Recycle of Contaminated Scrap Metal Comprehensive Executive Summary

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1. Executive Summary

Catalytic Extraction Processing (CEP) has been demonstrated to be a robust, one-step process that is relatively insensitive to wide variations in waste composition and is applicable to a broad spectrum of DOE wastes. Catalytic Processing Unit (CPU) design models have been validated through experimentation to provide a high degree of confidence in our ability to design a bulk solids CPU for processing DOE wastes. Substantial growth in the feed size and composition compatible with CEP has also been achieved in a relatively short period of time, providing the opportunity to substantially reduce the amount of feed characterization and preparation prior to processing. Two commercial CEP facilities have been placed in commission and are currently processing mixed low level wastes. These facilities provide a compelling indication of the maturity, regulatory acceptance, and commercial viability of CEP.

Research and development activities directed at bulk solid feed systems have achieved significant growth in the feed particle size acceptable for Catalytic Extraction Processing. An extensive series of experiments was conducted on feed materials that were surrogates for a typical waste stream that was provided by DOE. Based on R&D completed to date, a CPU can be confidently designed to accept a drum size unit of completely characterized waste and an approximately 15.24 cm (6 inch)/3539.6 cm³ (216 inch³) cube of completely uncharacterized waste as shown in Figure 1.1. The particle

Figure 1.1
Growth in CEP-Compatible Bulk Solid Feed Particle Size

size for completely uncharacterized waste is compatible with commercially available shredder technology. The current factor limiting the size of uncharacterized waste is the uncertainty in the volatile content of the waste stream. This increase in the feed size corresponds to a significant reduction in the amount of waste characterization and feed preparation prior to injection into the Catalytic Processing Unit (CPU).

The information and data contained in this report were principally derived from activities performed under this contract. However, the terms and conditions of this contract limited experimental testing to radionuclide surrogates. In order to bolster CEP technology to demonstrate its viability for processing DOE mixed wastes, MMT performed experimental testing of actual radionuclides that was funded separately by MMT and customers other than DOE. These experiments validated product partitioning data obtained from radionuclide surrogate experiments performed under this contract. These data have been included in this report as it is known to be a major DOE interest item.

Catalytic Extraction Processing achieved a major milestone in December 1995 when the two commercial facilities shown in Figure 1.2 were commissioned and subsequently began processing mixed low level waste. These privately funded, state-of-the-art Quantum-CEP™ facilities were completed on schedule and employ sophisticated automation and control systems for processing of radioactive wastes. The M4 facility was constructed in less than 5 months and is capable of processing up to 1 million pounds per year of heterogeneous liquid and solid mixed hazardous and radioactive wastes. The commercial nuclear waste processing facility is a robotically-operated plant that is capable of processing 650 High Integrity Containers (approximately 130,000 cubic feet) per year of organic mixed low level wastes. These facilities have demonstrated both the maturity and the economic viability of utilizing CEP for commercial processing of various mixed low level waste streams. Further, these facilities, in combination with previous and on-going research and development activities at MMT, provide important risk reduction for future DOE efforts to apply CEP to site specific requirements and to expand its application beyond MLLW to transuranic (TRU) waste and High Level Waste (HLW).

An important factor to be considered when developing and deploying technologies for the recycle, reuse, and disposal of materials from DOE decontamination and decommissioning activities is the ability to obtain the necessary permits to process these materials. While conventional wisdom may characterize CEP as a thermal treatment system which will likely be permitted as an incinerator, MMT's experience in obtaining permits for CEP facilities indicates otherwise. Five states (Massachusetts, Ohio, Tennessee, Texas, and Louisiana) have characterized CEP as a legitimate recycling process that is a non-combustion, non-incineration technology. The Texas Natural Resource Conservation Commission (TNRCC) has determined that CEP is a recycling technology that is distinct from incineration and can be considered a manufacturing

Figure 1.2
Commercial CEP Facilities Currently in Operation

M4 Facility, Oak Ridge, TN

Commercial Nuclear Waste Processing Facility, Oak Ridge, TN

technology because it converts wastes to useful products. Further, the state of Louisiana has ruled that CEP is a manufacturing process which is exempted from all RCRA regulation, including up-front storage of secondary material. The designation of CEP as a recycling technology is significant as it results in the CEP recycling unit being exempt from Resource Conservation and Recovery Act (RCRA) permitting requirements. The state of Tennessee has issued permits for the two CEP facilities that are currently in operation for processing mixed low level wastes (MLLW). The headquarters of the U.S. Environmental Protection Agency has also recently recognized CEP as having achieved the Best Demonstrated Available Technology (BDAT) for processing all wastes for which incineration was previously the only approved processing method. EPA's determination reinforces the position that CEP is fundamentally different from incineration, and further confirms EPA's previous conclusions that CEP supports the agency's efforts to reduce the quantity of residuals for land disposal and to minimize cross-media contamination.

The research and development activities performed under this contract have demonstrated Catalytic Extraction Processing to be a robust, one-step process that is relatively insensitive to wide variations in waste composition and is applicable to a broad spectrum of DOE wastes. Substantial growth in the feed size and composition compatible with CEP has been achieved in a relatively short period of time; additional research and development is expected to lead to the ability to accept a drum (and potentially larger) size feed of completely uncharacterized waste. Experiments have also validated our CPU design model, providing a high degree of confidence in our ability to design a bulk solids CPU for processing DOE wastes. Two commercial facilities have been placed in commission and are currently processing mixed low level wastes. These facilities provide a compelling indication of the maturity, regulatory acceptance, and commercial viability of CEP. Additional development work to address the specific requirements of a particular DOE site, and expansion in the application of Catalytic Extraction Processing to transuranic and high level wastes is considered to be the next logical step in the development and deployment of CEP for the recycle, reuse, and disposal of materials from DOE decontamination and decommissioning activities.

1.1. Program Goals and CEP Applicability

The leaders of our nation are under continual and ever-growing pressures to decrease the size of government and decrease spending, while at the same time improve the environment and eliminate the hazardous waste problems plaguing many parts of the country -- seemingly conflicting goals. These are all daunting in their magnitude individually, and when considered together pose a truly monumental task. The U.S. Department of Energy issued a Program Research and Development Announcement (PRDA) in 1993 with the objective of identifying unique technologies which could be applied to the most pernicious waste streams at DOE sites, and at the same time derive some financial benefit from the application of the technologies.

The DOE identified the problem of Low Level Mixed Wastes as one of the most difficult it faced. The combination of radioactive contamination with additional contamination by hazardous constituents such as those identified by the Resource Conservation and Recovery Act posed an especially challenging problem. Technologies which had traditionally been called upon to solve these problems were coming under increasing attack by regulators and stakeholders. Recent investigations by the EPA had revealed that many of the byproducts of traditional remediation technologies were considered detrimental to human health at extremely low levels. The DOE searched for technologies which could provide superior regulatory performance, achieve decontamination of the contaminated metals and provide a cost benefit to users.

Specifically, the objective of identifying a process for recycling the metal constituent of the waste streams was identified. The DOE's own stockpiles of contaminated scrap metals amounts to an enormous untapped resource of metals from steel and copper to metals which are in short supply, such as nickel. Millions of tons of metal bearing mild contamination could potentially be brought back to use within the complex, or conceivably to general commerce in the event of the establishment of a deminimis standard. In either scenario, the re-use of the metals would pre-empt the purchase of virgin materials from sources outside the government, thereby decreasing the outflow of capital and conserving precious natural resources. The DOE controls large quantities of radioactive scrap metal as delineated in Table 1.1 below. The DOE's supply of contaminated nickel alone is estimated to have a market value approaching \$1,500,000,000.00. By recovering these unexploited resources, DOE decreases U. S. dependence on foreign sources for strategic materials, such as Ni, Nb, and other materials vital to national security.

Table 1.1
Radioactive Scrap Metal Controlled by DOE

Metal	Market Price (US \$/ton)¹	Quantity (tons)	Market Value (US \$)
Nickel	6,000	245,000	1,470,000,000
Steel	107	1,300,000	140,000,000
Aluminum	600	195,000	115,000,000
Copper	1,500	38,000	57,000,000

¹Nominal Prices 1990-91

Annual production estimated at 15,000 tpy

Source: Environmental Restoration Opportunity-Recycle of DOE Contaminated Metal, William Murphie, et al., USDOE 1992.

Desirable recycling technologies were described by the DOE as easily installed, operated and maintained, exhibit superior performance at low cost, generate little secondary waste, remove contaminants to very low levels, reduce worker exposure to contaminants and be readily accepted by the public and regulatory agencies. Molten Metal Technology, Inc., (MMT) was awarded a contract as a result of the PRDA, to determine whether its proprietary technology, Catalytic Extraction Processing (CEP), could be applied to waste streams the DOE had identified, and meet the exacting requirements of a broad spectrum of evaluators.

Molten Metal Technology Inc.'s (MMT's) Catalytic Extraction Processing (CEP) is a proprietary technology that allows organic, organometallic, metallic and inorganic feeds to be recycled into useful materials of commercial value. CEP is an innovative technology that provides superior environmental performance, no cross-media transfer of pollutants, substantial pollution prevention/waste minimization benefits, and recovery of strategic resources of economic value. CEP can achieve or surpass existing concentration-based BDAT standards while also meeting EPA's goals under important initiatives, such as the Combustion Strategy and the Pollution Prevention Policy.

MMT's goal is to maximize recovery of feed materials on an elemental basis in a safe, efficient, and effective manner. At the core of CEP is a metal bath, operated at temperatures above its liquid state. The liquid metal acts as a catalyst and solvent in the dissociation of the feed and the synthesis of products. Processing of materials in CEP can be conceptually divided into two stages:

- Catalytic dissociation and dissolution: The catalytic effect of the molten metal causes complex compounds in the feed to be dissociated into their elements, which readily dissolve in the liquid metal solution to form elemental intermediates. The formation of dissolved elemental intermediates ensures that only elemental composition--not the physical form or molecular structure of the feed--affects CEP's performance.
- Product synthesis: By adding select co-reactants and/or controlling operating conditions, the dissolved elemental intermediates can be reacted to form desired products of commercial value. Thermodynamics determine product synthesis, while solution equilibria determine partitioning between the metallic, ceramic, and gaseous product streams.

1.1.1. CEP Technology Overview

CEP is a robust chemical process whose performance is based on fundamental chemical principles and engineering design. Given the chemical composition and physical form of the feed, the reaction pathways can be accurately predicted based on thermodynamics and the desired reactions can be carried out under appropriate engineering design. The process robustness while maintaining system performance is in contrast to combustion processes, where competing reactions take place and no accurate predictions of processing results can be achieved. The major technical features that allow CEP to provide significant environmental performance and waste minimization benefits are outlined below:

- CEP feed conversion efficiency is independent of the complexity of the molecular structure of the feed molecule. The liquid metal acts both as a homogeneous catalyst for the dissociation of complex feed molecules into their elemental constituents and as a solvent of these elemental intermediates. The flexibility and robustness of the CEP process are attributed to the 'singular' dissolved elemental intermediate through which reactions proceed. As a result, CEP feed conversion is independent of the complexity of the molecular structure of the feed molecule. DREs exceeding 99.9999% ('six nines') are typical in CEP regardless of the complexity of feed materials.
- CEP feed conversion efficiency is driven by solvation effects. CEP operates above the metal liquidus point, but does not use temperature as the primary means to change the physical and chemical composition of the feed materials. The carbon concentration in the liquid metal drives feed dissociation and dissolution. The lower the carbon concentration relative to saturation, the higher the forces driving the formation of the dissolved elemental intermediate (i.e., $\text{Fe}_3\text{C}(\delta)$) and hence more efficient feed conversion per unit time is achieved.
- CEP operates under a highly reducing environment, preventing the formation of undesirable oxidation byproducts (e.g., NO_x , SO_x). The solubility of carbon and oxygen in iron provides CEP with high chemical inertia and allows the system to maintain a highly reducing uniform environment over a wide range of operating conditions. No free oxygen can be found in the system to undergo alternate oxidation reaction pathways which lead to undesirable byproducts, such as NO_x , SO_x , dioxins, and difurans.
- CEP does not provide pathways for the formation of dioxins or difurans. Synthesis via CEP is achieved through specific dissolved intermediates in a highly reducing environment. No viable pathways are allowed for dioxins and difurans formation. Analysis of the CPU off-gas stream in commercial-scale demonstrations of chlorinated waste processing have consistently shown trace levels to be non-detect to the stringent targeted regulatory level of 0.1 ng/Nm³ (TEQ).

- CEP allows synthesis of high quality products. Synthesis of products is based on manipulation of the reaction pathways for the dissolved elemental intermediate through judicious addition of co-reactants and variation in operating conditions.

The liquid metal bath and the reaction pathway via a singular dissolved intermediate give CEP a number of distinct advantages over conventional thermal processes:

- The liquid metal is a homogeneous catalytic and solvation medium which dissociates the feed into its elements, ensuring no intermediate products of incomplete dissociation. In open flame processes, the extent of dissociation to the intermediate free radicals is dependent on the residence time, temperature, oxygen environment, and system turbulence which are difficult to control. These parameters are readily controllable in CEP.
- The liquid metal solvent has high chemical inertia providing a means of dampening the effect of variations in feed composition, hence allowing for robust control of product quality.
- In CEP, the liquid metal in contact with the liquid ceramic phase can be used to engineer the separation and recycling of inorganics or metal compounds from complex feeds. This multiphase interaction provides CEP with the flexibility to synthesize and recycle desired products of high commercial value.

1.1.2. Material Recovery and Reuse

CEP converts secondary materials into products including synthesis gas, ceramic products and ferrous as well as non-ferrous alloys (carbon steel, stainless steel, nickel, chromium, etc.).

Based upon market analyses, theoretical product modeling, and experimental verification of radionuclide partitioning and resultant product properties, MMT has identified multiple potential uses for Quantum-CEP™ products from the processing of DOE radioactively and RCRA-contaminated scrap metal and other waste forms expected to be generated by the decontamination and decommissioning (D&D) of DOE sites.

Synthesis gas (hydrogen and carbon monoxide) can be used as a low NO_x fuel. CEP product gases usually have a heating value of approximately 300-350 BTU/SCF, or approximately one-third that of natural gas (~1000 BTU/SCF).

Metal alloys containing deminimis radioactivity may be used for containment boxes, for shielding purposes, and with the establishment of a deminimis standard, for release to commerce. Without the establishment of a deminimis standard, multiple opportunities exist within the DOE complex for beneficial use of the recovered decontaminated metal product. Table 1.2 delineates some of these potential metal

products. With the establishment of a deminimis standard, such as in Europe, large commercial opportunities for scrap metal recycling would become available. Recovered condensed products from MMT's Fall River facility have been sold for \$40-\$4140/ton.

Table 1.2
Metal Products for Reuse within the DOE

Product	Material	Total Weight (tons)
Shield Blocks	Carbon or Stainless Steel	300,000
Vitrification Canisters	Stainless Steel	45,000
Universal Dry Fuel Casks	Stainless Steel	282,000
Transportation & Disposal Boxes	Carbon or Stainless Steel	500,000
Transportation & Disposal Drums	Carbon or Stainless Steel	1,250,000
Type A Shipping Casks	Carbon or Stainless Steel	150,000
Reinforcing Bar and Mats	Carbon or Stainless Steel	100,000
Totals:		2,627,000
Source: Workshop on Radioactive Scrap Metal Knoxville and Oak Ridge, TN University of Tennessee, via SEG 1993		

Many radionuclides found in DOE waste, such as U and Pu, will partition to a ceramic phase. MMT is currently exploring potential uses for these products. These materials may be useful as barriers or structural support in repositories. The ceramic composition may be appropriate for use as grouting material for buried high level waste (HLW) tanks. The glass formers found in the CEP product may make it an appropriate substitute for virgin glass to be used in a vitrifier of high level wastes and would be consistent with the objectives of MAWS (Minimum Additive Waste Stabilization). MMT's marketing and regulatory groups are exploring these potential uses to make beneficial use of all product streams from CEP and Quantum-CEP™.

