#4

Advanced Technologies for Decontamination and Conversion of Scrap Metal

Phase III Topical Report
Contract Number DE-AC21-93MC30170

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Abstract

The Department of Energy (DOE) faces the task of decommissioning much of the vast U.S. weapons complex. One challenge of this effort includes the disposition of large amounts of radioactively contaminated scrap metal (RSM) including but not limited to steel, nickel, copper, and aluminum. The decontamination and recycling of RSM has become a key element in the DOE's strategy for cleanup of contaminated sites and facilities. Recycling helps to offset the cost of decommissioning and saves valuable space in the waste disposal facilities. It also reduces the amount of environmental effects associated with mining new metals.

Work on this project is geared toward finding decontamination and / or recycling alternatives for the RSM contained in the decommissioned gaseous diffusion plants including approximately 40,000 tons of nickel. The nickel is contaminated with Technetium-99, and is difficult to remove using traditional decontamination technologies.

The project, titled "Advanced Technologies for Decontamination and Conversion of Scrap Metal was proposed as a four phase project. Phase I and II are complete and Phase III will complete May 31, 1999.

Stainless steel made from contaminated nickel barrier was successfully produced in Phase I. An economic evaluation was performed and a market study of potential products from the recycled metal was completed. Inducto-slag refining, after extensive testing, was eliminated as an alternative to remove technetium contamination from nickel.

Phase II included successful lab scale and pilot scale demonstrations of electrorefining to separate technetium from nickel. This effort included a survey of available technologies to detect technetium in volumetrically contaminated metals. A new process to make sanitary drums from RSM was developed and implemented.

Phase III included a full scale demonstration of electrorefining, an evaluation of electro-refining alternatives including direct dissolution, melting of nickel into anodes, a laser cutting demonstration, an investigation of commercial markets for RSM, and refinement of methods to quantify isotopic elements,

Acknowledgements

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List of Acronyms

A	amps
AFCH	Auto focus cutting head
ALARA	As Low As Reasonably Achievable
ANSI	American National Standards Institute
ASME	American Society of Mechanical Engineers
BNFL	British Nuclear Fuels
Bq	Bequerel
CSM	Colorado School of Mines
D&D	Decontamination and Decommissioning
DOE	Department of Energy
DWPF	Defense Waste Processing Facility
ETEC	Energy Technology Engineering Center
ETTP	East Tennessee Technology Park
EWI	Edison Welding Institute
FETC	Federal Energy Technology Center
g	gram
HEPA	High efficiency particulate air
INEL	Idaho National Engineering Labs
LGT	Lockwood Green Technologies
MRU	Metals recovery unit
MSC	Manufacturing Sciences Corporation
NDA	Non destructive analysis
NdYAG	Neodymium
ORNL	Oak Ridge National Laboratory
PLC	Programmable logic controller
PRDA	Programmatic Research and Development Agreement
QA	Quality Assurance
RSM	Radioactively contaminated scrap metal
VIDP	Vacuum Induction Degas and Pour
VIM	Vacuum Induction Melter

Executive Summary

Introduction

The DOE has begun to decommission the vast United States nuclear weapons complex. This effort includes the disposition of large amounts of radioactively contaminated scrap metal including but not limited to steel, nickel, copper, and aluminum. Diffusion plant nickel barrier from the Oak Ridge, Paducah, and Portsmouth diffusion plants accounts for nearly 40,000 tons of contaminated nickel.

Work on this project is geared toward finding decontamination and recycling alternatives for the nickel barrier. Technetium-99, present in the nickel barrier, is known to be particularly troublesome to remove using conventional decontamination technologies. Traditional chemical and mechanical means have been ineffective at removing this contaminate from the nickel barrier.

Recycle options available for the disposition of the nickel include decontamination and release, and recycled product manufacture.

Work during Phase I of this project:

- Successfully demonstrated the ability to make stainless steel from nickel barrier feed.
- After repeated trials and studies, the inducto-slag decontamination process was eliminated as a suitable alternative.

Phase II efforts included:

- Successful lab scale and pilot scale demonstrations of electrorefining to separate technetium from nickel.
- A survey of available technologies to detect technetium in volumetrically contaminated metals.
- A new process to make sanitary drums from RSM was developed and implemented.

Work in phase III of the project, as proposed, contained the following five elements:

- Nickel barrier decontamination using electrorefining
- Full scale laser cutting demonstration
- Develop and fabricate a new product made from RSM
- Refinement of methods to quantify isotopic elements
- Project Management

Purpose

The expected results for phase III, as proposed were:

- To remove technetium from nickel using a full scale electrorefining cell
- To determine if technetium could be removed from nickel in the classified form (without declassification by melting)
- To section a diffusion plant converter shell into pieces small enough to be processed through MSC's recycling facility
- To fabricate a new product using RSM

Background

On August 25, 1997, BNFL Inc. and the Department of Energy (DOE) signed a $238 million fixed price contract for the Decontamination and Decommissioning (D&D) of Three Buildings at the former K-25 site (now called the East Tennessee Technology Park – ETTP). BNFL Inc. is to dismantle, remove, decontaminate, and recycle the process equipment within the three Gaseous Diffusion Plant buildings K-33, K-31, and K29, which represent approximately five million square feet of floor space. The ETTP Three Building D&D project supports DOE's Vision 2010 to clean up these facilities and locate new commercial industries at the former K-25 site.

