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MASTER

IN SITU VITRIFICATION LARGE-SCALE OPERATIONAL ACCEPTANCE TEST ANALYSIS

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SUMMARY

A thermal treatment process is currently under study to provide possible enhancement of in-place stabilization of transuranic and chemically contaminated soil sites. The process is known as in situ vitrification (ISV). In situ vitrification is a remedial action process that destroys solid and liquid organic contaminants and incorporates radionuclides into a glass-like material that renders contaminants substantially less mobile and less likely to impact the environment. A large-scale operational acceptance test (LSOAT) was recently completed in which more than 180 t of vitrified soil were produced in each of three adjacent settings.

The LSOAT demonstrated that the process conforms to the functional design criteria necessary for the large-scale radioactive test (LSRT) to be conducted following verification of the performance capabilities of the process. The energy requirements and vitrified block size, shape, and mass are sufficiently equivalent to those predicted by the ISV mathematical model to confirm its usefulness as a predictive tool. The LSOAT demonstrated an electrode replacement technique, which can be used if an electrode fails, and techniques have been identified to minimize air oxidation, thereby extending electrode life. A statistical analysis was employed during the LSOAT to identify graphite collars and an insulative surface as successful cold cap subsidence techniques. The LSOAT also showed that even under worst-case conditions, the off-gas system exceeds the flow requirements necessary to maintain a negative pressure on the hood covering the area being vitrified. The retention of simulated radionuclides and chemicals in the soil and off-gas system exceeds requirements so that projected emissions are one to two orders of magnitude below the maximum permissible concentrations of contaminants at the stack.

The technical data gathered from the LSOAT enhances the capability to quantitatively predict operational behavior under conditions not previously tested. Operational experience has also verified the assumptions and results of previous cost estimates. In summary, the LSOAT has provided the technical data, equipment refinements, and operational confidence to enable us to

conclude that the large-scale system conforms to its design criteria and will be ready for the LSRT following a successful verification test.

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INTRODUCTION

As disposal of radioactive and chemical waste materials gains increased attention, new and innovative technologies are being sought to achieve cost-effective protection of the public and environment from these wastes over very long periods of time. Included in this search are remedial action techniques to be applied, where warranted, to soils contaminated with transuranic and chemical elements that have already been disposed or are impractical or hazardous to retrieve for other disposal.

Pacific Northwest Laboratory (PNL)^(a) is developing a remedial action process for contaminated soils that could be potentially significant in enhancing in-place stabilization of these wastes. The process is in situ vitrification (ISV). It is being developed for the U.S. Department of Energy (DOE) with emphasis on transuranic-contaminated soils. It is expected that with some additional development the process could also be applied to a wide variety of chemical waste sites.

In situ vitrification is a thermal treatment process that converts contaminated soil into a chemically inert and stable glass and crystalline product. Figure 1 illustrates how the process operates. A square array of four molybdenum electrodes is inserted into the ground to the desired treatment depth. Because soil is not electrically conductive when the moisture has been driven off, a conductive mixture of flaked graphite and glass frit is placed among the electrodes to act as a starter path. An electrical potential is applied to the electrodes to establish an electrical current in the starter path. The resultant power heats the starter path and surrounding soil to 2000°C, well above the initial soil-melting temperatures of 1100°C to 1400°C. The graphite starter path is eventually consumed by oxidation, and the current is transferred to the molten soil, which is electrically conductive. As the molten or vitrified zone grows, it incorporates radionuclides and nonvolatile hazardous elements, such as heavy metals, and destroys organic components by pyrolysis. The pyrolyzed byproducts migrate to the surface of the vitrified

(a) PNL is operated for the DOE by Battelle Memorial Institute under Contract DE-AC06-76RLO 1830.

FIGURE 1. The Process of In Situ Vittrification

zone where they combust in the presence of oxygen. A stainless steel hood placed over the area being vitrified directs the gaseous effluents to an off-gas treatment system.

Pacific Northwest Laboratory began developing ISV technology in 1980 under contract to the DOE. Since that time, numerous experimental tests under a variety of conditions and waste types have been conducted (Oma et al. 1983; Timmerman and Oma 1984; and Timmerman et al. 1983). Table 1 describes the different scales of testing units that PNL used in developing ISV technology. The successful results of the 38 engineering- and pilot-scale tests have proven the feasibility of the process. Also, economic studies have indicated that tremendous economies of scale are attainable with the ISV process (Oma et al. 1983). Successful testing of the large-scale version of the ISV process will

TABLE 1. Testing Units for Developing ISV Technology

<u>Equipment Size</u>	<u>Electrode Separation</u>	<u>Block Size</u>	<u>Tests Completed</u>
Engineering Scale	0.23 to 0.36 m	0.05 to 1.0 t	24
Pilot Scale	1.2 m	10 to 50 t	14
Large Scale	3.5 to 5.5 m	400 to 800 t	3

provide the developmental information necessary to demonstrate process applicability to transuranic contaminated soils on a production scale.

The large-scale process equipment has been fabricated and installed, and the LSOAT has been completed. The objective of the LSOAT was to verify conformance of the process characteristics to the established functional design criteria relevant to the large-scale radioactive test (LSRT). Since the LSRT is to be performed employing a graduated risk philosophy, only relevant aspects, such as block geometry, radionuclide retention in the vitrified soil and off-gas system, and off-gas flow capabilities under nominal conditions, need to be considered. The LSOAT did not determine conformance to the functional design criteria as they relate to large inclusions of solid and liquid organics, void volumes, metals, and sealed containers, because the LSRT is not expected to contain these types of inclusions.

The LSOAT consisted of three adjacent settings of electrodes. Each setting vitrified more than 250 t of soil, fusing the blocks into one monolith approximately 7-m wide by 18-m long by 4-m deep. The vitrified area simulated an actual contaminated trench site at Hanford. Simulated radionuclide and chemical contaminants were introduced to the trench to gather retention data for these elements. Careful measurements and observations were made to determine the effectiveness of cold cap subsidence techniques, in keeping porous vitrified soil from migrating to the soil surface during processing.