1.1.3. CEP Benefits Lead to Stakeholder Endorsement

MMT has built wide regulatory and community acceptance of CEP as a recycling technology. MMT has successfully obtained approval and acceptance from both state and federal government agencies that CEP is a global solution for environmental protection. This acceptance will expedite siting and permitting, thereby shortening commercialization schedule and lowering overall project cost (Table 1.3).

Table 1.3
Regulatory "Certifications" of CEP

Project	Time
U.S. EPA	
Determination that CEP provides Equivalent Treatment for all wastes which have a treatment standard of combination/incineration	Approved 2/96
EPA Region I Environmental Merit Award	April 1995
Determination that CEP provides Equivalent Treatment for RCRA-listed chlorinated wastes (F024) for which incineration had been mandated Best Demonstrated Available Technology	Filed 12/94 Approved 7/17/95
Determination that CEP provides Equivalent Treatment for eight RCRA-listed isocyanate waste codes for which incineration had been mandated Best Demonstrated Available Technology	Filed 4/22/94 and 5/5/94 Approved 10/24/94
EPA Metal Recovery Report to Congress (CEP featured as "Innovative Metal Recovery Technology")	Report to Congress June 1994
Determination that CEP provides Equivalent Treatment for all wastes for which incineration had been mandated Best Demonstrated Available Technology	Approved 3/12/96
Massachusetts	
MADEP Recycling R&D Permit (Fall River)	Filed 4/13/92 Approved 9/17/93
MADEP Application for R&D Recycling Certification (CEP of Biosolids Recycling)	Filed 10/31/93 Approved 12/1/93
MADEP Application for R&D Recycling Certification (CEP of Surplus Electronic Componentry)	Filed 3/15/94 Approved 5/17/94
MADEP Application for R&D Recycling Certification (CEP of Chlorinated Organic Hazardous Waste, F024, K019 and K020)	Filed 12/9/94 Approved 1/24/95
MADEP Application for R&D Recycling Certification (CEP of Toluene Isocyanate Residue K027)	Filed 1/30/95 Approved 3/1/95
Ohio	
OH EPA Preliminary Recycling Analysis (CEP recycling exempt from RCRA-permitting at central hazardous processing facility)	Filed 3/4/94 Approved 6/28/94
Texas	
TNRCC Designation as "Innovative Technology"	Approved 8/4/93
THRCC Regulatory Analysis (CEP processing considered legitimate recycling exempt from RCRA - permitting requirements)	Approved 2/27/96
California	
Application for Technology Certification (Certification of CEP as a Recycling Technology)	Filed 10/28/94 Approved 6/95
Tennessee	
DEC (Department of Environment & Conservation) determination that CEP is not combustion and constitutes legitimate recycling.	Approved 2/20/96
Louisiana	
LADEQ (Department of Environmental Quality) determination that CEP is a manufacturing process and is exempted from all RCRA regulation.	Approved 9/30/96

1.1.4. CEP Benefits Complement DOE Program Goals

The unique processing benefits of CEP combined with the material recovery attributes and broad regulatory acceptance serve the needs of DOE as outlined in the Program goals. The goals of Research Area 5, which specifically address the recycle of contaminated scrap metal, are listed in Table 1.4, with a corresponding listing of CEP's capabilities which meet or exceed the DOE's requirements.

Table 1.4
CEP Meets and Exceeds the DOE's Program Objectives

Research Area 5 DOE Program Goal	CEP's Capabilities
Develop a novel technique that will significantly reduce the potential for spread of contaminants and for worker exposure to radionuclide or hazardous material.	Through reactant addition, radioactive nuclides are engineered into a high density vitreous phase and hazardous components are rendered benign. The tightly-bound ceramic matrix will reduce potential spread and safe operation will assure no worker exposure. A sealed reactor design will prevent fugitive emissions, and up-front automation, protective clothing and monitors will prevent accidental worker exposure.
Develop processes to convert the scrap metal into a form that can be reused.	Reducible metals, such as Fe, Ni, Cr, etc., are captured in a molten metal phase, radionuclides are diverted to a vitreous phase, and hazardous organics are converted to H ₂ and CO and liberated as gas. The scrap metal is thus converted to a high-value ferrous alloy.
Minimize secondary waste generation.	The only potential waste material from CEP of scrap metal is the radioactive nuclide-containing vitreous matrix. Because the nuclides are concentrated in this phase, and the volume of this phase is low, waste generation is minimized. Recovery of the radionuclides for potential recycle may be possible in the future.
Comply with existing environmental, health, and safety regulations.	A CEP facility can render RCRA hazardous and TSCA toxic organics benign, capture volatile heavy metals, and concentrate/immobilize radioactive constituents. MMT will meet all EPA, CERCLA, DOE, TSCA, state and local standards.
Develop technologies that produce a reusable form, which is not hazardous under the provisions of RCRA.	CEP of the contaminated scrap metal will form two non-hazardous products: ferro-alloys and syngas. Organic hazardous constituents will be destroyed to form products, CO and H ₂ . Heavy metal contaminants can be captured and immobilized. The soluble metals, such as Fe and Ni, will concentrate in the metal bath, and other contaminants will be diverted to a ceramic phase.
Develop technologies that produce a reusable form that meets proposed IAEA standards for unrestricted release of bulk contaminated materials.	Preliminary partitioning (thermodynamic equilibrium) studies indicate that the concentrated metal product (ferro-alloy) may contain less than 0.2 Bq/g for alpha emitters and less than 1 Bq/g for high energy beta/gamma emitters.
Develop technologies that remove radioactive and hazardous materials from the scrap metal.	In CEP, the metal is captured as a ferro-alloy, while the radioactive species are reaction-engineered into a ceramic phase. Hazardous organics decompose into elemental constituents and then react to form H ₂ , CO, etc. Hazardous heavy metals may be captured either in the ceramic phase or condensed from the product gases. Therefore, radioactive, hazardous, and toxic constituents are removed from the metal.
Develop technologies with lower decontamination and recycling costs compared to current technologies.	The chemical inertia and thermal efficiency of the molten metal bath makes CEP processing highly cost-effective. In many commercial applications, the resale of two product streams offsets the processing cost.

1.2. Program Objectives and Accomplishments

The DOE sought to identify technologies which could recycle the valuable constituents of the feed streams and at the same time economically produce a suitable final waste form for disposal while achieving unprecedented environmental performance. While technologies existed to meet individual components of the overall program objectives, no technology had been demonstrated to achieve all components simultaneously.

In concert with the DOE, MMT established overall program objectives at the inception of the project which would challenge preconceptions of the limitations of waste processing technologies:

- *Demonstrate the recycling of ferrous and non-ferrous metals* -- to establish that radioactively contaminated scrap metal could be converted to high-grade, ferrous and non-ferrous alloys which can be reused by DOE or reintroduced into commerce.
- *Immobilize radionuclides* -- that CEP would concentrate the radionuclides in a durable vitreous phase, minimize secondary waste generation and stabilize and reduce waste volume.
- *Destroy hazardous organics* -- that CEP would convert hazardous organics to valuable industrial gases, which could be used as an energy source.
- *Recover volatile heavy metals* -- that CEP's off-gas treatment system would capture volatile heavy metals, such as mercury and lead.
- *Establish that CEP is economical for processing contaminated scrap metal in the DOE inventory* -- that CEP is a more cost-effective and complete treatment and recycling technology than competing technologies for processing contaminated scrap.

The execution of this program resulted in all objectives being met and exceeded as delineated in Table 1.5.

Table 1.5
PRDA Objectives Met and Surpassed

Program Objective	Representative MMT Accomplishments
Recycling of ferrous and non-ferrous metals	<ul style="list-style-type: none"> • 100% metal recovery for surplus metal componentry (64 wt% Ni, Cu, Cr, Fe, Ag, Co, Mo, Mn) • Decontamination of metals > 99.6% (analytically limited) • Recycling certification by MADEP (Recovered condensed phase products sold to local metal recycler.)
Immobilization of radionuclides	<ul style="list-style-type: none"> • Incorporation of both surrogates and actual radionuclides into stable glass structure proven using neutron activation analysis • All glass compositions passed TCLP
Destruction of hazardous organics	<ul style="list-style-type: none"> • DREs $\geq 99.9999\%$ for halogenated organics (K019, K020, F024), toxic organics (K027), plastics (PVC, PS), etc. • Negligible trace components, $\text{NO}_x < 3 \text{ ppm}$, $\text{SO}_x < 1 \text{ ppm}$, TEQ (2,3,7,8 TCDD) $< 0.1 \text{ ng/Nm}^3$ (stringent targeted regulatory limit) -- third-party analysis
Recovery of volatile heavy metals	<ul style="list-style-type: none"> • Demonstrated partitioning of volatile heavy metals to targeted phase • Volatile heavy metals engineered into durable ceramic product
Cost effective recycling of contaminated metal	<ul style="list-style-type: none"> • System design established with proven regulatory acceptance • Minimal feed analysis and post-treatment resulted in streamlined system with low capital and operating costs • No secondary combustion or vitrification required

1.3. Significant Findings and Program Overview

The information and data contained in this report combines the results of studies/experiments performed under this contract with research and development efforts that were funded separately by customers other than DOE. In particular, experimental information addressing radioactive waste was not performed under this contract, but has been included in this final report as it is a major DOE interest item.

Before designing the experiments to verify CEP's applicability to contaminated scrap metal, MMT performed an extensive literature search to establish the existing state-of-the-art in related fields. Previously published studies from numerous sources, including Oak Ridge and Argonne National Laboratories have demonstrated the ability to partition transuranic components, such as uranium and plutonium, from the metal phase into a vitreous phase via melt refining. Residual concentrations ranging from 0.05 ppm to 2 ppm of such radioactive components were achieved using diffusion of oxidizing, vitreous-forming agents to partition the radioactive components. Successful partitioning to less than 10 nCi levels has been demonstrated for uranium- and plutonium-contaminated mild steel, stainless steel, nickel, copper, and aluminum. While melt refining technology can enable partitioning of radionuclides, it can not simultaneously ensure complete conversion of RCRA organic constituents and containment and immobilization of RCRA inorganic constituents. Hence, melt refining is not applicable to DOE mixed wastes which are highly diverse in chemical and physical form, and may contain high concentrations of RCRA and/or metal components.

CEP can offer simultaneous radionuclide partitioning, recovery of RCRA inorganics, and conversion of RCRA organics for a diverse and ill-defined waste feed slate. CEP offers equivalent and potentially better radionuclide partitioning capabilities to melt refining, as the techniques involved are completely incorporated and enhanced in CEP technology. CEP incorporates active radionuclide partitioning through select co-reactant additions and enhanced mass transfer (e.g. convection), while melt refining is based upon "passive" diffusion-based partitioning. For example, CEP can directly add co-reactants into the metal bath. By injecting oxygen directly into the iron bath, oxidation of the radioactive components is greatly enhanced. Thus, significantly better partitioning is achieved with CEP than with traditional melt refining technologies. Bench, pilot, and demonstration-scale tests on common isotopes and DOE-recommended surrogates support these observations. Thermodynamic calculations support the experimental findings and both indicate that pre-contamination radioactivity levels (e.g., virgin metal levels) in the metal can be attained.

Demonstration- and pilot-scale studies have shown that metal decontamination can be achieved simultaneous to RCRA waste conversion. Surplus metal componentry (RCRA characteristic waste, when discarded) doped with radionuclide surrogates (Hf) and common isotopes (Zn) was successfully recycled via CEP. Organic conversion exceeded 99.9999% (DRE), metal recovery exceeded 98 wt% of the feed, and no

radionuclide surrogate was detected in the metal (analytically limited). The vitreous product from this demonstration was shown to be durable and chemically stable, passing all criteria for EPA TCLP analysis. Based upon detailed material balance analysis, product analysis, and evaluation of commercial product specifications, the Massachusetts Department of Environmental Protection (MADEP) certified this demonstration as recycling.

The report highlights the comprehensive nature of the efforts and key findings of the program. CEP's capabilities for targeted partitioning of radioisotopes to a stable, non-leachable phase with resultant metal decontamination and RCRA conversion were proven.

1.3.1. Support Facilities and Scope of Expanded Experimental Effort

MMT operates a Recycling-Research & Development Facility in Fall River, MA. The facility houses four bench-scale units, four pilot-scale units, seven physical models, and a commercial demonstration prototype unit, which are shown in Figure 1.3. This state-of-the-art facility is fully permitted by the Commonwealth of Massachusetts for recycling demonstrations using hazardous and non-hazardous materials as CEP feeds. MMT has received recycling certifications from the Massachusetts Department of Environmental Protection for the processing of RCRA and organometallic feeds and has sold the condensed phase products from this facility.

Bench- and pilot-scale experimental units are used to confirm theoretical modeling predictions, establish product quality, determine appropriate materials of construction, assess design parameters and confirm fundamental process chemistry. Many of these systems, including the Advanced Processing Units (APUs) which are appropriate for small commercial-scale operations, are operated round-the-clock for two to three-week experimental campaigns. Physical models are used to study fluid dynamics and transport phenomena, reactor flow patterns, unique reactor configurations, and final design parameters.

The commercial-scale prototype is used primarily for customer and regulatory demonstrations and establishment of commercial design scenarios. MMT has processed a wide range of hazardous, characteristic, and surrogate waste feeds in the commercial prototype. The commercial prototype typically operates round-the-clock during one-week demonstration campaigns, which may include several different feed materials. CEP demonstrations have proven waste recycling into commercially valuable products (>90% of the feed elements) while consistently achieving DREs $\geq 99.9999\%$. Trace components measure non-detect to the regulatory limits.

The demonstration-scale CPU and gas handling unit is equipped with approximately 2,000 monitored variables and 900 control loops linked into the computerized monitoring and control modules. Numerous sampling ports throughout the gas handling train enable full characterization of the product gas quality. Summa canister

sampling is performed to enable third-party analysis down to ppb levels via established EPA methods (TO-14). Mass spectrometer samples are drawn on-line and gas composition measurements are taken every twenty seconds for feed-back process control.

Figure 1.3
CEP R&D Units

Physical Model

Bench Scale

Pilot Scale

Commercial Scale

1.3.2. Task 1.1 - Design CEP System

1.3.2.1. Bulk Solids Feeding System Design and Analysis

An extensive series of theoretical and experimental studies were conducted on CEP reactor design modeling. Thermodynamics and computational fluid dynamics have been used to characterize the catalytic extraction process to develop a methodology and models for determining CEP reactor design parameters. Several studies were performed on the phenomenology of tuyere operation for injecting feed materials into the reactor. An accretion model was developed to predict the formation of accretions and to evaluate methods for their control. Studies were performed on the absorption behavior of a gas injected into a bath using a tuyere to characterize the various regions surrounding the tuyere plume. These studies provided important reactor design parameters including minimum bath height and maximum tuyere flow rates for effective processing. Extensive theoretical and physical model studies were performed on determining bubble size distributions from tuyere operation in molten metal baths. Calculated bubble size and residence time distributions were determined to be consistent with experimental results. Data obtained from the bubble size distribution study was then applied to a study on the carburization and decarburization of a molten metal bath. The inherent ability of the metal bath to dissolve carbon maintains the quality of the gas phase products by removing any unreacted hydrocarbon species. Finally, the data from these and other reactor-related experiments were used to design and evaluate an innovative, continuous, counter-current reactor which has the potential to yield high quality metal and ceramic products, reduce the volume of the ceramic phase, and reduce overall energy costs.