BNFL Inc. contracted with Manufacturing Sciences Corporation (MSC) to decontaminate and recycle more than 126,000 tons of metals, including the 6,000 tons of nickel. MSC will decontaminate the metals and recycle them where economically feasible. The contract recognized the value of recovering the metals, including nickel, and the DOE has received a credit for this material. The following is a quote from the DOE/ORO office web page:

> "The $238 M contract cost included a credit back to DOE of $55,569,748 for the recyclable material. This amounts to quarterly cost savings of $2,646,178 over 21 quarters for the materials recycled or reused. The recycling activities began in the 4th calendar year quarter of 1998 and will continue throughout the duration of the contract."

As part of the ETTP Three Building Decomissioning and Decontamination (D&D) Project, approximately 6,000 tons of radioactively contaminated nickel will be removed. This nickel will be decontaminated and eventually released for reuse. The process that will be used for decontamination of the nickel, electrorefining, was developed under the FETC contract during phases II and III.

Methodology

The detailed descriptions of experimentation that took place during phase III appear in this report. Also, activities that took place near the end of phase II, and due to timing of the report, were not reported in the phase II report, are described in section 3.0. This report is categorized into five main headings as follows:

- 3.0 Pilot Scale Electrorefining Cell (from Phase II)
- 3.1 Full Scale Electrorefining
- 3.2 Full Scale Laser Cutting Demonstration
- 3.3 Develop and Fabricate a New Product from RSM
- 3.4 Refinement of Methods to Quantify Isotopic Elements

3.0 Pilot Scale Electrorefining

3.0.1 Background and Results of Preliminary Research

DOE facilities have an estimated 1.5-million-ton inventory of radioactive scrap metal (RSM) that must be dispositioned. The safe recycle of the metals is environmentally preferable to disposal in waste facilities as it saves space, and reduces the overall environmental effects and cost associated with producing new metals.

Because much of the RSM is high-value nickel, the potential for cost recovery is significant. DOE's gaseous diffusion plants have an estimated 40,000 tons (80,000,000 pounds) of nickel barrier.

Manufacturing Sciences Corporation worked under a contract with the Federal Energy Technology Center (FETC) in Morgantown, West Virginia to research and develop processes to decontaminate the nickel barrier. This work was completed under a Programmatic Research and Development Agreement (PRDA) with FETC. The original objective of the research and development was to reduce the contaminants in the nickel to levels suitable for recycle.

3.0.2 Removing Contaminants from the RSM

The nickel in the gaseous diffusion process is contaminated with technetium (^{99}Tc) and uranium, along with possible trace amounts of transuranic elements (Np, Pu, and other actinides). The melting process removes most of the uranium & transuranic elements. However, ^{99}Tc becomes stubbornly bound to the nickel, making it particularly difficult to separate the two elements.

Two processes had been considered as good options for decontaminating the nickel; namely, melt refining and electrorefining. Melt refining is done in a vacuum induction melting (VIM) furnace in the presence of an oxidizing flux. The melt refining process eliminates the uranium and transuranic elements, but is ineffective in removing technetium. Electrorefining dissolves the nickel electrolytically (as an anode) in an acidic aqueous electrolyte; and the purified nickel is deposited in its metallic state on a cathode. Like melt refining, electrorefining is also capable of eliminating the uranium and transuranic elements. However, the process, as practiced by others prior to this PRDA agreement, is unable to reduce the amount of technetium in the nickel to levels near 1Bq/g.

Several secondary processes were investigated in an effort to separate the technetium from the nickel, including ion exchange, liquid-liquid extraction, chemical precipitation, evaporation, and carbon adsorption. These did not show promise. One option that did appear promising, however, was an electrorefining process that incorporated both a cationic membrane between the anode and cathode and a secondary cementation (or displacement reaction) treatment. The membrane filter and the displacement treatment removed technetium from the electrolyte before the nickel deposited on the cathode and proved to be exceptional in preventing the technetium from co-depositing with the nickel. Further, this process eliminated the need for the ion exchange treatments previously proposed for removing technetium.

An effective electrorefining and metal displacement approach to removing contaminants was proposed by Covofinish, Inc., Providence, Rhode Island.