CONCLUSIONS

The LSOAT produced much valuable technical data from which to conclude that the large-scale ISV unit will be ready for radioactive testing following a successful large-scale vitrification test (LSVT). The LSVT, which simulated the expected conditions of the LSRT, was needed to verify the performance of some important equipment refinements identified during the LSOAT and to verify the predictive capabilities developed from the LSOAT. The LSOAT developed some important conclusions about the behavior of the process.

The mathematical model developed from pilot-scale empirical data (Oma et al. 1983) proved to be an extremely useful tool in predicting the melt characteristics for the large-scale unit. All three settings of the LSOAT adequately represented the characteristics predicted by the model. Therefore, it is prudent to assume that the model's prediction of achieving melt depths between 13 m and 25 m (Buelte and Carter 1986) is accurate. The model can also be used with confidence to predict run time and energy requirements and select the appropriate and most cost-effective electrode separation for a particular site geometry.

Although three electrode failures occurred during the LSOAT because of air oxidation of Mo, the test provided sufficient empirical data to identify coating techniques and materials designed to extend electrode life. In addition, a cost-effective electrode recovery technique that can easily be applied during radioactive applications was demonstrated. Therefore, even though electrode life beyond 72 h has not been demonstrated on the large scale, the electrode coating and recovery techniques will ensure continuous operation for radioactive testing and potential field operation.

The LSOAT successfully demonstrated off-gas flow capabilities in excess of those required by the functional design criteria to maintain containment of off-gases within the hood. Even under worst-case effluent conditions, such as vitrifying through a large void, combustible volume, or sealed container, the off-gas system's capacity exceeds the established requirement.

The retention of radionuclides and chemical contaminants in the vitrified soil and off-gas treatment system also exceeded criteria. Since it has been shown that transuranic behavior is similar to that of Sr, the overall decontamination factor (DF), as defined by the ratio of transuranics in the soil to those at the release point, under maximum expected concentrations is 1.3×10^{10} . As a result, the transuranics of greatest concentration exited the stack a factor of 50 under the maximum permissible concentration. Fluoride retention in the soil also exceeded expectations, thus minimizing corrosion concerns in the off-gas system. Although sulfate retention in the soil was negligible, the decontamination by the off-gas system reduced SO₂ stack concentrations well below threshold limit values of 5 ppm for 8-h direct exposure to workers (American Conference of Governmental Industrial Hygienists 1984). All of these factors demonstrate the readiness of the large-scale ISV process for radioactive testing.

One of the more interesting discoveries during the LSOAT was the ability to suppress the formation of a rising cold cap above the soil surface. A statistical analysis was employed to identify and predict the effectiveness of cold cap subsidence techniques. Use of graphite collars and surface insulation verified predictions and successfully subsided the cold cap during processing. These techniques, which are now regularly employed for all ISV applications, will eliminate concerns regarding future subsidence and disposal of contaminated porous vitrified soil following radioactive testing.

The LSOAT supports the assumptions and results of cost estimates for ISV. The test verified that only two qualified operators are necessary for transuranic contaminated soil site applications, as assumed by the cost estimate. The ability to move the hood and trailer between settings in two shifts was also verified. The resultant costs from operations, which included labor, site preparation, consumables, and capital recovery, were within the reported cost range of \$160/m³ to \$330/m³ for transuranic contaminated soils, which is governed primarily by electrical power cost and soil moisture content (Buel et al. 1985).

PROCESS DESCRIPTION AND REFINEMENTS

The large-scale process equipment for ISV is depicted in Figure 2. Controlled electrical power is distributed to the electrodes and special equipment contains and treats the gaseous effluents. The process equipment required to perform these functions can be described most easily by dividing it into six major components:

- electrical power supply
- off-gas hood
- off-gas treatment system
- glycol cooling system
- process control station
- off-gas support equipment.

Except for the off-gas hood, all the components are contained in three transportable trailers (see Figure 3). They consist of an off-gas trailer, a process control trailer, and a support trailer. All three trailers are transportable to accommodate a move to any site over a compacted ground surface. The off-gas hood and off-gas line, which are installed on the site for collection of gaseous effluents, are partially dismantled and placed on a flatbed trailer for transport. The large-scale process equipment and its process capability requirements are described in detail by Buelt and Carter (1986).

The LSOAT effected some important refinements to the process equipment. The reliance of the off-gas system on pneumatic air pressure to operate the off-gas control valves became evident. Therefore, the original air compressor was replaced with a redundant dual-head compressor. If a malfunction occurs, such as a leak in the air system or failure of the main compressor head, the redundant compressor head will supplement the system capacity. The system has been satisfactorily tested under these conditions.

Volatilization of sulfates from the molten soil creates creates acidic conditions in the scrub solution. Even slight concentrations of chlorides or fluorides at low pH can create unacceptable corrosion environments for the 300-series stainless steel off-gas system. Therefore, a caustic addition

FIGURE 2. Setup for In Situ Vitriification

FIGURE 3. Process Trailers for the Large-Scale ISV Unit

system was added to the scrubber system to keep $\text{pH} > 7$, thus attaining an expected 10-yr lifetime. The caustic addition system has been successfully tested in the latter settings of the LSOAT.

Many other equipment improvements incorporated as a result of the LSOAT need to be demonstrated during the LSVT prior to radioactive testing. They include the following:

- upgrade of the electrode power supply high current wiring and cooling system
- upgrade of the off-gas trailer's heating and ventilating system
- replacement of carbon steel bearings in the pneumatic valves with malcomized 300-series stainless steel bearings
- installation of a radiation sampling system around the hood to ensure containment and treatment of volatilized radionuclides
- installation of redundant level-detection instruments for the scrub solution tanks to prevent scrub pump cavitation
- installation of an independent hood blower in the event of complete failure of the off-gas system.