A critical requirement in DOE's efforts to recycle, reuse, and dispose of materials from its decontamination and decommissioning activities is the design of a robust system capable of processing a wide variety of bulk solid feeds. There are several reactor concepts for bulk feed processing being considered. Common to these concepts is the use of two processing zones: a dissolution/volatilization zone (Zone 1) and a gas polishing zone (Zone 2). A comprehensive series of experiments was performed in the 4-Ton Demonstration Unit using DOE-provided surrogates of typical waste components to obtain data to characterize the dissolution/volatilization zone and to compare that data with predictions based on theoretical calculations. In the continuous treatment of bulk solids in the CEP process, solids are fed into Zone 1 where they are dissolved and/or volatilized at a rate that is dependent on particle size, material properties, and bath conditions. The dissolution process can be mass transfer or heat transfer controlled. The operability range of a reactor can be determined for a given feed material, approximate size range, and specified reactor conditions. The boundaries of the operability range are established by the maximum power capacity of the system, the maximum feedable particle size, the maximum capacity of the gas handling train (GHT), the mass or heat transfer limit, and the volatility of the feed material. A typical reactor operability range for a mass transfer controlled process is

shown in Figure 1.4. A high degree of correlation was observed between data obtained during the experimental test program and theoretically-based Zone 1 design equations, thereby providing a high degree of confidence in our ability to design a reactor for processing bulk solids wastes generated from DOE decontamination and decommissioning activities.

Figure 1.4
Operability Limits for a Mass Transfer Controlled Process

1.3.2.2. Energy Addition

The addition of a horizontal processing extension to a CEP reactor for processing of bulk solids increases overall process flexibility; however, it presents several challenges including bulk feed addition, secondary treatment, condensed phase product removal, and energy addition. The energy addition requirements for a CEP reactor include pre-heat of both wetted and non-wetted containment surfaces, melt of the initial charge, provide sensible, latent, and reaction heat for feeds, maintain metal and ceramic phase product temperatures to enable secondary processing and product removal, and to enable the system to recover from any process upsets that may occur during processing. Figure 1-5 displays the temperature in the vertical headspace and horizontal sidearm of a CEP reactor as a function of distance from the spout (i.e., the center of the bath). Temperature losses in the sidearm drop relatively rapidly, which requires supplemental energy addition in the sidearm to maintain the condensed phase products operable.

Figure 1-5
Radiative Heat Transfer from Induction Heating

MMT conducted an experimental program to evaluate the use of a plasma torch to provide supplemental heating in the sidearm of the APU-SE reactor. Use of a plasma torch for supplemental heating offers numerous advantages, including the flexibility to provide either localized or global heating, and to vary its location within the reactor. While plasma torches are commercially-available, they were not designed to operate in the closed, splashing environment of a CEP reactor; therefore, MMT made a number of modifications to the torch to facilitate its operation in the APU-SE reactor. Figure 1-6 presents the results from plasma torch integrated testing in the APU-SE, which clearly demonstrates the increased headspace temperature resulting from plasma torch operation.

Figure 1-6
Results from Plasma Torch Integrated Testing in APU-SE

The experimental program successfully demonstrated the flexibility and robustness of using a plasma torch for supplemental heating, including reactor pre-heating, sidearm heating, and headspace skulling minimization. Further, the plasma torch provide superb recovery from an process upset created by an unintended trip of the induction furnace for approximately 15 to 20 minutes. The plasma torch provided sufficient heating to maintain the bath in an operable condition while correcting and recovering from the process upset. Based on these experimental results, supplemental heating using a plasma torch has been integrated into the conceptual design of a CEP system for processing bulk solids.

1.3.2.3. Product Removal

In order to fully realize the benefits of CEP application to DOE wastes, it is necessary to be able to efficiently remove condensed phase products from a reactor, preferably without interrupting the processing of feed materials. MMT has developed and demonstrated an innovative approach to product removal that utilizes an induction coil and susceptor pipe as shown in Figure 1.7. When the valve is in the closed state, the susceptor pipe is plugged with solidified product. The valve is opened by activating the induction coil which melts the solid plug, thus allowing the product to flow through the susceptor pipe into product containers/molds. Metal phase product removal is accomplished through a vertical tapping valve, while ceramic phase product

removal is accomplished through a horizontal tapping valve. Remote-controlled, in-process (i.e. reactor tipping not required) product removal using this design has been successfully demonstrated on a pilot scale, providing the basis for continued evolution to commercial-scale product removal system design and development required for processing DOE wastes.

Figure 1.7
Pilot Scale Tapping Valve Design

1.3.2.4. On-Line Sensors and Instrumentation

Development of advanced sensors, models, and techniques to monitor key process parameters is critical to robust, automated CEP operation. MMT has successfully a robust reactor control system which utilizes commercial off-the-shelf equipment to the maximum possible extent to monitor critical reactor parameters including bath temperature, headspace temperature, bath composition, bath level, bath pressure, containment system, tuyere injection, and visual observation. Emphasis was placed on

the use of non-invasive techniques to minimize both the potential for interruption in the flow of operations and potential delay opportunities for feedback to optimize process control. For example, an IR lightpipe sensor has been successfully demonstrated to provide in-situ, continuous, lag-free, and non-invasive sensing of bath temperature. A 24-hour period of automatic bath temperature control of the 4-Ton Demonstration Unit using a lightpipe is shown in Figure 1.8. Similarly, sensors for the remaining critical reactor parameters and control system hardware/software which integrates the data provided by these diverse sensors into a robust reactor control system have also been successfully demonstrated. Further, these sensors and their control system have been successfully implemented into the two commercial CEP facilities which are currently processing mixed low level wastes.

Figure 1.8
IR Lightpipe and Automatic Temperature Control Results

1.3.2.5. Particulate Characterization and Control

CEP has been identified as a technology whose robustness allows the processing of extremely heterogeneous wastes which are likely to contain volatile heavy metals and dust-producing elements. MMT has successfully developed and demonstrated particulate control methods based on the basic mechanisms for dust and particulate generation. Innovations in gas handling train technology and operation have led to both the suppression of dust formation, the recyclability of any dust that may form during processing, and the recovery of volatile heavy metals. Research and

development efforts directed at particulate suppression indicate that with the exception of species predicted to volatilize in our system (e.g. lead, mercury), less than 2% will be carried over as dust and can be recycled to the bath (e.g. FeO). Further, the distribution in the particle size is skewed heavily towards the large end of the scale (i.e. greater than 10 microns), which makes them relatively easy to separate from the gas stream. The result is a system which produces a clean gas product that exceeds industrial specifications using proven, reliable technology.

1.3.3. Task 1.2 - Experimental Test Program

MMT developed and executed a comprehensive experimental test plan that was designed to provide performance data on the application of the Catalytic Extraction Process to DOE wastes. The test plan was developed to demonstrate selective radionuclide partitioning and immobilization using surrogates; destruction of hazardous organic constituents; partitioning of non-radioactive recyclable metals, and processing of bulk solids. These experiments also addressed waste feed management, safety management, general operations management, and off gas management. The results of this experimental test plan, which are used throughout this final report, have successfully demonstrated the versatility of CEP to process a broad spectrum of waste materials in the DOE complex. A listing of the experiments performed under this contract have been categorized by major tasks in the statement of work and are presented in a separate appendix. It should be noted that experiments addressing the partitioning of actual radionuclides were not funded by the current contract; therefore, they have not been included in these tables.

1.3.4. Task 1.3 - Experimental Testing

A substantial amount of theoretical work has been performed on the separation of chemically similar metals such as technetium (Tc), uranium (U), and nickel (Ni). It is clearly desirable to separate these long half-life contaminants from short half-life and nonradioactive constituents of the waste material and contaminated metals. With uranium this is more readily accomplished due to the ease of oxidizing its oxide (UO_2) into its dication (UO_2^{2+}). Technetium also forms a dioxide (TcO_2) and it is also possible to form a similar dication (TcO_2^{2+}). If TcO_2^{2+} forms readily, technology can be developed to effect a phase transfer of technetium from its metallic state to an ionic or vitreous phase. High level non-empirical quantum calculations on Tc- and Ni-oxides were performed to derive estimates of the energetics of reactions which can potentially be used for the separation of technetium from nuclear waste or metal surfaces. Theoretical results suggest that oxidation and subsequent migration from the metal phase to the ceramic phase should have a much more favorable equilibrium constant for extraction of technetium oxides (TcO and TcO_2) than for the nickel oxides. Given that the oxidation potential for the uranium oxides (UO and UO_2) are significantly lower than those of technetium, the oxidation of technetium metal into its oxide in the presence of nickel and its subsequent transfer from the metal phase to the ceramic phase is

considered to be feasible. The calculated value of the Gibb's Free Energy for oxidation of technetium suggests that the reaction is thermodynamically favorable; however, additional work is required to assess the accuracy of the computational data and to estimate the free energy of the TcO_2 phase transition from available experimental data. This important theoretical work will provide the foundation for a future experimental program which may lead to the ability to recycle and reuse the approximately \$1.5 billion of radioactive contaminated nickel in the DOE inventory.

A series of experiments was performed with a divinyl benzene organic combined with either surrogate or actual radionuclides to measure the partitioning of radionuclides in the metallic, ceramic, and gas phases. The radioactive portion of these experiments was not performed under this contract; however, the data has been included in this final report because it is known to be a major DOE interest item. The focus of these experiments was to establish operating requirements to partition radionuclides to the desired phase. MMT has successfully demonstrated that CEP can substantially reduce the volume of radioactive wastes while producing a highly stable waste form for eventual disposal. As predicted, processing conditions enabled complete capture of non-volatile radionuclides into the metallic and ceramic phase. Further, MMT has successfully demonstrated the ability of CEP to control partitioning of radionuclides to either the metal phase which provides a stable waste form with significant self-shielding (the self-absorption of radiation-reducing radiation levels), or to the ceramic phase which also provides a stable waste form and achieves substantial volume reduction. Volatile radionuclides were captured from the gas phase with measured decontamination factors in excess of 10^4 . These experiments clearly demonstrate CEP's ability to recycle and reuse radioactive contaminated wastes, effectively control partitioning to the desired product phase, substantially reduce the volume of radioactive wastes, and produce a highly stable waste form for eventual disposal.

Two major experimental campaigns were conducted under ECP III of this contract: bulk solids processing and the effects on the partitioning of radionuclide surrogates in the presence of halogens, alkali metals, sulfur, and phosphorus. The bulk solids experimental program consisted of two elements: characterization of the dissolution/volatilization zone (Zone 1) and validation of Zone 1 design models, and multi-zone processing of bulk solid feed materials using a reactors-in-series configuration. The results of the Zone 1 characterization experiments were previously addressed in Section 1.3.2.1.

MMT performed a comprehensive experimental test program focused on determining the environmental performance of close-coupled reactors connected by a transfer pipe for processing bulk solid materials. The reactors-in-series configuration is presented in Figure 1-9. The design and operation of the transfer pipe leveraged the results of particulate characterization and control experiments performed during this contract.

Figure 1-9
Reactors-in-Series Configuration

Polypropylene and toluene diisocyanate (TDI) residue were used as feed materials and were introduced into Zone 1 using a lock hopper. Data from both Zone 1 and Zone 2 were collected using the mass spectrometer, two Gas Chromatographs to obtain speciated hydrocarbon information, two THC analyzers, and two CO/CO₂ analyzers. Representative data from the experimental campaign are presented in Figure 1-10 and Figure 1-11. Figure 1-10 compares the total hydrocarbon content (THC) of the off gas from Zones 1 and 2, while Figure 1-11 presents the off gas composition (i.e., hydrogen, carbon monoxide, and carbon dioxide) from Zones 1 and 2. These figures clearly indicate the gas polishing that occurs in Zone 2. While this pilot-scale reactors-in-series equipment configuration was developed from existing assets and thus was not optimized for multi-zone processing, excellent environmental performance was achieved. The transfer pipe performed very well, with <0.1% of the feed material depositing in the pipe. The reactors-in-series configuration for multi-zone processing of bulk solid feed materials has been successfully demonstrated and is scalable to commercial-scale systems required for processing DOE waste streams containing bulk solids.

Figure 1-10
Comparison of Zone 1 and Zone 2 THC Data (TDI Injection 2)

Figure 1-11
Off-gas Composition Data (TDI Injection 2)

MMT also conducted a major experimental program to determine the effect of halogens, alkali metals, sulfur, and phosphorus on the partitioning of radionuclide surrogates. MMT performed experiments to characterize the behavior of sulfur in an iron bath under Phase I and II of this contract, which determined that sulfur alloys with iron to form ferrosulfur (FeS) at concentrations up to 36 wt%. Phosphorus and sulfur behavior was predicted to be similar as both are non-metallic, third row elements with access to 3d orbitals that exist in a variety of allotropes and can have multiple valances. A progressive experimental program plan was developed to perform bench-scale experiments addressing both phosphorus partitioning in iron and the iron-phosphorus-sulfur ternary system, followed by pilot- and commercial-scale testing with and without the presence of a ceramic phase. The experimental program culminated in a commercial-scale experiment that processed approximately 1700 lb. of flyash (a low organic, heterogeneous, solid feed material) spiked with cerium oxide (CeO_2) as a radionuclide surrogate.

Bench-scale experiments determined that phosphorus will alloy with iron to form ferrophosphorus (Fe_2P) at concentrations approaching 24 wt%, with near complete capture of phosphorus up to 18 wt%. Incomplete phosphorus capture provides an indication that the bath is approaching saturation and thus can be used as the basis for a process control system. Bench-scale testing also determined that carbon does not affect the partitioning of phosphorus in iron and that no special containment systems are required to contain ferrophosphorus baths. Bench-scale experiments of the Fe-P-S ternary system showed clear phase separation of Fe_2P and FeS , confirming their immiscibility. The relative amount of each phase can be determined by monitoring off-gas composition and mass balances. Conclusions from the bench-scale studies include that phase separation of FeS and Fe_2P should be anticipated in the design of the containment system for a CEP reactor processing mixed waste containing sulfur and phosphorus. It was also concluded that Fe-P-S-C systems are both operable and controllable using gas phase sensors. Two operating strategies for processing of mixed wastes containing sulfur and phosphorus were also developed, including the impact on GHT system design associated with each proposed operating strategy.

Upon completion of the initial bench-scale experiments, the experimental program progressed into its second phase which were designed to determine the partitioning of phosphorus and sulfur with a ceramic phase present. Phosphorus and sulfur partitioning in the presence of a ceramic layer for a carbon-saturated iron bath and a copper bath with low carbon content is presented in Table 1.6 and Table 1.7, respectively.

