an effective pilot-scale field demonstration to be planned.

Additional laboratory studies could be done in ceramic crucibles using conventional high-temperature-radiant-heated furnaces. The principle focus of this effort would be to formulate the best mixture of glass forming additives and Basin F materials for rapid melting without producing secondary phases such as sulfate salts of excessive crystals in the melt. The resulting glass must have the proper viscosity and electrical conductivity to process through the furnace. Refractory corrosion rates are also generally examined during the glass formulation phase of the work. Techniques have been developed to study melting kinetics at this scale. An examination of melting rate as a function of feed particle size and degree of homogeneity is probably warranted for a preliminary assessment of whether further feed pretreatment should be pursued.

Testing at the engineering-scale level would follow to confirm proper feed formulation. In addition, these tests would identify any basic processing problems associated with either glass melting or equipment design before a pilot-scale field demonstration unit is developed. Furnace performance in terms of throughput and destruction efficiency of organics can be established in general for a wide range of variations in operating parameters. Process control parameters for safe and efficient operation could be outlined at this stage for confirmation during pilot-scale tests later on.

The pilot-scale field demonstrations are required to validate the process and equipment design basis, prepare detailed operational procedures, and conduct engineering optimization before the full-scale process is fixed. A one tenth-scale or larger field demonstration would typically be considered adequate for meaningful results when attempting to validate the scale-up correlations and relationships between equipment performance and process control parameters.

Effluent characteristics must be more fully defined to confirm that the proposed treatment system is appropriate for meeting regulatory release requirements. The engineering-scale tests can not completely address this issue because of significant differences in the furnace design and operation at this small scale. Pilot-scale tests would provide the required data to answer these questions as well as whether the natural gas burner is needed to complete the volatile organic destruction in the melter. The pilot-scale field demonstration is also required to examine the adequacy of the Basin F materials size reduction and transfer systems. Tests can be completed to help decide if any improvements in melting efficiency associated with better blending and size reduction are significant.

The fritted glass product will need to be studied for leaching behavior to determine if it can pass requirements for direct disposal in municipal land fills. The glass product from field demonstrations is preferred for use in these tests.

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