CONFORMANCE TO FUNCTIONAL DESIGN CRITERIA

The objective of the LSOAT was to verify conformance to the elements of the Process Capability Requirements relevant to the first radioactive demonstration. The requirements, summarized by Buelt and Carter (1986), identify the functional design criteria that the process must maintain to be suitable for use at a transuranic contaminated soil site. The relevant elements include geometric depth and width requirements, electrode performance, containment of gaseous effluents, and retention of radionuclides and chemicals in the vitrified soil and off-gas system.

CONFORMANCE OF GEOMETRIC SHAPE TO MATHEMATICAL PREDICTIONS

A mathematical model, developed and confirmed through engineering- and pilot-scale tests specifically for ISV, has been used to predict operating parameters of the large-scale process (Oma et al. 1983). One of the objectives of the LSOAT was to determine the ability of the model to predict run time, depth, width, and shape of the melt, energy requirements to vitrify a given volume of soil, and voltage and current relationships during processing. The accuracy of these predictions provides confidence in our ability to predict geometric limitations of the process.

Table 2 is a comparison of predicted vs measured process parameters for the three LSOAT settings. The range of predicted values is bounded by different assumptions related to the heat losses emanating from the surface of the vitrified zone. The low heat loss assumption accounts for the presence of a cold cap over the molten soil, while the high heat loss model assumes the elimination of the cold cap and its associated insulation value. The different modeling assumptions only affect process efficiency, as demonstrated by run time and energy comparisons. LSOAT-1 and -2 operated with the existence of a cold cap while LSOAT-3 did not. In LSOAT-3, the elimination of the cold cap was assisted by 8 cm of fiberglass insulation over the molten surface, with an insulation value equivalent to that of a cold cap. Consequently the actual process parameters more closely match the low heat loss model. The measured geometric shape, however, differed slightly from the predicted results.

TABLE 2. A Comparison of Predicted and Measured Parameters During the First Large-Scale Setting (LSOAT-1)

	Predicted Results		Actual Results		
	Low Heat Loss	High Heat Loss	LSOAT-1	LSOAT-2	LSOAT-3
Run Time, h - excluding downtime	62.4	76.1	62	71.8	71.2
Melt Depth, m	4.00	4.00	4.0	3.0	3.0
Melt Width, m	7.2	7.2	7.0	7.1-8.2	---
Volume, m ³	165	165	---	---	---
Mass, t	265	265	265	187	265
Final Voltage/ Current, V/A	735/2450	735/2450	600/2800	620/2400	620/2170
Average Power, kW - excluding downtime	3370	3370	3400	2160	2900
Energy Dissipated, MWh	210	256	210	148	210
Energy/Mass, kWh/kg	0.79	0.97	(0.79)	(0.79)	(0.79)

Figure 4 depicts a comparison of predicted vs achieved melt shape. The LSOAT produced vitrified blocks slightly wider and shallower than predicted, although the total vitrified volume and dissipated energy matched exactly. The variance may have been due to the fact that the LSOAT was conducted in partially disturbed soil that may encourage outward growth. This variance would not affect the ability of the process to meet the attainable depth of 13 m reported by Buelt and Carter (1986). In fact, since the results more closely match the low heat loss model, the practical depth limit of 25 m is more likely to be obtained.

The geometric data on the vitrified blocks were obtained from underground mapping techniques using ground-penetrating radar (GPR) and physical measurements taken after partial excavation. Figure 5 is a map of the test site showing the areas covered by radar surveys. The locations of the electrodes for each block are shown as heavy black dots. These measurements used a Geophysical Survey Systems, Inc., GPR unit with a 500-MHz (nominal)

FIGURE 4. Predicted vs Achieved LSOAT Melt Shape

antenna. The antenna, or transceiver unit, transmits and receives radar signals in a frequency band of approximately 250 to 750 MHz.

Bright reflection patterns evident in the radar profiles generated by the survey indicate that the vitrified blocks contain interfaces (compositional or textural) that reflect the radar signals. The strongest reflections occurred

FIGURE 5. Interpreted and Measured Boundaries of the Vitrified Blocks (coordinates are in feet)

at depths greater than 1.8 m. This is probably the floating rock layer observed in pilot-scale runs. Along most of the profiles, the edges of the blocks seem to be well defined by abrupt transitions from zones of low reflectivity to zones of high reflectivity.

Excavation of the north end of the LSOAT-2 and the south end of LSOAT-1 revealed the true profiles of the blocks. Figure 5 shows the maximum width of the blocks, which occurs at the 1.8-m depth, superimposed over the shape determined by the GPR unit. The block is assumed to be symmetrical to obtain the shapes of the east, west, and south sides. The GPR technique is fairly accurate within a ± 0.6 -m deviation, which is adequate for verification of the vitrified block shape. However, the technique did not detect the presence of a rectangular basalt block protruding from the north end. Alternative GPR test conditions and alternative underground survey techniques, such as geophysical diffraction tomography (Witten and Stevens 1985), are worthy of further consideration to improve the sensitivity of underground mapping techniques for determining block shape and identifying underground inclusions.

ELECTRODE PERFORMANCE

Present electrode design calls for a 5.1-cm-dia Mo rod inserted into the entire depth of the contaminated soil to be vitrified. The upper regions of the electrodes, which are exposed to air when the molten surface subsides during processing, are encased in a 6.4-cm-ID by 15-cm-OD graphite collar. As described later in this report, the graphite collar helps prevent the formation of a rising cold cap above the soil surface.