Table 1.6
P and S Partitioning in a C-saturated Fe Bath with Ceramic Phase

Run	Element	Metal (%)	Ceramic (%)	Particulate (%)
026	P	89.7	3.7	6.6
	S	74.9	19.9	5.2
005	P	91.5	2.5	6.0
	S	32.8	64.5	2.6

Table 1.7
P and S Partitioning in Cu Bath with Ceramic Phase

Run	Element	Metal (%)	Ceramic (%)	Particulate (%)
021	P	5.5	94.5	0.0
	S	94.0	4.7	1.3
039	P	0.0	89.2	10.8
	S	92.8	1.5	5.7
043	P	0.0	99.9	0.1
	S	98.5	0.5	1.0

These data indicate that at low partial pressures of oxygen, phosphorus partitions to the metal phase while sulfur shows mixed partitioning in the presence of a ceramic phase. Increasing the partial pressure of oxygen results in phosphorus partitioning to the ceramic phase and sulfur partitioning to the metal phase.

The final experiment in this experimental campaign was performed on a commercial-scale unit using approximately 1700 lb. of flyash spiked with cerium oxide (CeO_2) as a radionuclide surrogate and lanthanum oxide (La_2O_3) as a tracer. Flyash is a low organic, heterogeneous material whose composition is considered representative of tank waste. The compositions of two samples of flyash are provided in Table 1.8.

Table 1.8
Flyash Composition Data

Element	Sample 1	Sample 2
Si	15.59	12.29
Ti	1.15	1.45
Ca	16.67	17.89
Al	4.45	5.83
Mg	1.00	1.61
Fe	1.71	2.92
O	41.33	39.67
S	1.60	3.73
P	0.44	1.03
Cl	7.73	4.07
Na	2.68	2.03
K	1.47	2.71
C	2.05	1.12
Mn	0.12	0.11
H	0.08	0.13
Zn	0.58	1.5
Pb	0.26	0.48
Ba	0.10	0.32
Cr	0.02	0.12
Ni	0.01	0.02
Cu	0.07	0.09
Cd	0.006	0.009
Hg	ND	0.0002
moisture	0.76	0.62

The feed material was processed at a rate averaging approximately 10 lb. per minute. The ceramic phase layer depth was approximately 19 inches at completion of the experiment. The final condensed phase product compositions were determined using X-ray Fluorescopy (XRF) and it was determined that 99% of both the cerium and lanthanum radionuclide surrogates partitioned to the ceramic phase. The ceramic phase products were then subjected to TCLP and PCT leaching resistance testing. All samples passed the TCLP test for RCRA metals and neither the cerium nor lanthanum leached from the samples to the analytical detection limit of 0.01 ppm.

The experimental program to assess the effect of halogens, alkali metals, sulfur, and phosphorus on radionuclide surrogate partitioning was successfully completed. The results conclusively demonstrate CEP's ability to partition radionuclide surrogates to the targeted phase in the presence of complex process chemistry.

MMT has performed an extensive series of experiments to optimize the ceramic phase for stabilization of radioactive species. While most of this work was performed at MMT's Fall River R&D facility using radioactive surrogates, some experiments have been performed at Oak Ridge, TN using actual radionuclides. Tests using actual radionuclides have successfully verified previously conducted experiments for partitioning their surrogates radionuclides to the ceramic phase. Further, experiments involving the recycle of uranium hexafluoride and West End Treatment Facility sludge were not funded under this contract; however, the results have been included in this final report as they are major DOE interest items.

Figure 1.12
Identification of Radionuclide Surrogate

Table 1.9
Physical Properties of Uranium and Surrogates

Element	Oxide		Metal	
	m.p. (°C)	Density (g/cm ³)	m.p. (°C)	Density (g/cm ³)
U	2,878	10.96	1,132	19.05
Hf	2,758	9.68	2,227	13.31
Zr	2,715	5.6	1,852	6.49
Fe	1,369	5.7	1,535	7.86
Ni	1,984	6.67	1,455	8.90

The experimental design parameters evaluated were metal system, gas environment and ceramic phase composition. Radioactive surrogate partitioning was measured using neutron activation analysis (NAA) for non-destructive trace analysis. The hafnium lower detection limit (LDL) was 0.2 ppm in iron and 2 ppm in nickel. X-ray fluorescence (XRF) was used for analysis of the vitreous phase composition. Metal samples were taken at different positions in the metal bath to ensure uniform decontamination. The contract objective was to demonstrate uniform metal decontamination above 98%. In addition, backscattered electron imaging and x-ray analysis were used to investigate the nature of the capture and stabilization of the radionuclide surrogate in the vitreous phase.

Average decontamination of the metal samples were > 99% and exceeded the contract's 98% objective in all experimental trials. The calculated decontamination factors were analytically limited with no radioactive surrogate detected in the metals. Table 1.10 summarizes the results. V1 refers to aluminosilicate vitreous compositions while V2 refers to borosilicate vitreous compositions.

Table 1.10
Radionuclide Partitioning

Metal	Vitreous Phase	Decontamination
Iron	V1	≥ 99.62%
Nickel	V1	≥ 99.76%
Iron	V2	≥ 99.08%

Samples taken at different positions in the metal bath demonstrate uniform distribution of any trace amounts of hafnium across the metal (Figure 1.13 and Figure 1.14). This indicates that radioisotope transport to and incorporation in the oxide phase is very efficient under CEP operating conditions.

Figure 1.13
Sampling across Radius of Metal

Figure 1.14
Sampling from
Top to Bottom of Metal

Ceramic samples were analyzed using backscatter electron imaging to identify the nature of the radionuclide capture and stabilization in the ceramic phase. This work indicated that operating conditions and vitreous phase composition can affect radionuclide capture and stabilization. Figure 1.15 shows the backscatter image of a vitreous sample which has separated into hafnium rich and hafnium poor regions. This is in contrast to the vitreous sample shown in Figure 1.16, where hafnium is uniformly distributed. CEP conditions have been identified to achieve this desired vitreous radionuclide capture and stabilization mechanism.

Figure 1.15
Backscatter Image:
Phase Separation Exhibited

Figure 1.16
Backscatter Image:
No Phase Separation Exhibited

MMT has successfully demonstrated the ability of CEP to partition radionuclides to the ceramic phase, thereby achieving substantial volume reduction of the radioactive waste and producing a highly stable waste form for eventual disposal. Uranium (U) and cerium (Ce) radionuclides were successfully partitioned to a ceramic phase composed of either calcium aluminosilicate or lithium boron calcium aluminosilicate. A glass synthesis experiment was also conducted using known quantities of UO_2 and CeO_2 to evaluate high levels of waste loading and incorporation of these elements into ceramics. The partitioning experiments were performed using two different ceramic phase compositions to evaluate operability, waste loading, and leach resistance. Calcium aluminosilicate was selected for its relatively low viscosity and its leach resistance. Lithium boron calcium aluminosilicate was selected to increase waste loading and decrease viscosity. Both ceramic phase compositions exhibited minimal chemical interaction with the containment system. Four types of analyses were performed on the samples obtained from these experiments: neutron activation analysis (NAA), gamma spectroscopy, a Tennelec (Oxford) $\alpha\beta$ counting system, and a frisker (Geiger counter) for gross initial measurements. Alpha, beta, and gamma activity were measured as a function of ceramic phase sample mass, which was then used to calculate activity/gram of sample and activity/gram of uranium. A comparison of the alpha, beta, and gamma activities as a function of uranium content in the crushed ceramic phase samples is presented as Figure 1.17. Analysis of the metal samples, presented in Table 1.11, demonstrate metal decontamination in excess of 99%, which is consistent with the data obtained from the ceramic phase samples. The data obtained from these experiments conclusively demonstrate CEP's ability to partition actual radionuclides to the ceramic phase, thus achieving a high degree of metal decontamination which is necessary for the recycle, reuse, and disposal of materials from DOE decontamination and decommissioning activities.

Figure 1.17
Comparison of Alpha, Beta, and Gamma Activity as a Function
of Uranium Content in Crushed Glass Samples

Table 1.11
Uranium Concentration in Metal Samples

Sample	Uranium Concentration (ppm)	Decontamination (%)
M7	0.28	99.97
M8	0.62	99.93
M9	0.69	99.93

MMT has performed a comprehensive series of experiments that have successfully verified the previously established thermodynamic feasibility of using catalytic extraction processing as an efficient and cost-effective means of converting uranium hexafluoride (UF_6) to uranium oxide (UO_x) and anhydrous hydrogen fluoride (HF). These experiments were not funded under this contract, but the results have been included in this final report as it is a major DOE interest item. Uranium hexafluoride is one of the most widely used chemical compounds of uranium. Over one billion pounds of UF_6 have been produced over the past 50 years in support of the U.S. uranium isotope enrichment enterprise. There is a growing concern regarding the long-term environmental risks associated with continued storage of large UF_6 .

inventories. DOE currently stores approximately 610,000 metric tons of UF_6 , with an additional 22,000 metric tons stored by the U.S. Enrichment Corporation (USEC). MMT has successfully demonstrated the ability of CEP to safely and reliably process UF_6 to produce a more stable uranium compound for recycle within the DOE complex or nuclear industry while producing valuable, high quality anhydrous HF which can be recycled to the commercial chemical market. In the two tests performed to date, no residual UF_6 was detected in either the process off-gas or the chemical traps to a Lower Detection Limit (LDL) of 100 ppm. Safety programs for UF_6 processing have also been demonstrated, confirming the functionality of the facility design including safe handling of the UF_6 and HF, effective chemical trapping of HF product, containment of carbon monoxide and hydrogen, environmental monitoring, and on-line instrumentation. These experiments have successfully demonstrated the applicability of catalytic extraction processing to effectively and economically recycle an important waste form that exists in a large quantity within the DOE complex.

MMT has also performed a series of experiments to optimize the ceramic phase for CEP processing of mixed-waste sludge. These experiments were also not funded under this contract, but the results have been included in this final report as it is a major DOE interest item. The sludge used in these experiments is a byproduct of industrial waste water treatment and was obtained from DOE's West End Treatment Facility (WETF) in Oak Ridge, TN. The WETF sludge contains a significant amount of water and is contaminated with residual organics, chlorine, phosphorous, RCRA metals, nickel, and radionuclides including uranium ($1.2 \times 10^3 \mu\text{Ci/g}$), technetium ($18.7 \times 10^4 \mu\text{Ci/g}$), and cesium ($1.8 \times 10^6 \mu\text{Ci/g}$). The objective of these experiments was to engineer a ceramic phase that is operable (i.e. workable viscosity and minimum containment corrosivity), functional (i.e. accommodates major ceramic-forming components and captures any hazardous and/or radioactive components in a non-leachable form), stable during long-term storage, and maximizes volume reduction. Fifteen bench-scale experiments and a 27 hour pilot-scale experiments were performed during this test program, as well as a series of potential-driven neutralization tests, recipe development tests for cement product development, and slurry injection system development. This test program has successfully developed and demonstrated an operable, functional and stable ceramic phase that achieves a significant amount of volume reduction. Experimental results included the ceramic phase product meeting all Envirocare Waste Acceptance Criteria requirements including passing the toxicity characteristic leaching procedure (TCLP), achieving a mass balance closure of $100 \pm 5\%$, demonstrating uranium capture in the ceramic phase, and meeting market specifications of targeted products for DOE on-site reuse (ceramic product development). Further, this experimental program also successfully demonstrated the versatility of CEP for processing DOE wastes including highly aqueous waste streams such as WETF sludge.

MMT has performed an extensive series of tests to optimize the ceramic phase product for stabilization of radioactive species. A primary consideration for CEP of DOE waste streams containing ceramic phase formers include the composition and physical

properties of the ceramic phase produced. An important advantage of CEP is its flexibility in producing either a glassy or crystalline ceramic phase product based on customer requirements. MMT completed a study designed to determine the effects of microstructure on the leaching characteristics of the ceramic phase produced by CEP. The difference in leaching resistance between four pairs of ceramics that have the same composition but different microstructures (i.e., glassy or crystalline) was examined for four pairs spanning the operable range of the $\text{CaO-Al}_2\text{O}_3\text{-SiO}_2$ (C-A-S) ternary phase diagram for CEP.

Three different leaching resistance tests were performed on the ceramic samples: MMT's in-house Leaching and Dissolution Resistance (LADR) test, the Toxicity Characteristic Leaching Procedure (TCLP), and the Product Consistency Test (PCT). The Toxicity Characteristic Leaching Procedure (TCLP) test is used by the U.S. Environmental Protection Agency to classify materials as toxic or non-toxic with respect to disposal. The TCLP test measures the concentrations of RCRA metals (As, Ba, Cd, Cr, Pb, Hg, Se, and Ag) in the leachate solution. The concentrations of Ca, Al, Si, and Ba are shown in Table 1.25. The other 7 RCRA metals were non-detect in all 8 samples, including the radionuclide surrogates cerium and lanthanum contained in samples 7 and 8.

Figure 1-18
TCLP Test Results

Sample	Type	Concentration in Leachate (mg/L)			
		Ca	Al	Si	Ba
1	Glass	8.2	ND	ND	ND
2	Crystal	150	1.4	11	ND
3	Glass	660	ND	34	ND
4	Crystal	970	ND	40	ND
5	Glass	41	42	35	ND
6	Crystal	66	17	13	ND
7	Glass	81	7.3	22	ND
8	Crystal	690	4.0	96	34

The 34 mg/l of barium present in the leachate of sample 8 is well within the regulatory limit of 100 mg/l. Overall, the eight samples tested would all be classified as non-toxic by the EPA based on these results. While all ceramic phase product samples complied with established regulatory concentration limits, the leaching resistance of glassy samples was determined to superior to that of the corresponding crystalline sample. The data obtained from this experimental program will be used to determine the quench rate for tapped ceramics in selected compositional ranges to meet customer and regulatory requirements for leachability.

1.3.6. Task 1.5 - Experimental Testing of Resource Conservation and Recovery Act (RCRA) Wastes

The investigation of CEP's conversion and immobilization capabilities for RCRA components of contaminated scrap metal feeds has been well proven during the experimental campaign of this contract. MMT brought to the contract a solid foundation of understanding of the behavior of toxic and halogenated feeds in CEP, and proceeded to optimize the chemistry of the system for the feeds anticipated. Efforts began on bench-scale apparatus, and subsequent prototype trials validated bench-scale results.

Table 1.12 demonstrates CEP's environmental and waste minimization performance for a wide range of heterogeneous wastes, encompassing practically every chemical and physical form. These diverse waste forms span the chemical compositions and physical forms which are encountered throughout the DOE complex. A recent benchmarking procedure developed by DOE for evaluating mixed waste treatment technologies identified naphthalene, dichlorobenzene, and monochlorobenzene as being representative of DOE wastes. CEP environmental performance on these species, surpassing current EPA standards, is presented herein. This same benchmarking procedure identified Cr, Ni, Pb, and Cd as RCRA constituents in DOE wastes. These metals have been successfully recovered as commercial products as part of the surplus metal componentry recycling and biosludge processing. DOE-recommended and MMT identified radioisotope surrogates (Hf, Zr) have been successfully partitioned to a stable, non-leachable phase, also as part of the surplus metal componentry recycling

CEP has robustly recycled a variable and chemically diverse feed slate as represented by the feeds in Table 1.12. High molecular weight aromatics were converted to synthesis gas with a conversion efficiency > 99.9999% with no products of incomplete conversion detected (chemically analogous to benzene and naphthalene, DOE-recommended surrogates). Complex chlorinated organics including aromatics, aliphatics, plastics (PVC), and heavily-chlorinated RCRA listed wastes K019/20 and F024 have been successfully converted to products such as synthesis gas or condensed phase chlorinated products (chemically analogous to PCBs). Trace components such as dioxins and difurans were not detected to the targeted regulatory limit of 0.1 ng/Nm³ (TEQ 2,3,7,8-TCDD) as shown in Table 1.13. Organically-bound nitrogen and highly toxic isocyanates (RCRA listed waste K027) were synthesized into nitrogen (N₂), hydrogen (H₂) and carbon monoxide (CO) with no detection of NO_x formation to 1 ppm. Highly heterogeneous wastes including surplus metal/weapon componentry (chemically and physically analogous to decommissioning materials) and biosludge

Table 1.12
Major Feeds Processed at the Demonstration Prototype

			Waste Minimization Performance			Environmental Performance		
Feed	Key Elements	Chemical Structure	Product Recovery	% of feed Recycled	Residual	DRE	TCLP	Trace
Representative Surrogate Feeds								
Polystyrene/graphite	C, H	High molecular weight aromatic	Syngas	99% to syngas	Dust Negligible	≥ 99.9999%	N/A	PICs < LDL ¹ of 1 ppm CO ₂ < 1% NO, SO ₂ < LDL of 100 ppm
Chlorotoluene/heavy organics	C, H, Cl	Halogenated aromatic	Syngas Ceramic	87% to syngas 12% to ceramic	Dust Negligible	≥ 99.9999%	Passes TCLP	PICs < LDL of 1 ppm CO ₂ < 1% NO, SO ₂ < LDL of 100 ppm
Dimethyl Acetamide/heavy organics	C, H, N	Organically-bound nitrogen	Syngas Nitrogen	96% to syngas 3% to nitrogen	Dust Negligible	≥ 99.9999% (based on THC)	N/A	PICs < LDL of 1 ppm CO ₂ < 1% NO, SO ₂ < LDL of 100 ppm ² NO _x , SO _x < LDL of 1 ppm ³
Representative Hazardous Waste Feeds								
Industrial Biosludge waste	C, H, N, S, Ash (P, Na, Ca, Mg)	Highly variable heterogeneous organic and inorganic sludge	Syngas Nitrogen Ceramic Ferroalloy	70% to syngas 8% to nitrogen 20% to ceramic 1% to ferroalloy	Dust Negligible	≥ 99.9999% (based on THC)	Passes TCLP	PICs < LDL of 1 ppm CO ₂ < 1% NO, SO ₂ < LDL of 100 ppm
Surplus Metal/Weapon Componentry	80% of Non-radioactive elements	Precious, volatile (Pb, Zn), and reducible metals (Cr, Ni), plastics, exothermic inorg. uncharacterized solids	Syngas Ceramic Ferroalloy	25% to syngas 8% to ceramic 63% to ferroalloy	Dust Negligible	N/A	Passes TCLP	PICs < LDL of 1 ppm CO ₂ < 1% NO, SO ₂ < LDL of 100 ppm
K019/K020 ⁴ /chlorobenzene/Fuel oil	C, H, Cl	Residuals from PVC mfr; unsat'd Cl-organics	Syngas	76% to syngas 23% to ceramic	Dust Negligible	≥ 99.9999%	Passes TCLP	PICs < LDL of 1 ppm CO ₂ < 1% NO, SO ₂ < LDL of 100 ppm
K027 ⁵	C, H, N	Isocyanates, highly toxic	Syngas Ferroalloy (Fe - Ni)	93% to syngas 5% to nitrogen <1% to ceramic <1% to ferroalloy	Dust Negligible	≥ 99.9999%	Passes TCLP	PICs < LDL of 1 ppm CO ₂ < 1% NO, SO ₂ < LDL of 100 ppm
F024 ⁶ /Fuel oil/chlorotoluene	C, H, Cl	Halogenated aliphatics	Syngas HCl gas	82% to syngas 13% to HCl gas <1% to ceramic	Dust Negligible	≥ 99.9999%	Passes TCLP	PICs < LDL of 1 ppm CO ₂ < 1% NO, SO _x < LDL of 3 ppm and 1 ppm respectively

¹ LDL = Lower Detection Limit.
² As measured by on-line mass spec.
³ As measured by third party analytical equipment placed on-line.

⁴ EPA listed hazardous waste stream: ethylene dichloride/vinyl chloride heavy ends.
⁵ EPA listed hazardous waste stream: toluene diisocyanate distillation residues.
⁶ EPA listed hazardous waste stream: chlorinated aliphatic hydrocarbons.

Table 1.13
Trace Component Off-Gas Concentrations during CEP of Chlorinated Wastes

Description:		Aromatics - 20% PVC	Biosludges - 2% PVC	Biosludges - 2% PVC	F024	F024
Time	Initial:	15:10	18:34	10:02	0:54	2:50
	Final:	15:40	19:12	11:02	2:18	4:03
Gas Meter Volume (ft ³)	Initial:	619.67	296.70	303.59	319.26	339.35
	Final:	639.67	303.59	313.61	339.35	360.65
Gas Meter Outlet (°F)	Initial:	66	70	70	70	70
	Final:	67	70	70	70	70
Total Sample Volume	SCF:	20.13	6.89	10.02	20.09	21.30
	Nm ³ :	0.53	0.18	0.26	0.53	0.56
Analytes (ng)						
Total TCDD:		ND	ND	ND	ND	ND
Total PeCDD:		ND	ND	ND	ND	ND
Total HxCDD:		ND	ND	ND	ND	ND
Total HpCDD:		ND	ND	ND	ND	ND
Total OCDD:		ND	ND	ND	ND	ND
Total TCDF:		ND	ND	ND	ND	ND
Total PeCDF:		ND	ND	ND	ND	ND
Total HxCDF:		ND	ND	ND	ND	ND
Total HpCDF:		ND	ND	ND	ND	ND
Total OCDF:		ND	ND	ND	ND	ND
2,3,7,8-TCDD toxicity equivalents (ng/Nm ³)						
EPA-1989		ND	ND	ND	ND	ND
NATO		ND	ND	ND	ND	ND
Nordic		ND	ND	ND	ND	ND
UBA		ND	ND	ND	ND	ND
BUS		ND	ND	ND	ND	ND

ND = Not Detected to targetted regulatory standard of 0.1 ng/Nm³.

wastes (chemically and physically analagous to DOE sludges) from wastewater treatment (both potentially RCRA characteristic wastes when discarded), which contained from 0 to > 60% metals, were successfully recycled into commercially valuable products as confirmed by MADEP (Massachusetts Department of Environmental Protection) Recycling Certifications. In all cases environmental standards were met and surpassed. Table 1.14 shows that all ceramic material generated in CEP passed TCLP forming a safe, non-leachable potential final form for radionuclides.

Table 1.14
CEP Ceramic Products Form Non-Leachable Matrices

Waste Processed	TCLP Metals Regulated Limits (mg/l)							
	As 5.0	Ba 100	Cd 1.0	Cr 5.0	Pb 5.0	Hg 0.2	Se 1.0	Ag 5.0
K019/K020 ¹	ND	ND	ND	0.3 ²	ND	ND	ND	ND
PVC and Polystyrene	ND	ND	ND	0.3 ²	ND	ND	ND	ND
Spent Metal Componentry with Cl-plastics ¹	ND	ND	ND	0.5 ²	ND	ND	ND	ND
PVC and Polystyrene ³	NA	NA	ND	ND	ND	NA	NA	NA
Biosludge with 1% Cl	ND	ND	ND	ND	ND	ND	ND	ND
F024	ND	ND	ND	ND	ND	ND	ND	ND
ND = Not Detected; NA = Not Applicable ¹ The full TCLP was run for these samples. All other regulated species were ND. ² Cr ₂ O ₃ -Al ₂ O ₃ refractory bricks were present during these runs which provided a source of Cr ₂ O ₃ . Total Cr has been measured and no hexavalent Cr ⁺⁶ was detected. The refractory has been optimized to eliminate Cr. ³ A partial TCLP test was performed for Cd, Cr and Pb								

1.3.6.1. Mass Balance Closure on Materials Representative of DOE Feeds

MMT has successfully processed and recovered products from multiple materials of direct interest to DOE. Materials such as surplus metal componentry, biosludge, and chlorinated RCRA wastes are analogous to metal, sludge, and organic wastes within the DOE complex. Of particular interest to DOE are radionuclides such as uranium, plutonium and organic contamination originating from sources such as lubricating oils, cutting fluids, solvents, and PCBs. All material which may potentially contaminate scrap metal or be contained in the debris are candidates for processing by CEP. Mass Balance results for a variety of feeds processed via CEP are shown in Table 1.15.

Table 1.15
Demonstration-Scale Mass Balances for RCRA and RCRA-like Feeds
Analagous to DOE Wastes

Components (lbs.)	F024 RCRA Chlorinated Waste	Biosludge Waste	K019/20 RCRA Chlorinated Waste	Surplus Metal Componentry
Waste Feed ¹	666	4684.00	238	496.19
Methane ²	32	175.58	13.54	10.71
Oxygen ²	668.13	2022.92	275.70	205.74
Carbon Added to Bath		380.00	70	110
Iron Melt ³	3510	3180.00	3838	3838
Flux Additions	65	218.00	438.4	176.87
Ceramic in Bath	800	NA		
Total Input	5741.13	10660.50	4873.64	4837.51
Hydrogen ⁴	73.27	379.02	28.36	15.71
Carbon Monoxide ⁴	1097.69	5846.31	561.13	386.38
Carbon Dioxide ⁴	29.47	936.58	9.90	30.59
Hydrogen Chloride ⁴	84.88		16.06	
Ferro alloy Product ⁵	3505	3225.00	3829.29	4200
Ceramic Product ⁶	905	410.00	450.70	312
Total Output	5695.31	10256.92	4895.44	4946.61
Closure	99.20%	96.2%	100.45	102%
¹ Feed composition based on third party elemental feed analysis; total amount of feed injected was calculated based on change in feed tank weight data installed in the 20-second data log program. ² Co-feeds measured by on-line flowmeters (20-second datalog program) ³ Amount of metal added was recorded in the operator book. Carbon concentration in metal was determined by on-site LECO analysis. Pre-injection metal analysis (third party analysis) were used in some cases. ⁴ Argon was used as tracer gas to determine the total off-gas flow. Individual product gas flow was determined by mass spectrometer results (20-second datalog program) ⁵ Usually a tracer like nickel was used to determine the total weight of metal in bath. The calculation was confirmed by the tapped metal weight. Post-injection metal analysis determined the composition. ⁶ Usually a tracer like hafnium was used to determine the total ceramic phase amount. Post injection ceramic analysis determined the composition.				

1.3.6.2. Material Recovery

Following the dissolution process characteristic of CEP, the addition of co-feeds and engineering of process variables results in targeted product formation. Very high

conversion of waste feeds has been demonstrated. Demonstrations verify that all organic materials in CEP feed materials are converted to synthesis gas; non-reducible metals such as aluminum, calcium, silicon, etc. are recovered as a ceramic product; reducible non-volatile metals such as copper, iron, cobalt, chromium, vanadium, etc. are recovered as metal product; volatile reducible metals such as zinc and lead are captured and collected in the gas handling train (GHT). The potential of each of these product phases to meet specifications for on-site use as raw materials or commercial market usage has been demonstrated. MADEP has certified CEP as recycling for heterogeneous organic, metallic, and organometallic wastes. The following tables summarize the recovery results in various product phases for CEP.

Table 1.16
Experimental Recovery for Synthesis Gas

	Synthesis Gas Formers (wt% feed)			% Feed	% Recycled	% Recovered
	C	H ¹	O			
RCRA Waste F024	72.50	9.46	0.1	82.06	80.84	98.51
RCRA Waste K019/020	68.1	8.07	0.9	77.07	76.3	99.0
Surplus Metal	32.09	3.46	23.37	58.86	57.46	97.62
Surplus Metal Surrogate	19.11	1.76	4.22	25.09	23.76	94.691
Biosludge (1)	45.43	6.57	26.98	78.98	73.57	93.151
Biosludge (2)	45.43	6.57	26.98	78.98	74.29	94.921
Note: The % Feed represents the potential amount of feed that could form the product. The % Recycled represents the amount of the elements present in the actual product formed based on treatability study results. The % Recovered is the ratio of % Recycled to % Feed which shows the efficiency of the CEP for waste recycling. ¹ Attributable in part to feed variability						

Table 1.17
Experimental Recovery for Ceramic Product

	Feed Composition Elemental wt%												% Feed	% Recycled	% Recovered
	Al	Ca	Si	Mg	Ba	O	Cl	S	P	Na	K	Ti			
Biosludge(1)	0.05	0.81	0.35	0.20	-	4.44	0.06	0.36	0.17	1.76	0.48	-	8.69	8.69	100
Biosludge(2)	0.05	0.81	0.35	0.20	-	4.44	0.06	0.62	0.01	1.76	0.24	-	8.54	8.54	100
Actual SMC	8.69	-	7.48	0.23	0.53	-	0.68	0.54	-	-	-	0.70	18.5	18.5	100
Component SMC	7.61	-	-	-	-	-	-	-	-	-	-	-	7.61	7.61	100

Note: The % Feed represents the potential amount of feed that could form the product. The % Recycled represents the amount of the elements present in the actual product formed based on treatability study results. The % Recovered is the ratio of % Recycled to % Feed which shows the efficiency of the CEP in actual recovery performances.

Table 1.18
Experimental Recovery for Metal

	Feed Composition Elemental wt%								% Feed	% Recycled	% Recovered
	Fe	Cu	Ni	Cr	Co	Mn	Ag	Mo			
Actual SMC	6.97	5.16	1.93	0.61	0.4	0.18	0.15	0.1	15.5	15.5	100
Component SMC	22.53	37.14	1.68	1.97	-	-	0.61	-	64	64	100

Note: The % Feed represents the potential amount of feed that could form the product. The % Recycled represents the amount of the elements present in the actual product formed based on treatability study results. The % Recovered is the ratio of % Recycled to % Feed which shows the efficiency of the CEP in actual recovery performances.

1.3.6.3. Environmental Performance and Regulatory Standards

1.3.6.3.1. Destruction and Removal Efficiency (DRE) Results

Destruction Removal Efficiency (DRE) has been the standard method in the regulatory environment to determine the effectiveness of waste treatment. The amount of principle organic hazardous constituent (POHC) before and after treatment is compared using the formula:

$$(In - Out) / In * 100\%$$

where In = Amount of hazardous constituent in the waste feed

Out = Amount of the same hazardous constituent in the off-gas

In CEP, feed constituent composition is determined by third party analytical laboratory results. SUMMA canisters are filled with off-gas samples at the baghouse outlet during the injection period. Consistently, the hazardous waste materials processed in the feed were not detected in the off-gas down to the lowest detection limits in the range of ppb, resulting in DREs of $\geq 99.9999\%$ far surpassing the current regulated standard 99.99%.

Table 1.19 shows the results of a chlorinated organic material (PVC/Polystyrene mixture) processed via CEP. Both polyvinyl chloride (PVC) and polystyrene are polymeric molecules that did not volatilize and therefore, would not be detected in the off-gas. An alternative approach for calculating DRE values was based on the assumption that PVC could be modeled as vinyl chloride (CH_2CHCl) or as chloroethane (CH_3CHCl), which are very similar structurally to the PVC monomer ($-[CH_2CHCl]-$). Similarly, polystyrene ($-[CH_2-CH(C_6H_5)]-$) can be modeled as styrene ($CH_2=CH(C_6H_5)$) or as ethyl benzene ($CH_3CH_2(C_6H_5)$). The modeled compounds were analyzed by EPA Method TO-14. The model compounds were not detected in the off-gas, resulting in DREs of $\geq 99.9999\%$.