Subsidence of the cold cap exposes the graphite collar to air at high temperatures (i.e., $>600^{\circ}\text{C}$). The life of the graphite under these conditions has not been demonstrated beyond 40 h. The graphite collar is eventually consumed by oxidation, exposing the Mo electrode to the same oxidizing conditions at these temperatures. As Stanek (1977) reports, Mo exposed to temperatures greater than 600°C in air oxidizes rapidly. Successful elimination of the rising cold cap in the third setting of the LSOAT created these conditions, causing three electrodes to fail, beginning at 53 h into the run.

The failures were caused by oxidation, not breakage, as evidenced by visual observations. Figure 6 depicts the condition of the electrodes before and after the third setting of the LSOAT. It verifies that the failure point was above the molten soil surface, which varied from 76 cm below the electrode buss at the beginning of the run to between 122 cm and 152 cm after the completion of the setting. The failure points shown in the figure varied from 75 cm to 145 cm below the buss. If the failure mechanism had been breakage, rather than oxidation, followed by arcing well above the glass surface, significant concentrations of Mo would have been collected by the off-gas system. Figure 7 shows that the amount of Mo accumulated in the off-gas system, including plateout in the hood and off-gas lines, is 190 g. This would account for less than 1 cm of electrode length. Although the sudden rises in Mo accumulation are due to arcing to the molten surface after failure, the failure mechanism was undoubtedly oxidation at the point above the molten surface indicated in Figure 6.

The northwest (NW) electrode that remained intact throughout the run showed diameter loss at the failure point of the graphite collar. In previous experiments, before employing the graphite collar technique for enhancing cold cap subsidence, a protective layer of molten soil adhered to the Mo electrodes, protecting them from oxidation. The area of oxidation on the NW electrode is within the original location of the graphite collar, which prevented molten soil from forming a protective coating around the Mo electrode. Since the graphite collar prevents the natural protective glass coating from forming, a substitute coating material is needed over the Mo electrodes when graphite collars are employed.

An electrode analysis team was formed to identify an alternative electrode design to protect it from oxidation; the most prospective alternative is a conductive ceramic coating (88% zirconium diboride and 12% molybdenum disilicide) over the portions of Mo electrodes exposed to air at high temperatures. In addition, the conductive graphite flakes in the annulus between the Mo and graphite collar are replaced with the powdered $\text{ZrB}_2/\text{MoSi}_2$ mixture to augment the effectiveness of the coating.

FIGURE 6. Condition of the Electrodes Before and After LS0AT-3

FIGURE 7. Molybdenum Accumulated in the Off-Gas System
(Plateout in Hood and Off-Gas Lines)

A literature study showed that 88%-ZrB₂/12%-MoSi₂ coatings greatly reduce the oxidation rates of Mo in air at 195°C. Vendors indicate that it can be applied over 1.8-m lengths of Mo electrodes. The joints would be coated with ZrB₂ paint during assembly. Laboratory tests were recommended with Mo electrode samples coated with 88%-ZrB₂/12%-MoSi₂.

An electrode recovery technique was employed in the LSOAT to demonstrate recovery from a failed electrode. The technique developed from the demonstration lowers the failed electrode into the molten soil by connecting additional 1.8-m lengths of 5-cm-dia Mo rod to the Mo buss at the top of the off-gas hood. This technique eliminates withdrawal of potentially contaminated electrode materials from the hood, thereby keeping occupational exposure to a minimum.

Oxidation/corrosion below the glass melt was negligible during all three tests, indicating expected long life under soil melting conditions.

OFF-GAS FLOW CAPABILITIES

The functional design criteria reported by Buelte and Carter (1986) identify the requirements for the off-gas system. It must contain gaseous releases resulting from vitrifying gas-generating soil inclusions, such as void volumes, combustible packages, liquid organics, sealed containers, and combustibles

mixed with soil. The off-gas system was conservatively designed to contain and treat the maximum credible release from vitrifying an inclusion with a design factor of two. The maximum credible releases are based on measurements obtained from engineering- and pilot-scale tests.

In order to maintain a negative pressure of 0.25 cm of water on the hood under nominal conditions, a flow rate of 50 standard (std) m³/min is required. Only minute quantities of process gases are being generated, so the off-gas system flow rate is balanced by air inleakage through hood panel joints and around the hood's perimeter. Operation of just one scrubber system achieves an off-gas flow rate of 58 std m³/min and a negative pressure of 0.75 cm of water in the hood, thus exceeding this requirements. A backup scrubber system is always in reserve, ready for operation.

Operation of both scrubber systems is necessary however to meet the design requirement of 104 std m³/min. This flow is required to contain the maximum credible gaseous release from vitrifying soil inclusions, while providing adequate combustion air, when necessary, at a design factor of two. Operation of both scrubber systems achieves an off-gas flow of 116 std m³/min, within 80% of the maximum power rating of the blower. The off-gas system's flow capacity ensures that even the maximum credible release will be contained for treatment with no gaseous releases to the atmosphere.

RETENTION OF SIMULATED RADIONUCLIDES AND CHEMICALS

The efficiency of retaining or destroying hazardous chemicals, transuranics and associated fission products in the molten soil can be expressed as the DF. It is defined as follows:

$$DF = \frac{\dot{m}_i}{\dot{m}_e}$$

where $\dot{m}_i$ = initial or input mass of contaminant in the control volume per unit time and

$\dot{m}_e$ = exit mass of contaminant from the control volume per unit time.

The control volume may be defined as the molten soil, off-gas system, or a combination of both. Decontamination factors for the molten soil have been measured for Sr, nitrates, fluorides, and sulfates.