Table 1.19
DREs for PVC and Polystyrene

Sample No	Sampling Time	Tracer	Compound	DRE
DRE-16-BO	1:49-2:33	Vinyl chloride	PVC	≥99.99998%
DRE-16-BO	1:49-2:33	Chloroethane	PVC	≥99.99998%
DRE-17-BO	4:14-4:50	Vinyl chloride	PVC	≥99.99998%
DRE-17-BO	4:14-4:50	Chloroethane	PVC	≥99.99998%
DRE-16-BO	1:49-2:33	Styrene	Polystyrene	≥99.99994%
DRE-16-BO	1:49-2:33	Ethyl benzene	Polystyrene	≥99.99993%
DRE-17-BO	4:14-4:50	Styrene	Polystyrene	≥99.99994%
DRE-17-BO	4:14-4:50	Ethyl benzene	Polystyrene	≥99.99994%
Trace molecules not detected in the CEP product gas. DREs limited by the lower detection limits				

Another example of effective DRE performance was demonstrated in F024 processing. The POHC of the feed (defined by EPA methods), chlorobenzene; and the major hazardous organic (the high concentration in the feed), trichlorobenzene was used to perform DRE calculations. Both of the constituents were not detected to the lowest detection limit in the range of ppb, resulting a DRE of ≥99.9999%. A summary of the F024 test results are presented in Table 1.20 through Table 1.23.

Table 1.20
DREs for Chlorobenzene and Trichloroethene

Sample No.	Sampling Time	DRE for Chlorobenzene (POHC)	DRE for Trichloroethene
1T-94-009-BO-12	0:39	≥99.9999	≥99.9999
1T-94-009-BO-13	1:39	≥99.9999	≥99.9999
1T-94-009-BO-14	1:39	≥99.9999	≥99.9999
1T-94-009-BO-15	2:42	≥99.9999	≥99.9999
1T-94-009-BO-16	2:42	≥99.9999	≥99.9999
1T-94-009-BO-17	3:50	≥99.9999	≥99.9999
1T-94-009-BO-18	3:50	≥99.9999	≥99.9999
Note: Chlorobenzene and trichloroethene were non-detectable in the off-gas. DRE's were limited by the lower detection limit (LDL)			

Table 1.21
Off-Gas Performance of F024 Processing
(Based on TO-14 Data)

Constituents	Feed Composition (mg/l)	CEP Product-gas (ppm)
1,1,2-Trichloroethane	18,000	ND (0.00037)
Chlorobenzene	12,000	ND (0.00037)
1,2-Dichloroethane	6,800	ND (0.00044)
1,1,2,2-Tetrachloroethane	11,000	ND (0.00029)
Trichloroethene	24,000	ND (0.00038)
ND = Not Detected (lowest detection limit) Off-gas regulations are based on the performance of thermal oxidizers; regulations not available for specific organic constituents.		

Table 1.22
CEP Performance under Non Wastewater Regulations
(Based on F024 Processing)

Constituents	Feed Composition (mg/l)	CEP Ceramic Product (mg/kg)	CEP Metal Product (mg/kg)	EPA Non Wastewater Regulation (mg/kg)
1,1,2-Trichloroethane	18,000	ND (0.0075)	ND (0.0075)	6.0
Tetrachloroethene	9,700	ND (0.0075)	ND (0.0075)	6.0
Chlorobenzene	12,000	ND (0.018)	ND (0.018)	6.0
1,2-Dichloroethane	6,800	ND (0.0075)	ND (0.0075)	6.0
1,1,2,2-Tetrachloroethane	11,000	ND (0.005)	ND (0.005)	6.0
Trichloroethene	24,000	ND (0.005)	ND (0.005)	6.0
Xylenes	610	ND (0.005)	ND (0.005)	3.0
1,1,1,2-Tetrachloroethane	1,000	ND (0.025)	ND (0.025)	6.0
Hexachlorobutadiene	3,600	ND (0.025)	ND (0.025)	5.6
Naphthalene	900	ND (0.025)	ND (0.025)	5.6
ND=Not Detected (Detection limit)				

Table 1.23
CEP Performance in Wastewater Regulations
(Based on F024 Processing)

Constituents	Feed Composition (mg/l)	Scrubber Water (mg/l)	EPA Wastewater Regulation (mg/l)
1,1,2-Trichloroethane	18,000	ND (0.0015)	0.054
Tetrachloroethene	9,700	ND (0.0015)	0.056
Chlorobenzene	12,000	ND (0.0035)	0.057
1,2-Dichloroethane	6,800	ND (0.0015)	0.21
1,1,2,2-Tetrachloroethane	11,000	ND (0.001)	0.057
Trichloroethene	24,000	ND (0.001)	0.054
Xylenes	610	ND (0.001)	0.32
1,1,1,2-Tetrachloroethane	1,000	ND (0.001)	0.057
Hexachlorobutadiene	3,600	ND (0.001)	0.055
Naphthalene	900	ND (0.001)	0.059
ND=Not Detected (Detection limit)			

1.3.6.3.2. Best Demonstrated Available Technology (EPA) Standards

One of the main goals at MMT is to provide an environmental solution to waste treatment by converting hazardous materials to uniform and consistent products of high quality in a safe, efficient, and effective manner. Currently the effectiveness of waste treatment is regulated by the concentration of hazardous materials in the after-process waste water and non-waste water residuals. All processes have to ensure that organic constituent concentrations in individual waste water and non wastewater streams are lower than the regulated limits set by EPA. CEP demonstrations surpass the current limits even when comparing with the Best Demonstrated Available Technology (BDAT) for various classes of waste streams.

Consistent with this aim, CEP was recently approved by EPA on 3/12/96 as a non-combustion Best Demonstrated Available Technology equivalent for all wastes for which incineration was previously the only approved processing method. EPA's determination reinforces that CEP is fundamentally different from incineration.

1.3.6.4. Toxic Characteristic Leaching Procedure Results

TCLP (Toxicity Characteristics Leaching Procedure) has been performed on the CEP ceramic material. All the ceramic samples from CEP runs were subjected to TCLP tests for RCRA metals and all of them passed the tests. Full TCLP analysis including testing for Acid/Base/Neutral Extractables, Pesticides/Herbicides in addition to RCRA metals were carried out on selected feeds. All samples passed TCLP as shown in Table 1.24.

Table 1.24
Full TCLP Results for Major Feeds at CEP

Parameter	Surplus Metal	K027	Biosludge	K019/20
RCRA Metals (mg/l)				
Arsenic	ND	ND	ND	ND
Barium	ND	ND	ND	ND
Cadmium	ND	ND	ND	ND
Chromium	ND	ND	ND	0.3 ¹
Lead	ND	ND	ND	ND
Mercury	ND	ND	ND	ND
Selenium	ND	ND	ND	ND
Silver	ND	ND	ND	ND
Acid/Base/Neutral Extractables (mg/l)				
Total Cresol	ND	ND	ND	ND
2,4-Dinitrotoluene	ND	ND	ND	ND
Hexachlorobenzene	ND	ND	ND	ND
Hexachloro-1,3-butadiene	ND	ND	ND	ND
Hexachlorobenzene	ND	ND	ND	ND
Nitrobenzene	ND	ND	ND	ND
Pentachlorophenol	ND	ND	ND	ND
2,4,5-Trichlorophenol	ND	ND	ND	ND
2,4,6-Trichlorophenol	ND	ND	ND	ND
Pyridine	ND	ND	ND	ND
Pesticides/Herbicides (mg/l)				
Chlorodane	ND	ND	ND	ND
Endrin	ND	ND	ND	ND
Heptachlor	ND	ND	ND	ND
Heptachlor epoxide	ND	ND	ND	ND
Lindane	ND	ND	ND	ND
Methoxychlor	ND	ND	ND	ND
Taxaphene	ND	ND	ND	ND
2,4-D	ND	ND	ND	ND
2,4,5-TP	ND	ND	ND	ND
ND=Not Detected ¹ Cr ₂ O ₃ -Al ₂ O ₃ refractory bricks were present during these runs which provides a source of Cr. Total Cr was measured in the ceramic phase. Cr ⁶⁺ is not formed under CEP operating conditions. Cr within the CEP system has been analyzed and no hexavalent Cr ⁶⁺ has been detected. The detected concentration was a full order of magnitude below the regulatory limit 5 mg/l.				

1.3.6.5. Operability and Reliability for Commercial Applications

Extensive demonstration runs at commercially applicable processing rates (up to 15,000 tons per year) have been successfully performed verifying the long-term operability of the CEP system with respect to on-stream factor, steady-state operation, product quality and consistency, and reliability. MMT has applied over 40 operational performance criteria for verification of efficient and effective processing. Steady-state operation have consistently been demonstrated. On-stream factors > 90% have been achieved (Figure 1.19).

Figure 1.20 and Figure 1.21 demonstrate representative steady-state operation during a chlorinated waste (F024) recycling campaign.

Figure 1.19
Biosludge Injection

Figure 1.20
F024 Heavily-Chlorinated Liquid Feed Rate

Figure 1.21
F024 Heavily-Chlorinated Liquid Feed - Off-Gas Composition

1.3.7. Task 1.6 - Conceptual Design Development for a CEP Processing Facility

MMT has developed a conceptual design of a CEP system in which organic sludges, inorganic sludges, scrap metals, soils, construction debris, and organic debris could be processed through the reactor at feed rates compatible with the natures of the various

waste compositions, and in such a manner to provide valuable information that will be required for the subsequent scale-up to a full-scale CEP system. This conceptual design integrates the myriad of data collected and processed during performance of this contract with the experience and lessons learned obtained from designing, constructing, and operating the two commercial CEP facilities currently in commission.

The CEP system components are arranged into three general areas: feed handling, reactor systems, and the gas handling train. All systems or components used in these areas reflect transportability requirements by incorporating multiple features into modules or skids.

The feed handling area is comprised of equipment required to off-load and handle incoming containers, safely remove their contents, and condition the feed for delivery into the reactor. Each step in the feed handling process contains design features to isolate and protect the operating staff in accordance with MMT standards which meet or exceed all applicable health and safety requirements.

The reactor systems design is based on operating reactor units and incorporates the latest innovations and improvements. The reactor will contain approximately 1,100 lb (as determined during the design phase) of molten iron and a proportional amount of ceramic in the horizontal section. The reactor is equipped with a 600 kW induction coil power supply and additional heat capacity required to preheat the vessel and to supplement heating the horizontal section during product removal operations, lance operations, or to deskull the reactor's interior surfaces.

The capability to sample the metal and ceramic phases, observe key parts of the bath during reactor operation, and to monitor all aspects of the reactor's performance are included in the conceptual design. Upon shutdown, an automatic injection of purge gases will occur to ensure that safe conditions are maintained throughout the system. Ancillary systems for the reactor include a means for discharging the bath contents into a closed container and a system for discharging accumulated ceramic product during extended operations. All reactor components are based on modular concepts to facilitate future alterations that may be required for feed streams other than those currently identified.

The gas handling train (GHT) is capable of safely handling a broad spectrum of gas compositions generated from the incoming feeds. The GHT has the capability to filter or scrub carryover particulate, safely collect potentially reactive dusts and volatile heavy metals, and condition gases for potential reuse. All off-gas passes through a series of absorbents and filters to ensure that no radioactive or hazardous component is discharged into downstream systems. The entire process is fully instrumented and monitored to ensure safe operation for employees and the community.

CEP typically generates hydrogen and carbon monoxide product gases during operations. MMT has combined its numerous years of experience with handling these

gases with practices established by the Government and other industries to develop a safe GHT design. GHT components are designed to minimize the generation of secondary wastes and include the option to recycle most of the secondary waste streams generated through CEP.

Table 1.25 outlines the expected staging of waste streams with their respective functional requirements. This table was used to establish the boundaries for the conceptual design.

The following list of priorities were used while performing tradeoff studies to establish this conceptual design based on the boundaries established in Table 1.25:

1. Safety
2. Environmental Integrity
3. Availability (Reliability)
4. Maintainability
5. Schedule
6. Operating Cost
7. Capital Cost

The conceptual design groups equipment in a logical manner into a module, skid, or prefabricated unit that can be easily installed in the field. The building layout features radiological controlled-access and uncontrolled-access work and support areas for highly effective management of surface/airborne contamination and radiation sources. Confinement systems, including primary and secondary confinement barriers and associated ventilation systems, provide a controlled airflow pattern from the environment into the confinement building, and then from the non-contaminated areas to the potentially contaminated areas to the normally contaminated areas of the building. Figure 1.22 presents a high level process flow diagram of the conceptual design.

Table 1.25
System Functionality by Waste Stream

Waste Stream	Functionality
Soil	<ul style="list-style-type: none"> • Screening/sizing of clay material with organic and radionuclide contamination • Blending of soil with co-feeds • Tuyere feeding at average 125 lbs soil per hour¹ • Destruction of PCBs/organics • Non-continuous ceramic phase removal during run • VHM capture in GHT • Capture of radioactive and reactive particulates in GHT • Safe containment of radionuclides, VHM, H₂/CO • Demonstrate potential for reuse of syngas² • Process instrumentation and control • Analytical support equipment
Sediment	<p>In addition to above:</p> <ul style="list-style-type: none"> • Screening/sizing of sandy materials with high mercury and radionuclide contamination • Significant VHM capture
Inorganic Sludge	<p>In addition to above:</p> <ul style="list-style-type: none"> • Removal of excess water in presence of organics and mercury • Processing of wastes with high water content (>20 wt%)
Organic Sludge	<p>In addition to above:</p> <ul style="list-style-type: none"> • Filtering of organic sludges with high solids content • Chlorine capture in ceramic phase • Adequate refractory lifetime for chloride wastes
Scrap Metal Processing Equipment	<p>In addition to above:</p> <ul style="list-style-type: none"> • Bulk feeding of metal pieces up to 6-8 inches • Non-continuous bulk feeding of metal pieces • Non-continuous metal tapping during run • Sufficient residence time to flux minor radionuclide contamination • Sufficient power to reactor to process 50 lbs/hr
Construction debris	<p>In addition to above:</p> <ul style="list-style-type: none"> • Sizing/sorting sufficient for bulk feeding (2-4 inches) • Sufficient residence time for formation of ceramic phase with desired properties
Spent Activated Carbon	<ul style="list-style-type: none"> • Tuyere feeding of dusty solids with high gas generation • Feeding of bulk solids with high gas evolution rates
Personal Protective Equipment	<ul style="list-style-type: none"> • Shredding/sizing of clothing, plastics, etc.

Figure 1.22
CEP Facility Conceptual Design Process Flow Diagram

1.3.8. Economic Analysis

The experimental test program executed under this contract has convincingly demonstrated the ability of Catalytic Extraction Processing (CEP) to recycle radioactive contaminated ferrous and non-ferrous scrap metals which can be re-used by DOE or potentially re-introduced into commerce (assuming *de minimus* limits are established by the U.S. Environmental Protection Agency and Nuclear Regulatory Commission), to partition surrogate radionuclides to the desired product phase and immobilize them, to destroy hazardous organics, and to recover volatile heavy metals. CEP has been demonstrated to be a robust process that is relatively insensitive to wide variations in waste composition and is applicable to a broad spectrum of DOE mixed low level wastes (MLLW). The U.S. Environmental Protection Agency has determined that CEP is a non-incineration process that provides equivalent treatment for all wastes for which incineration had been mandated Best Demonstrated Available Technology (BDAT). The relatively broad waste acceptance criteria of CEP offers DOE the opportunity to reduce the significant costs associated with waste characterization prior to treatment and the capital costs associated with construction of multiple, limited applicability waste processing facilities. Further, its volume reduction capability offers DOE the opportunity to reduce disposal costs. Extension of CEP's current commercial MLLW capability to include processing of high level wastes (HLW) will potentially provide a cost-effective approach for processing DOE's HLW, particularly the 126,000 ft³ of calcined HLW currently in storage at INEL.