Results from the radioactive pilot-scale test show that the retention of Sr in the soil during processing matches that of transuranics (Timmerman and Oma 1984). This data is confirmed by nonradioactive pilot-scale tests with transuranic analogs (i.e., rare earth elements). Since these analogs are too costly to include in large-scale nonradioactive testing, the retention data for Sr signify the anticipated characteristics for the transuranics in the large-scale process. Core samples from virgin soil and the vitrified soil produced in LSOAT-2 showed Sr concentrations of 0.034% and 0.042%, respectively. The virgin soil concentration is due to natural Sr concentrations, while the higher concentration in the vitrified soil is due to the natural concentration plus simulants added to the test site prior to vitrification. These concentrations provide an adequate Sr source to determine the soil-to-off-gas DFs. From the power curve for LSOAT-3 in Figure 8, the LSOAT-3 block weight can be estimated to be 265 t assuming an energy-to-mass ratio of 0.79 kWh/kg. Therefore, assuming that the Sr concentration is similar to that analyzed for LSOAT-2, the 265-t LSOAT-3 block contains 110 kg of Sr. Since only 3.22 g of Sr were exhausted to the off-gas system, as determined by analyses of scrub solutions, high-efficiency particulate air (HEPA) filters, and internal piping smears, the soil-to-off-gas DF is 3×10^4 . This value exceeds previously reported values

FIGURE 8. LSOAT-3 Power Curve

from pilot-scale tests, and the retention of transuranics in the large-scale blocks can be expected to exceed that reported for the pilot-scale test.

The LSOAT also produced valuable data on the behavior of nitrates mixed with the soil. Nitrates have been added to many of the contaminated soil sites during their active lives. Thermal treatment processes generally decompose the nitrates to nitrous oxide gases. However, LSOAT data show that the large-scale process is an efficient NO_x destructor. Prior to vitrification, nitrate salts were added to each of the three electrode settings to represent conditions at an actual transuranic contaminated soil site at Hanford. Prior to the third setting, soil analyses revealed that a minimum of 5.9 kg of nitrates were in the soil to be vitrified. During vitrification, these nitrates were effectively reduced to diatomic nitrogen and oxygen, based on nondetectable quantities of nitrates collected by the scrub system and exhausted by the stack. Normally, NO_x would be expected to be collected by the scrubber system, or an average concentration of 12 ppm NO_x would be exhausted out the stack. As it was, less than 0.07 kg of nitrates were collected by the scrubber system, and less than 0.05 ppm NO_x were exhausted out the stack. This results in a minimum NO_x destruction efficiency of 99.6%. This is an important characteristic because many contaminated soil sites may be rich in nitrates.

Numerous technical studies have been performed on the behavior of nitrates in thermal treatment processes to explain the NO_x destruction characteristic of ISV. There have been a considerable number of basic kinetics studies of NO decomposition over temperatures from 25°C to 5000°C. Above 1300°C the thermal decomposition of NO is homogeneous and occurs by a mechanism involving O_2 atoms. Atomic O_2 is believed to be in thermal equilibrium with molecular O_2 at these high temperatures and is assumed to react with NO by:

which is followed by the much faster reaction:

These two reactions, together with the O_2-O equilibrium reaction:

where M is a third body, are referred to as the Zeldovich mechanism (Zeldovich 1946). In situ vitrification molten soil temperatures have been measured between 1400°C and 2000°C, so this is a likely mechanism for destruction of NO_x in the ISV process.

The presence of pyrolysis and combustion products from vitrifying organics in the soil will aid NO_x destruction at lower temperatures. In a study by Mori and Ohtake the focus of NO decomposition was in the temperature region of 650 to 950°C. Measurements were made to determine the effects of the reducing species, carbon monoxide and hydrogen, in combustion gas.

Mori and Ohtake concluded that NO decomposition is obtained by the decomposition reactions of the reducing species. The effects of the reducing components CO and H_2 , in the combustion gas, on the NO decomposition rate were investigated using three different mixtures of nitrous oxide and argon, one pure, one with CO, and one with H_2 . The decomposed products of NO are N_2 and O_2 , and the overall decomposition reaction of NO is expressed by the stoichiometric ratio as follows:

In the NO-CO-Ar mixture the NO decomposition rate is in linear proportion to the CO concentration. The overall reaction is expressed by the stoichiometric ratio as follows:

In the NO- H_2 -Ar mixture the NO decomposition rate increased almost in proportion to the H_2 concentration. The overall reaction is given as:

The decomposition rates of NO-NO, NO-CO, and NO-H₂ reactions depend on the CO-H₂ concentration when the NO concentration is higher than about 400 ppm but do not depend on NO concentration itself. However, for NO concentrations lower than 250 ppm the decomposition rate decreases with NO concentration. Therefore, this mechanism of NO decomposition may play an important role in ISV above the molten soil where temperatures are lower and reducing components exist. This mechanism for NO_x destruction is supported by analyses of gas bubbles taken in the cold cap region of LSOAT-1. Table 3 gives gas bubble compositions at three depths within the cold cap. The main constituents, N₂ and CO₂, support the theory that NO_x destruction is accomplished with the aid of reducing agents, as Mori and Ohtake postulated.

The retention of fluorides in the vitrified soil is an important consideration when applying ISV to contaminated soil sites. For example, the 216-Z-12 Crib at Hanford received more than 2.8×10^8 L of 0.05 M fluoride waste during its 25-yr lifetime (Kasper 1982). Much of this sorbed to the underlying soil. High retention of the fluorides in the molten soil will minimize fluoride corrosion of the off-gas system. The fluoride analytical results from the virgin and vitrified soil samples, scrub solution, and HEPA filters provide conclusive data that retention of fluoride is 98.7%. In the case of 216-Z-12 Crib, high retention of fluorides keeps the expected accumulation in the off-gas system scrub solution to 0.18 wt%, less than half the functional design criteria limit. This eliminates the need to change out scrub solutions frequently.

TABLE 3. Gas Bubble Composition in Above Molten Surface of LSOAT-1

Gas	Composition, %		
	Top	Middle	Bottom
H ₂ O	0.7	0	0
N ₂	55	52 to 88	6
O ₂	6	3 to 6	0
CO ₂	39	45 to 6	94
SO ₂	trace	<0.01	<0.01

Sulfates on the other hand demonstrate no propensity to be retained by the molten soil. Natural concentrations of sulfates in the soil and those added as waste simulants completely volatilize and are collected by the scrub solution. Normally, vitrification processes retain up to 1 wt% sulfur trioxide in glass. However, the reducing conditions and high temperature that are prominent in ISV and responsible for NO_x destruction reduce solubility of SO_3 in the molten soil. The negligible retention of SO_3 eliminates the concern for creating leachable phases of sodium sulfate in the waste form.

ELEMENT RETENTION BY THE SCRUB SYSTEM

Even though the retention of transuranics, fission products, and most chemical contaminants in the vitrified soil is high, the gaseous effluents must be decontaminated before they are exhausted to the atmosphere. Analyses of the scrub solution and HEPA filters provide conclusive data on the performance of the off-gas system during processing. Because of the potential for water carryover from the wet scrubbers to the HEPA filters, the first two settings (LSOAT-1 and -2) were conducted with a wet scrubber differential pressure of 46 cm of water. Since this pressure is below that recommended by the manufacturer, the efficiencies of the mist eliminators and assumably the tandem nozzle scrubbers were sacrificed. Figures 9 and 10 show the accumulation of Sr

FIGURE 9. Accumulation of Sr in Scrub Solution Tanks for LSOAT-1

FIGURE 10. Accumulation of Cs in Scrub Solution Tanks for LSOAT-1

and Cs in the scrub solution tanks during vitrification. As evidenced by the decaying mass of nuclides in the tanks, the Sr and Cs collected by the scrubbers are rapidly re-entrained through the mist eliminators and collected on the HEPA filters. The initial quantities of Cs and Sr in LSOAT-1 were a result of transfers from a supply tank that was slightly contaminated with nonradioactive Sr and Cs. The supply tank was cleaned for later settings, consequently initial quantities of Cs and Sr for LSOAT-2 and -3 were much lower. No detectable concentrations of Cs were measured in the scrub solution for these two settings, so Cs data is not available. Figure 11 shows the accumulation of Sr in

FIGURE 11. Accumulation of Sr in Scrub Solution Tanks for LSOAT-2

the scrub solution tanks during second setting (LSOAT-2). The amount of Sr collected on the filters during LSOAT-2 totaled 19 g. Because only 1.2 g remained in the scrub solution tanks after this setting, the majority of the contamination from the process was collected on the filters. Although the overall required DF was met by the two-stage filters, this situation is unacceptable from an operational perspective because of high expected doses and particulate loadings on the filters.

Jumper modifications before the third setting greatly reduced the water carryover potential. Consequently, the scrubbers were operated between 102 cm and 152 cm of water. Analytical results of Sr in the scrub solution from LSOAT-3, shown in Figure 12, demonstrated that Sr accumulated steadily with no propensity to be re-entrained. The fact that the higher differential pressure improved the efficiency of the mist eliminators is supported by HEPA filter analyses. Multiple samples from both stages of filters showed that the maximum amount of Sr entrained by the wet scrubber system is 7.4 mg, a reduction from the previous run by a factor of 2500. The overall DF of the wet scrubber system for Sr is 440. Taking credit for the efficiency of one stage of HEPA filters ($DF = 10^3$) (Flanders Filters, Inc. 1984), the off-gas system's cleanup capability ($DF = 4.4 \times 10^5$) exceeds the requirement of 10^5 as specified in the functional design criteria (Buelte and Carter 1986). This does not account for the expected additional DF of 10^3 for the second-stage filter.

FIGURE 12. Accumulation of Sr in Scrub Solution Tanks for LSOAT-3

Note the relative slopes of the accumulation curve at different times of the run. The rate of accumulation is high during the first 25 h of the test, but falls off rapidly during the later stages. Examination of the potential causes indicates the obvious reason for this behavior. Oma et al. (1983) have correlated a strong dependency of burial depth with retention in the soil. Since the maximum accumulation rate is achieved during the first 25 h when the melt depth is less than 1.5 m, this explanation is quite plausible. Examination of the power curve in Figure 8 shows that most of the energy dissipation, and consequently the maximum melting rate, was achieved between 15 and 55 h. This time period does not coincide with the rate of accumulation of Sr and thus is eliminated as a significant cause. Analysis of the soil prior to vitrification of the power curve in Figure 8 shows that most of the energy dissipation, and consequently the maximum melting rate, was achieved between at and 55 h. This time period doe not coincide with the rate of accumulation of Sr and thus is eliminated as a significant cause. Analysis of the soil prior to vitrification revealed the highest concentration of Sr in the soil at the 2.7-m depth, with only natural soil concentrations of Sr at shallower depths. This depth was not achieved until after 50 h into the test, and therefore could not account for the early accumulation rate. The data strongly indicate that burial depth is the controlling factor for soil retention. At depths greater than 1.5 m, the process can be expected to achieve extremely high retention values for transuranics and fission products in the vitrified soil.