At DOE's direction, MMT has performed a cost-benefit analysis on a commercial-scale Quantum-CEP[®] facility for processing 25,000 tons per year of a generic, heterogeneous, bulk solid waste stream consistent with DOE's decontamination and decommissioning activities, which is discussed in Section 1.3.8.3.1. CEP was determined to be a cost-effective method for processing DOE MLLW, which is consistent with the independent findings of DOE's Integrated Thermal Treatment System (ITTS) Study Phase 2 Results published in February 1996. A summary of the Planning Life-Cycle Cost Estimates and Sensitivity Analysis contained in the ITTS Study is presented in Section 1.3.8.3.2.

1.3.8.1. CEP Applicability to DOE Mixed Low Level Waste Inventory

The U.S. Department of Energy stores 167,000 cubic meters of mixed low-level (MLLW) and mixed transuranic waste (MTRU) from over 1,400 mixed radioactive and hazardous waste streams at 38 sites located in 19 states.¹ As estimated by the Site Treatment Plans (STPs), there are approximately 115,400 m³ of MLLW and 51,600 m³ of MTRU in DOE's inventory awaiting treatment and disposal.² This inventory, however, will be increased with newly generated mixed waste resulting from DOE's ongoing

¹ Mixed Waste Characterization, Treatment and Disposal Focus Area Technology Summary, Office of Science and Technology, U.S. Department of Energy, August, 1996, p.5.

² Ibid, p. 12-15.

processes, environmental restoration, facility decontamination, and facility transition activities.

The Mixed Waste Focus Area (MWFA) has created five technical product lines based on waste types: 1) Wastewaters and Slurries, 2) Combustible Organics, 3) Sludges and Soils, 4) Solids, Debris, and Soils, and 5) Unique Wastes. Table 1.26 summarizes the MWFA product lines including their respective percentages of the total mixed waste inventory and a general description of each waste stream.

Table 1.26
Mixed Waste Product Line Summary

Product Line	% of Mixed Waste Inventory	General Description
Wastewaters and Slurries	3.2	Waste streams with < 1% total organic carbon (TOC) and total suspended solids (TSS) < 30%.
Combustible Organics	1.8	Waste streams including liquids and slurries containing > 1% TOC and solids with a primarily organic weight structure such that a maximum of 20 wt% would remain as residue following incineration. Solids are defined, including sludges, as having a TSS > 30%.
Sludges and Soils	46.8	Waste streams with inorganic sludges (including water) > 50 vol%. Sludges are defined as having a TSS > 30%. A sludge may be a mixture with a stabilization agent that has properly solidified, or a mixture with absorbent materials. This category also includes inorganic particulate, paint wastes, and salt washes.
Solids, Debris, and Soils	44.5	Waste streams that contain at least 50 vol% materials that meet EPA land disposal restrictions (LDR) criteria for classification as debris (intended disposal particle size > 60 mm). This category also includes waste that is estimated to be 50 vol% soil, including sand, silt, rock, or gravel, which does not meet LDR criteria.
Unique Wastes	3.7	Waste streams including lead shielding, other elemental heavy metals, batteries, fluorescent light bulbs, explosives and propellants, compressed gases, lab packs, etc., which are not included in the other waste types. Generally, acceptable treatment technologies for these mixed waste streams do not exist.

The experimental program performed under this contract has provided MMT with the opportunity to demonstrate the ability of Catalytic Extraction Processing to process representative components of the DOE's MLLW inventory. The success of CEP with these varied waste streams is documented in previous sections of this report, indicating CEP's applicability to a broad spectrum of DOE wastes. Table 1.27 provides a summary of the various waste streams within each MWFA product line that are compatible with CEP. As shown, all of the wastes in the MWFA product lines are considered compatible with CEP. In selected cases, the economics of CEP may be less than competitive than those for a process optimized for a specific waste stream; however, the cost-effectiveness and competitiveness of CEP becomes apparent when considering the overall costs for processing the myriad waste streams at various DOE sites.

Table 1.27
CEP Applicability to DOE MLLW

Product Line	% MLLW Inventory	Waste Streams	CEP Compatibility
Wastewaters and Slurries	3.2	Wastewaters	PC
		Slurries	V
Combustible Organics	1.8	Liquids	V
		Slurries	V
Sludges and Soils	46.8	Sludges	V
		Soils	PC
		Inorganic particulate	V
		Paint wastes	PC
		Salt wastes	V
Solids, Debris, and Soils	44.5	Scrap Metal	V
		Wood	V
		PVC	V
		Concrete	V
		Misc. Debris/Soils	PC
		PPE	V
		Filters	V
Unique Wastes	3.7	Lead shielding	PC
		Elemental heavy metals	V
		Batteries	PC
		Fluorescent light bulbs	PC
		Explosives	PC
		Propellants	V
		Compressed gases	PC
		Lab packs	PC

LEGEND:
V: Validated Experimentally
PC: Projected Capability not yet validated experimentally

1.3.8.2. Advantages of CEP

CEP is a robust chemical process whose performance is based on fundamental chemical principles and engineering design. Given the chemical composition and physical form of the feed, the reaction pathways can be accurately predicted based on thermodynamics and the desired reactions can be carried out under appropriate engineering design. The process robustness while maintaining system performance is in contrast to combustion processes, where competing reactions take place and no accurate predictions of processing results can be achieved. The major technical features that allow CEP to provide significant environmental performance and waste minimization benefits are summarized below:

- CEP feed conversion efficiency is insensitive to the complexity of the molecular structure of the feed molecule. The liquid metal acts both as a homogeneous catalyst for the dissociation of complex feed molecules into their elemental constituents and as a solvent of these elemental intermediates. The flexibility and robustness of the CEP process are attributed to the 'singular' dissolved elemental intermediate through which reactions proceed. As a result, CEP feed conversion is independent of the complexity of the molecular structure of the feed molecule. DREs exceeding 99.9999% ('six nines') are typical in CEP regardless of the complexity of feed materials.
- CEP feed conversion efficiency is driven by solvation effects. CEP operates above the metal liquidus point, but does not use temperature as the primary means to change the physical and chemical composition of the feed materials. The carbon concentration in the liquid metal drives feed dissociation and dissolution. The lower the carbon concentration relative to saturation, the higher the forces driving the formation of the dissolved elemental intermediate (i.e., $\text{Fe}_3\text{C}(\ell)$) and hence more efficient feed conversion per unit time is achieved.
- CEP operates under a reducing environment, preventing the formation of undesirable oxidation byproducts (e.g., NO_x , SO_x). The solubility of carbon and oxygen in iron provides CEP with high chemical inertia and allows the system to maintain a reducing uniform environment over a wide range of operating conditions. Excess free oxygen cannot be found in the system to undergo alternate oxidation reaction pathways which lead to undesirable byproducts, such as NO_x , SO_x , dioxins, and furans.
- CEP does not provide pathways for the formation of dioxins or furans. Synthesis via CEP is achieved through specific dissolved intermediates in a reducing environment. No viable pathways are allowed for dioxins and furans formation. Analysis of the CPU off-gas stream in commercial-scale demonstrations of chlorinated waste processing have consistently shown trace levels to be non-detect to the stringent targeted regulatory level of 0.1 ng/Nm^3 (TEQ).

- CEP allows synthesis of high quality products. Synthesis of products is based on manipulation of the reaction pathways for the dissolved elemental intermediate through judicious addition of co-reactants and variation in operating conditions.
- CEP is not a thermal treatment technology. The effectiveness of a thermal treatment technology depends on the operation temperature, but this variable is not core to CEP's operation. Carbon concentration in the metal bath has been shown to be a variable for process optimization. CEP also allows a wide range of product formation, which can be engineered via the use of different metal solvents and co-reactants, depending on the desired ultimate uses of the feed materials. CEP provides minimum residual and undesirable gaseous emission because of its homogenous reaction environment and complete conversion.

The liquid metal bath and the reaction pathway via a singular dissolved intermediate give CEP a number of distinct advantages over conventional thermal processes:

- The liquid metal is a homogeneous catalytic and solvation medium which dissociates the feed into its elements, ensuring no intermediate products of incomplete dissociation. In open flame processes, the extent of dissociation to the intermediate free radicals is dependent on the residence time, temperature, oxygen environment, and system turbulence which are difficult to control. These parameters are readily controllable in CEP.
- The liquid metal solvent has high chemical inertia providing a means of dampening the effect of variations in feed composition, hence allowing for robust control of product quality.
- In CEP, the liquid metal in contact with the liquid ceramic phase can be used to engineer the separation and recycling of inorganics or metal compounds from complex feeds. This multiphase interaction provides CEP with the flexibility to synthesize and recycle desired products of high commercial value.

1.3.8.2.1. CEP Benefits Lead to Stakeholder Endorsement

MMT has built wide regulatory and community acceptance of CEP as a recycling technology. Four states (Massachusetts, Tennessee, Texas, and Louisiana) have designated CEP as a recycling process that is a non-combustion, non-incineration technology. The Texas Natural Resource Conservation Commission (TNRCC) has determined that CEP is a recycling technology that is distinct from incineration and can be considered a manufacturing technology because it converts wastes to useful products. Further, the state of Louisiana has ruled that CEP is a manufacturing process which is exempted from all RCRA regulation, including up-front storage of secondary material. The designation of CEP as a recycling technology is significant as it results in exemption from Resource Conservation and Recovery Act (RCRA) permitting

requirements, thereby avoiding the significant cost and time required to obtain a RCRA permit.

The state of Tennessee has issued permits for the two Quantum-CEP[®] facilities that are currently in operation for processing low level wastes. The headquarters of the U.S. Environmental Protection Agency has also recently recognized CEP as having achieved the Best Demonstrated Available Technology (BDAT) for processing all wastes for which incineration was previously the only approved processing method. EPA's determination reinforces the position that CEP is fundamentally different from incineration, and further confirms EPA's previous conclusions that CEP supports the agency's efforts to reduce the quantity of residuals for land disposal and to minimize cross-media contamination. MMT has also submitted a petition to EPA Region 4 requesting de-listing of its radioactive ceramic phase product from Quantum-CEP[®] processing which, if approved, will result in a reduction in disposal costs and may reduce stakeholder apprehension regarding disposal of the final product form.

MMT has successfully obtained approval and acceptance from both state and federal government agencies that CEP is a global solution for environmental protection. This acceptance has expedited siting and permitting for the two commercial-scale Quantum-CEP[®] facilities in operation and the commercial-scale CEP facility under construction for processing chlorinated organic wastes, thereby shortening their commercialization schedule and lowering overall project costs.

1.3.8.3. Cost-Benefit Analysis

At DOE's direction, MMT has performed a cost-benefit analysis for a conceptual 25,000 ton per year (tpy) bulk solids Quantum-CEP[®] facility for processing materials from DOE's decontamination and decommissioning activities. MMT has also included a summary of the independent life cycle cost analysis performed during Phase 2 of the DOE's Integrated Thermal Treatment System (ITTS) Study, as well as a brief discussion of Quantum-CEP[®] competition in the open market. Each analysis provides important information regarding the cost-effectiveness of using Quantum-CEP[®] for processing DOE MLLW. The cost analysis for a first-of-its-kind 25,000 tpy bulk solids processing facility provides projected capital and operating costs based on the status of CEP at the completion of this contract; ongoing MMT-sponsored R&D is anticipated to substantially reduce both the capital and operating costs for future CEP systems with similar capabilities within the next few years. The total life cycle cost estimates contained in the ITTS Study Phase 2 Results provides important comparative cost information for 19 MLLW thermal treatment systems based on a consistent set of assumptions and evaluation criteria. The ITTS Study concluded that CEP treated the largest percentage of MLLW of all primary treatment technologies and had the lowest overall total life cycle cost. While the absolute value for total life cycle cost may be arguable, the relative rankings provide DOE with valuable insight as CEP was among

the five lowest total life cycle cost systems for the 19 thermal treatment systems evaluated.

1.3.8.3.1. Bulk Solids Materials from Decontamination and Decommissioning Activities

DOE requested MMT provide a conceptual design for a Quantum-CEP[®] facility capable of processing 25,000 tons per year of bulk solids materials representative of its planned decontamination and decommissioning activities, which is provided in Section 7 of this report. The composition of the waste stream is presented in Table 1.28.

Table 1.28
Typical DOE Surrogate Waste Components

Waste Component	Wt %
Activated Carbon	5
Wood (4 x4, random lengths to 4 ft., simulated pellets)	15
PVC (Pipe, Sch 40, 2-4" OD, Random lengths to 4 ft)	10
Neoprene Rubber	3
Glass Beads (1/4" diameter)	5
Sheet Metal, galvanized (crushed desks, ducts, cabinets)	15
Mild Steel (4" width, random length up to 4 ft)	15
Stainless Steel (Pipe, 3 to 4 ft long, 1 to 4 inch OD)	8
Alumina Crucibles	7
Leaded Gloves	3
Water	4
Cement (Chunks: 4-12" diameter)	5

As discussed in Section 2.2 of this report, MMT has estimated that a 12 ton Catalytic Processing Unit is required for Zone 1 processing. To perform the requested cost-benefit analysis, MMT has estimated the capital and operating costs for this conceptual facility to remain consistent with DOE's overall guidance. Operations were projected to begin in 1998 and to be completed by 2010. The capital and operating costs for the conceptual 25,000 ton per year Quantum-CEP[®] processing facility are summarized in Table 1.29 and Table 1.30, respectively. The capital and operating cost estimates reflect the current projected costs associated with this first-of-a-kind bulk solids processing facility; ongoing MMT-funded initiatives are expected to substantially reduce both capital and operating costs in the near term. The operating cost estimate assumes that all ceramic phase product is disposed in a RCRA-permitted engineered disposal facility at \$243/ft³ (consistent with the ITTS Study) and that all metal phase product is reused within the DOE complex. The potential economic benefits that would be derived from disposal of delisted ceramic phase product at \$100/ft³ (consistent with the ITTS Study) or re-use within the DOE complex (e.g., abrasive material) have not been considered.

Table 1.29
Capital Cost Information for a 25,000 tons per year Quantum-CEP® Facility
Processing Heterogeneous Mixed Waste from DOE D&D Activities (1997 dollars)

Item	Cost
Building/Utilities/Facilities/Process Equipment	\$ 27,677,579
Material	\$ 17,417,425
Field Labor	\$ 20,198,653
Construction Indirects-Contractor	\$ 10,635,125
Construction Indirects-MMT	\$ 2,695,943
Total Engineering	\$ 33,233,558
Miscellaneous	\$ 6,417,947
Start-up	\$ 14,766,998
Management Reserve	\$ 23,446,154
TOTAL	\$ 156,499,370
Note: Feed delivery, siting, and other Outside Battery Limits constraints can significantly affect facility complexity and overall cost.	