The analytical results from the scrub solution and HEPA filter samples also provide off-gas system DFs for fluorides and sulfates. Figures 13 through 18 plot the accumulation of sulfates and fluorides in the scrub solution tanks for all three settings (LSOAT-1, -2, and -3). Unlike Cs and Sr, the accumulation plots for sulfates and fluorides do not show a strong tendency to re-entrain these compounds at low scrubber differential pressures. However, the HEPA filters for LSOAT-1 and -2 were not analyzed for these compounds to verify this observation. Analyses of the HEPA filter and scrub solution from LSOAT-3 showed wet scrubber DFs of 140 and 70 for fluorides and sulfates, respectively. These values are consistent with those of Sr reported for LSOAT-3. However, the overall DF of SO_x gas, as measured by stack samples, ranges from 1000 to 7000 for all three settings. The additional DF was

FIGURE 13. Accumulation of Sulfate in Scrub Solution Tanks for LSOAT-1

FIGURE 14. Accumulation of Sulfate in Scrub Solution Tanks for LSOAT-2

FIGURE 15. Accumulation of Sulfate in Scrub Solution Tanks for LSOAT-3

FIGURE 16. Accumulation of Fluoride in Scrub Solution Tanks for LS0AT-1

FIGURE 17. Accumulation of Fluoride in Scrub Solution Tanks for LS0AT-2

FIGURE 18. Accumulation of Fluoride in Scrub Solution Tanks for LS0AT-3

obtained from sulfates depositing on the filters. Because the filters would not be effective in removing SO_x gas, we can only conclude that the sulfate deposits on the filters in LSOAT-3 were re-entrained from the off-gas scrubbers. The consistently high DF for SO_x indicates that high scrubber differential pressures (>100 cm of water) are necessary to scrub and retain particulates, but only moderate differential pressures (46 cm of water) are necessary to remove SO_x gas from the off gas.

Comparison of the accumulation curve for sulfates in LSOAT-3 (Figure 15) with the power dissipation curve (Figure 8) reveals a direct correlation. The data indicates that the release of sulfates from vitrified soil is a direct function of melting rate and is independent of burial depth. However, comparison of the accumulation curve for fluorides (Figure 18) with the power curve reveals no such correlation. Fluoride behavior is similar to that of Sr, indicating that burial depth plays the controlling factor for fluoride retention.

The data indicate that the mist eliminators following the wet scrubber systems play an important role in removing particulates from the gaseous effluents. Manufacturer specifications (Euroform 1984) confirm that vane separators employed in the large-scale unit operate effectively only at high gas flows and differential pressures. By increasing the differential pressure from 46 cm to 102 cm of water, the flow increased from 80 std m^3/min to 110 std m^3/min . Assumably, this eliminated water droplet carryover and the tendency to re-entrain radionuclides from the scrub solution to the HEPA filters. Subsequent scrubber performance tests at the higher differential pressure showed that moisture losses from the scrub solution tank were due entirely to humidification of the off gas and not aerosol entrainment.

Since transuranic elements such as Pu and Am behave similarly to Sr, soil and off-gas DF measurements for Sr can be applied to compare expected stack concentrations with maximum permissible concentrations (MPC) published in 10 CFR 20 Table II (U.S. Code of Federal Regulations 1984). Oma et al. (1983) postulated the maximum concentration of Pu-239, Pu-240, and Am-241 at a contaminated soil site at Hanford to be 380 nCi/g, 100 nCi/g, and 1540 nCi/g, respectively. Vitrification of contaminated soil with these concentrations of Pu-239, Pu-240, and Am-241, at a vitrification rate of 4400 kg/h and a combined

soil and off-gas system DF of 1.3×10^{10} , produces the maximum stack concentrations listed in Table 4. The maximum stack concentrations fall well below the maximum permissible concentrations, thus indicating that the measured DF of 1.3×10^{10} for the large-scale system is quite sufficient for radioactive testing.

TABLE 4. Maximum Stack Concentrations of Pu-239, Pu-240, and Am-241 and Their Maximum Permissible Concentrations

<u>Transuranic</u>	<u>Maximum Stack Concentration, $\mu\text{Ci/mL}$</u>	<u>Maximum Permissible Concentration, $\mu\text{Ci/mL}$</u>
Pu-239	1.8×10^{-14}	1×10^{-12}
Pu-240	4.6×10^{-15}	1×10^{-12}
Am-241	7.5×10^{-14}	4×10^{-12}

SUITABILITY FOR RADIOACTIVE TESTING

In addition to conforming to the functional design criteria established for the large-scale ISV unit, the process must also exhibit other operational characteristics to be suitable for radioactive testing. These include the ability to enhance subsidence of the cold cap on the melt surface and the ability to predict operational characteristics under previously untested site geometries and waste inclusions.

COLD CAP SUBSIDENCE

During the initial test of the large-scale unit (LSOAT-1), a cold cap of porous molten soil formed on the surface of the melt, eventually rising above the original soil surface. This phenomenon creates a difficult closure problem requiring excessive backfill in the surrounding area and potential future subsidence. A rising cold cap also has the potential of bringing buried transuranics above the soil surface in the porous vitrified soil. Therefore, it was highly desirable to develop ways to eliminate the formation of the rising cold cap and to promote subsidence.

A great deal of experience has been amassed on cold cap subsidence with the 13 pilot-scale and 3 large-scale settings, each producing varying degrees of subsidence. A statistical analysis was employed prior to the third large-scale setting to determine the parameters that control cold cap subsidence. Although many factors are suspected to govern cold cap subsidence, two parameters have the greatest influence: graphite collars and surface insulation. Use of both helps to keep the surface molten, thereby encouraging gas release during vitrification; the graphite collars enhance the thermal conductivity of the electrode bringing heat to the surface, and the insulative layer keeps the vitrified surface molten. A 1 to 10 rating system was developed to quantify the degree of subsidence for each test conducted prior to LSOAT-3. The ratings were fit into a matrix shown in Table 5. A three-way fixed-effects-analysis-of-variance technique was used to help predict cold cap subsidence for the third large-scale setting (LSOAT-3). Although insufficient data were available to make a quantitative prediction, a qualitative judgment could be made that

TABLE 5. Statistical Matrix Ranking Cold Cap Subsidence with Subsidence Technique

Electrode Spacing	Without Graphite Collar				With Graphite Collar			
	With Insulation		Without Insulation		With Insulation		Without Insulation	
	Rating	No. of Tests	Rating	No. of Tests	Rating	No. of Tests	Rating	No. of Tests
0.6			2	1	10	1	10	1
1.2			3	2	8.5	2	6.7	6
4.5	4	1	1	1				

the graphite collars and surface insulation would be effective in causing the cold cap to subside below the soil surface. The results verified the statistical prediction with the cold cap subsiding between 50 cm and 80 cm, equivalent to a rating of 7.

Another suspected important parameter in suppressing cold cap formation is the oxidizing/reducing condition of the vitrified soil. Technical studies have been performed, relating the glass-foaming potential to the oxidizing/reducing condition of the feedstock for high-level nuclear waste vitrification processes. Goldman and Bright (1984) discovered that control of the ferrous/ferric ratio in the glass product between 0.1 and 0.3 is important to reduce the foaming potential. The ferrous/ferric ratio for LS0AT-2, which received a cold cap subsidence rating of 4, was 0.3. Since graphite is a reducing agent used in the glass industry to control gas bubble generation (Cable et al. 1968), we postulate that one of the reasons the graphite electrodes contribute to cold cap subsidence is their reducing potential. Core samples of the large-scale vitrified block produced with graphite collars are not yet available to verify this conclusion.

ABILITY TO PREDICT RESULTS

Another measure of the large-scale unit's readiness for radioactive testing is the ability to predict operational behavior during processing. A number of predictive tools have been developed to accomplish this objective. The mathematical model discussed previously (Oma et al. 1983) predicts melt

depth, width, run time, and energy requirements. The technical data on the retention of radionuclides and chemical contaminants in the molten soil and off-gas system are used to predict concentrations for different site geometries. These tools have been employed to predict the results of a LSVT, which has recently been conducted at a simulation of an actual contaminated soil site at the 216-Z-12 Crib at Hanford.

The 216-Z-12 Crib is a flat-bottom, 6.1-m by 91-m, rectangular excavation 6.1-m deep with 1.5 h:1 v sloping sides. The bottom 1.3 m of the crib has been filled with various gradations of course gravel, which is covered with a polyethylene membrane. At 0.91 m from the bottom, a 30-cm-dia perforated vitrified clay (VCT) pipe runs along the entire centerline length of the crib. The clay pipe, which received the waste from a diversion box, distributed the waste to the gravel layer.

The results of the LSOAT provide a strong data base from which to predict the results of the LSVT at the simulated 216-Z-12 Crib site. A satisfactory comparison of the actual results with the predicted results and the performance criteria will provide support for authorizing operations for the LSRT. Relevant results that must be confirmed by the verification test prior to conducting the radioactive test include the following areas:

- cold cap subsidence
- final block width
- melt depth as a function of time
- electrode voltage, current, and power as functions of time
- retention of Cs, Sr, and F in the block
- wet scrubber system DFs for Cs, Sr, SO_x, and F as measured by accumulations in the scrub solution and on the HEPA filters
- stack concentrations of SO_x and NO_x.

Predicted cold cap subsidence is based on the results of the matrix of tests conducted with the pilot- and large-scale equipment. Cold cap subsidence techniques, namely graphite collars with cold cap insulation, will be employed to achieve cold cap subsidence. Predicted subsidence is based on linear

extrapolation of the LSOAT-3 results, which produced a cold cap subsidence of 0.45 m at a vitrification depth of 3 m. At the 7.1-m depth planned for the LSVT, the cold cap subsidence can be expected to be as great as 1 m.

Geometric and electrical predictions are conservatively based on the high heat loss version of the mathematical model. The maximum block width at the completion of the test is estimated to be 7.5 m. The melt depth measured by the depth monitors is expected to follow the plot shown in Figure 19. Expected voltage, current, and power are shown in Figures 20 through 22.

The retention of Cs, Sr, and F in the vitrified product is determined by off-gas system samples during and after the run. In the cases of Sr and F, the retention figures will be verified by analysis of block cores after cooling. Anticipated soil-to-off-gas DF figures, based on measurements during the LSOAT, are 2×10^3 for Cs, 3×10^4 for Sr, and 80 for F.

Off-gas samples from the scrub solution and HEPA filters will determine the DF of the wet scrubber system for Sr, F, SO_x , and Cs. The accumulation of the first three elements will be plotted with time. The wet scrubber system DFs for each element are expected to lie between 140 and 400, as measured for F and Sr during LSOAT-3. These DFs will be more than adequate to conduct a

FIGURE 19. Predicted Melt Depth as a Function of Run Time for the LSVT

FIGURE 20. Predicted Electrode Voltage as a Function of Run Time for the LSVT

FIGURE 21. Predicted Amps as a Function of Run Time for the LSVT

successful radioactive test. Stack concentrations of SO_2 and NO_x provide the overall off-gas system DF for these gases. Concentrations of NO_x are not expected to surpass the detection limit of 0.05 ppm because the LSOAT has proven ISV as an effective NO_x destructor for contaminated soils. Besides, no significant nitrate deposits (<1 kg) will be added to the test site. The

FIGURE 22. Predicted Power as a Function of Run Time for the LSVT

natural sulfate deposits in the soil will decompose and be captured by the off-gas system. Overall system DFs are expected to lie between 1000 and 7000 as measured during the LSOAT. Maximum stack concentrations are not expected to exceed 200 ppb, well within established criteria.

VERIFICATION OF COST ESTIMATES

Cost is an important incentive when discussing the advantages of ISV. Buelt et al. (1985) have reported estimated production costs for the ISV unit to range from \$160/m³ to \$330/m³. The completion of three LSOAT settings provides adequate data to confirm these costs. The actual costs incurred to vitrify soil during preparation and operation of the LSOAT were within the estimated range of production costs. The costs include the operational labor, cost of electrodes, HEPA filters, other materials, site preparation activities (exclusive of site simulation activities), amortized capital, and energy costs. The operational experience confirmed the cost estimate assumptions of being able to move the hood and trailers in two shifts and of conducting the vitrification process with two operators. In a production environment, the operational efficiency would undoubtedly be improved to reduce the operational costs even further.

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