Table 1.30
Annual Operating Cost Information for a 25,000 tons per year Quantum-CEP® Facility
Processing Heterogeneous Mixed Waste from DOE D&D Activities (1997 dollars)

Item	Cost
Operating Personnel	\$ 36,776,025
Consumables	\$ 694,980
Operations	\$ 14,073,345
Ceramic Handling and Disposal	\$ 34,633,170
Stabilized Material Disposal	\$ 2,162,160
Sampling and Analysis	\$ 10,270,260
Maintenance	\$ 17,271,701
TOTAL	\$ 115,888,641
Operating cost per pound feed	\$ 2.32

The overall cost per pound for treatment and disposal of this waste stream, including return on capital, profit, insurance, facility overhead, taxes, etc. is approximately \$3.75/lb (in 1997 dollars). The heterogeneous feed processed in the Quantum-CEP® facility makes it difficult to make cost comparisons with competing technologies as most publicly available data is for relatively homogeneous waste streams (e.g., radioactive scrap steel). A summary of various cost estimates for processing DOE wastes obtained from a variety of sources is presented in Table 1.31. Given differences in waste composition, facility throughput, cost estimating assumptions, and maturity of

each of the technologies shown in the table, CEP is clearly a cost-effective method for processing DOE MLLW.

Table 1.31
Cost Estimates for Processing DOE Wastes

Waste Stream	Technology	Estimated Cost
Heterogeneous Waste from D&D Activities	19 systems evaluated in ITTS Study	\$ 9.00 - \$12.60/lb ¹ (treatment + disposal)
Low Level Waste	Various	\$1618/m ³ average ² (treatment + disposal)
Mixed Low Level Waste	Various	\$10,188/m ³ average ² (treatment + disposal)
Radioactive Scrap Steel	Small scale demonstrations	\$3.90 - \$14.00/lb ³
	Surface decontamination	\$1.30 - \$5.71/lb ³
	Other volume recycling methods	\$2.25 - \$3.00/lb ⁴
Organic Liquids/Solids	TSCA Incinerator	\$7.00 - \$8.00/lb ⁵
Inorganic Liquids/Solids	Transportable Vitrification	\$6.00 - \$7.00/lb ⁶
Inorganic Liquids/Solids	Grout	\$7.00 - \$8.00/lb ⁶

¹ Integrated Thermal Treatment System Study-Phase 2 Results, February 1996.

² Waste Management Progress Report, August 1996.

³ "Metal Products and Outlets," Trinity Environmental Systems, January 1995.

⁴ "Reclamation and Reduction of Nuclear Residues," South Carolina Research Authority, October 1994..

⁵ Salmon, R, SL Loghry, and WH Hermes, "Operating Cost Guidelines for Benchmarking DOE Thermal Treatment Systems for Low-Level Mixed Waste", Martin Marietta Energy Systems, Inc., ORNL/TM-12828, November 1994.

⁶ Sexton, JL and LR Dole, "Life Cycle Benefit-Cost Analysis of Alternatives for the Deployment of the Transportable Systems for Low-Level Mixed Waste, Martin Marietta Energy Systems, Inc., ORNL/TM-12828, July 1996.

1.3.8.3.2. Summary of ITTS Study Phase 2 Results

The U.S. Department of Energy's Environmental Management Office of Technology Development (EM-50) commissioned the ITTS Study to assess alternative systems for treating contact-handled, alpha and nonalpha, mixed low level waste (MLLW). The purpose of the study was to conduct a systematic engineering evaluation of a variety of MLLW treatment system alternatives using a common set of assumptions and evaluation criteria. The Planning Life-Cycle Cost (PLCC) Estimates and Sensitivity Analysis performed as part of ITTS Phase 2 included 19 thermal treatment systems DOE considered appropriate for processing MLLW. The PLCC was calculated over a 20 year period for a feed rate of 2,927 lb/hr and 4,032 operating hours per year, which equates to approximately 5,900 tons per year (approximately 24% of the capacity of the CEP facility discussed in Section 1.3.8.3.1). The various waste types used in the study were obtained from the Mixed Waste Inventory Report (MWIR), which provided the

basis for a detailed estimate of the physical and chemical composition of the waste for each DOE site. The composition of the MLLW feed slate used in the ITTS Study is provided in Table 1.32, which represents an "average" waste composition for the DOE Complex.

Table 1.32
DOE MLLW Feed used in ITTS Study³

Type of Waste	Mass Feed Rate (lb/hr)	Wt%
Metal with surface contamination	468	16.0
Metal with fixed contamination	149	5.1
Combustible solids, liquids, and debris	660	22.5
Noncombustible solids, liquids, and debris	1,340	45.8
Lead with surface contamination	5	0.2
Lead with entrained contamination	21	0.7
Aqueous waste	80	2.7
Mercury contaminated waste	50	1.7
Special waste	153	5.2
Sulfate and halide salts	1	< 0.1
TOTAL	2,927	100

Each of the 19 systems were evaluated for the ability of the primary treatment system to treat the input waste stream; the results are presented in Figure 1-23. CEP was evaluated to process the largest percentage of input waste (90%), followed by slagging rotary kiln and plasma-based technologies at 73%. Thermal desorption with either mediated electrochemical oxidation or supercritical water oxidation had the lowest percentage of input waste treated by the primary treatment system (15%).

³ Feizollahi, Fred and Quapp, William J., "Integrated Thermal Treatment System Study-Phase 2 Results," Idaho National Engineering Laboratory, Lockheed Idaho Technologies Company, February 1996.

Figure 1-23
Percentage of Input Waste Treated by Main Treatment Technology as a Percentage of
Total Waste Input to the Facility⁴

The Planning Life-Cycle Cost (PLCC) estimates and sensitivity analysis performed for the ITTS Study Phase 2 were based on a consistent set of assumptions and evaluation criteria. The PLCC estimates included both treatment, disposal, and facility decontamination and decommissioning costs. A summary of the PLCC estimates for the 19 thermal treatment systems is presented in Figure 1-24.

⁴ Ibid, p. 3-25.

Figure 1-24
Total Life-Cycle Cost Estimates Based Upon 20 Years Operation⁵

The cost-effectiveness for employing Quantum-CEP[®] for processing DOE MLLW becomes more apparent when comparing the percentage of input waste treated by the primary treatment system with the total life cycle cost (TLCC) for the five treatment systems with the lowest TLCC. From examination of Figure 1-25 and Figure 1-26, it can be concluded from these figures that CEP is the most cost-effective technology for processing DOE MLLW; the conclusion remains the same if the relatively minor variations in TLCC are discounted due to the broad spectrum of DOE MLLW that can be processed using Quantum-CEP[®].

⁵ Ibid, p. 4-40.

Figure 1-25
Feed Treatability Comparison for Top 5 Treatment Systems⁶

Figure 1-26
Total Life Cycle Cost Comparison for Top 5 Treatment Systems⁷

The ITTS Study also determined the processing cost and the disposal cost for each of the 19 thermal treatment systems, which are presented in Table 1.33. According to the ITTS results, CEP has the lowest treatment cost, the second lowest disposal cost, and the lowest overall cost for treatment and disposal per pound of input waste. A disposal

⁶ Ibid, p.3-25.

⁷ Ibid, p. 4-40.

cost of \$243 per cubic foot was used based on an average sized, RCRA-permitted, engineered disposal facility. The ITTS Study also indicated that disposal costs could be reduced to \$100 per cubic foot for delisted wastes, which represents a significant potential savings for treatment systems producing delisted products for final disposal. MMT has submitted a petition to EPA Region IV requesting de-listing of its radioactive ceramic phase product from Quantum-CEP[®] processing; a ruling is expected in mid 1997.

Table 1.33
ITTS Study Processing and Disposal Cost Summary⁸

Thermal Treatment System	Treatment Cost (\$/lb.)	Disposal Cost (\$/lb)	Treatment + Disposal Cost (\$/lb.)
Catalytic Extraction Processing	8.02	0.96	8.98
Joule-Heated Vitrification	8.14	1.13	9.27
Plasma Gasification	8.17	1.10	9.27
Slagging Rotary Kiln	8.11	1.20	9.31
Plasma Furnace	8.39	1.10	9.49
Plasma Furnace, CO ₂ retention	8.88	1.25	10.13
Rotary Kiln, Air	9.18	1.13	10.31
Rotary Kiln, O ₂	9.18	1.13	10.31
Rotary Kiln, Air, Max recycle	9.47	0.89	10.36
Steam Gasification	9.29	1.13	10.42
Rotary Kiln, Air, Wet APC	9.08	1.41	10.49
Indirectly Heated Pyrolyzer	9.50	1.13	10.63
Molten Salt Oxidation	9.64	1.21	10.85
Fixed Hearth Pyrolyzer, CO ₂ retention	9.91	1.26	11.17
Rotary Kiln, Air, Polymer stab.	8.83	2.37	11.20
Rotary Kiln, Air, CO ₂ retention	9.67	1.63	11.30
Rotary Kiln, Air, Thermal desorption	9.28	2.56	11.84
Thermal desorption and SCWO	8.89	3.37	12.26
Thermal desorption and MEO	9.08	3.54	12.62
Average Cost	8.98	1.55	10.54

1.3.8.3.3. Competition in the Open Market

The DOE, in its "Report of the Technical Review Team on the Catalytic Extraction Process", Office of Science and Technology, DOE-EM (March 1996), found CEP to be "sufficiently developed that MMT should be able to compete in the marketplace by

⁸ Ibid, p. 4-30.

adapting CEP for processing DOE's various types of mixed low-level waste". In fact, MMT will deploy two facilities for LLW and MLLW processing in 1997.

The first of these is the Quantum-CEP® operation in Oak Ridge, TN, for the processing of spent ion exchange resins and other wet solid wastes generated from radioactive water processing at nuclear reactors. Because of rising disposal costs for spent resins and Quantum-CEP's volume reduction and recycle advantages, MMT is uniquely positioned to provide a cost-effective solution to the commercial nuclear industry and expects to capture a significant percentage of this market. Through the first quarter of 1997, Quantum-CEP® has treated nearly 200,000 lb. of resins since beginning radioactive operations. Both the DOE and DOD, specifically the nuclear Navy, generate a significant amount of spent resins. As experience is gained during the initial period of operations and operational efficiencies are improved, these waste streams could become target streams for Quantum-CEP®.

The M4 Technology Center, also in Oak Ridge, contains two Quantum-CEP® units, one for solids processing (RPU-3) and the other for liquids (RPU-4). Both units were operated in 1996 for research and development activities to support Quantum-CEP® designation as a recycle technology. In mid-summer 1997, both units will begin production-scale processing. Contracts have been signed for processing of nuclear utility MLLW and MMT has begun discussions with DOE for processing problematic mixed wastes.

1.3.8.4. Conclusions

This contract has established the technical feasibility and economic viability of Quantum-CEP® for processing the broad spectrum of mixed low level waste in the DOE inventory. Quantum-CEP® has been demonstrated by this report to be a cost-effective solution for processing DOE MLLW. MMT's successful construction, licensing, and operation of two commercial-scale Quantum-CEP® processing facilities in Oak Ridge, TN, and signed contracts for treatment of commercial wastes demonstrate achievement of DOE's overall objective for this contract, which was to establish the commercial capability of Catalytic Extraction Processing. Further, the Environmental Protection Agency's determination that CEP is a non-incineration process that provides equivalent treatment for all wastes for which incineration had been mandated Best Demonstrated Available Technology (BDAT) will substantially enhance CEP's commercial potential. Current and future MMT-sponsored capital and operating initiatives to improve capabilities and reduce costs, and a pending ruling by EPA Region IV regarding the delisting of Quantum-CEP® ceramic phase products will further increase CEP's benefits to DOE and other customers.

1.4. Conclusions and Recommendations

1.4.1. Conclusions

The research and development activities performed under this contract have met and/or exceeded all objectives defined in the Statement of Work. MMT has convincingly demonstrated Catalytic Extraction Processing (CEP) to be a robust, one-step process that is relatively insensitive to wide variations in waste composition and is applicable to a broad spectrum of DOE wastes. Substantial growth in the feed size and composition compatible with CEP has been achieved in a relatively short period of time, providing the opportunity to substantially reduce the amount of feed characterization and preparation prior to processing. Additional research and development is expected to lead to the ability to accept a drum (and potentially larger) size feed of completely uncharacterized waste. Experiments have also validated our Catalytic Processing Unit (CPU) design model, providing a high degree of confidence in our ability to design a bulk solids CPU for processing DOE wastes. Quantum-CEP[®] has successfully demonstrated its ability to partition radionuclides to the ceramic phase, thereby achieving a significant reduction in the volume of radioactive waste while attaining metal decontamination factors in excess of 99.9%. CEP has also successfully demonstrated its ability to recycle RCRA hazardous wastes and achieve destruction removal efficiencies exceeding 99.9999% for organic wastes. The Commercial Nuclear Waste and M4 commercial-scale CEP facilities have been placed in commission and are currently processing mixed low level wastes, which provides a compelling indication of the maturity, regulatory acceptance, and commercial viability of CEP. A third commercial facility for processing chlorinated organic feed materials is currently under construction in Bay City, TX, and is expected to be commissioned in 1997.

1.4.2. Recommendations

The efforts concluded under this contract have shown conclusively that CEP is an effective means for decontaminating scrap metal and other DOE waste streams for recycling. It provides superior environmental performance in a cost effective manner, yielding very high metal decontamination factors due to its unique process chemistry. Continued studies in select areas within CEP's operating scheme will allow its designers to optimize systems and advance the progress of CEP's commercialization and minimize the time to actual implementation of a full scale system. Further, future development work should include studies that address the specific needs of particular DOE processing sites in preparation for establishing CEP facilities to process waste streams located at those sites.

This contract has provided the means to evaluate several concepts proposed for the addition of bulk solids to CEP systems to provide the most cost-effective alternative for recycling of DOE's contaminated scrap metal. The experiments proved that the techniques will work. Further refinement could allow the addition of whole drums or

similar-sized materials to the bath which eliminate the need for most size-reduction feed pretreatment systems, thereby achieving a substantial savings in the total cost for decontamination and decommissioning of DOE sites.

This contract has provided the means to theoretically predict the separation of uranium and transuranics (TRU) from contaminated scrap metal making use of the well-controlled operating chemistry of CEP. Future development work should be considered to continue studies on the application of CEP to TRU, including the processing of actual TRU radionuclides, and expand studies on its application to high level wastes (HLW) in the DOE inventory. Progressing beyond these isotopes, the DOE has a long-standing interest in the separation of metals of similar thermochemistry (e.g., Tc and Ni) which are not easily separated using classical techniques (e.g., oxidation). MMT has performed preliminary investigations into several promising methods for separation of similar metals that support scale-up to molten metal test systems. CEP offers a potential means to recycle and reuse the approximately \$1.5 billion of radioactive contaminated nickel in the DOE inventory.

MMT has processed RCRA-listed wastes, and has proven CEP's ability to provide complete destruction of the hazardous constituents while maintaining high regulatory integrity and producing a useful gas phase product. Due to the unique set of regulations governing the processing of RCRA listed materials, the disposal of a stable final form containing radionuclides but not exhibiting any RCRA characteristic would benefit from delisting. Delisting would provide increased flexibility in terms of disposal destination and cost. MMT submitted a delisting petition to EPA Region IV in November, 1996; a ruling is expected in mid 1997.

The U.S. Department of Energy's Ten-Year Plan initiative is intended to lay a path for accelerating clean-up and progressing as much as possible at DOE's sites within a ten-year period. CEP has been proven to be a viable solution to many of DOE's waste problems, providing recycling, environmental integrity and dramatic volume reduction of radionuclide-containing waste forms. Two privately funded Quantum-CEPTM facilities are currently operational in Oak Ridge, TN, for processing commercial and DOE mixed wastes. These facilities attest to MMT's commitment to assist DOE and the commercial nuclear industry in processing their wastes. Detailed design and construction of additional Quantum-CEP[®] facilities will provide DOE with a cost-effective means of achieving the challenging goals included in its Ten-Year Plan.