3.0.3 Electrorefining Research

MSC subcontracted with Covofinish to apply their electrorefining and displacement reaction process toward removing technetium from contaminated nickel barrier. The initial objectives of these experiments were:

- to determine whether or not decontamination could be effectively and economically achieved through electrorefining; and if so,
- to optimize the parameters needed for successful pilot-scale operations.

Covofinish researchers, under the subcontract, designed and developed a lab-scale electrorefining system to conduct experiments on clean nickel. In addition, Covofinish contracted Brown University to establish an operational equilibrium expression that would accurately predict the effects of pH, temperature, and nickel concentration on nickel plating and technetium displacement. Brown researchers also documented other kinetic data. A brief summary of the lab-scale research, the overall results, and the parameters established for a pilot-scale electrorefining cell are provided in the following paragraphs. (Details of the Covofinish and Brown University research are available in a topical report issued to FETC at the end of Phase II titled "Advanced Technologies for Decontamination and Conversion)

Brown University researchers determined that both temperature and pH significantly effected the displacement reaction of ^{99}Tc from the nickel sulfate solution. Researchers found that the optimal displacement environment (rate of displacement and quality of nickel plating) occurred at a temperature of about 40 degrees C, with pH levels of 4.0 to 4.5. By fitting experimental results with first-order reaction rate expressions, Brown researchers also developed an equation for the rate of displacement reaction as a function of temperature and nickel surface area.

Optimized parameters provided by the Brown research were incorporated into the 500 ml lab-scale electrorefining system set up at Covofinish. Using the equation and other data provided by Brown University, Covofinish researchers developed a numerical model to calculate other parameters, such as the amount of nickel powder necessary to maintain the desired ^{99}Tc concentrations during cementation processes and the rate of acid addition to maintain the pH and increase the effectiveness of the liquid/solid contactor.

Finally, Covofinish performed a 42-hour experiment using pieces of previously melted contaminated nickel barrier (supplied by MSC). A lab-scale tank was filled with technetium-free Watt's nickel electrolyte, and the anolyte was separated from the catholyte by a Nafion™ cationic membrane. During the concluding lab-scale experiments, the nickel anode was electro-dissolved; and the technetium was successfully separated from the nickel ions. Technetium ions, which were confined to the anolyte by the Nafion™ membrane, were subsequently displaced onto a fresh nickel surface in the cementation unit. The nickel ions were free to pass through the membrane and plate out on the cathode without a co-deposit of technetium.

The nickel plating on the cathode was sampled at two time intervals at current densities of 100A/ft^2. and 34 A/ ft^2. Also, the technetium concentrations in both the anolyte and the catholyte were periodically monitored. The 460 Bq/g of contamination initially found in MSC's nickel ingot was reduced by the electrorefining processes to values around 1 Bq/g.

3.0.4 Initial Development of a Pilot-Scale Electrorefining Cell

After successfully completing the lab-scale experiments, Covofinish researchers began developing a pilot-scale electrorefining cell for further optimizing the process. The pilot-scale research efforts were divided into four major tasks; design, fabrication, proof of concept, and electrorefining of the ^{99}Tc contaminated nickel. As in the lab-scale experiments, the nickel in the pilot-scale cell would be electrodissolved into the electrolyte, passed through the cationic membrane, and plated out onto the cathode. The technetium contamination would be trapped by the membrane and would remain in the anolyte (the solution surrounding the anode). Finally, the anolyte would be passed through a regenerative process (that is, a displacement reaction in the cementation unit) which would remove the technetium and other contaminates.

3.0.5 Design and Fabrication of Pilot Scale Cell

The objectives for designing and building a pilot-scale electrorefining cell at MSC were:

1. To demonstrate that the parameters for the lab-scale electrorefining experiments could be equally as successful in a production environment.
2. To provide important production data for establishing design criteria and operating parameters needed to develop a full-scale cell.

Generally, the pilot-scale cell was a modification of the lab-scale design, both in configuration and in materials. The anolyte in the pilot-scale cell was designed to be continuously circulated through a cementation chamber that could capture the technetium ions and prevent them from co-depositing with the nickel on the cathode. Major components of the pilot-scale electrorefining cell as set up at Covofinish included:

Cell Tank: The cell was scaled up from the 500 ml lab-scale cell to a 200 gallon pilot cell. The cell was fabricated from 1/2-inch, stress-free polypropylene.

Anodes and Cathodes: For initial trial "clean" testing, anodes were made from clean nickel, and a stainless steel sheet with a nonconducting edge frame was used as the initial cathode. The system was operated at 20 A/ ft^2, to allow nickel plating on the cathode starter sheet. The cathode was withdrawn and the edge frame removed. Deposited nickel foil sheets peeled from the stainless steel cathode were straightened, equipped with nickel foil hanging straps, and used as the cathode starter sheets in subsequent experiments.

Electrolyte Solution: Watts nickel electrolyte was prepared and added to the cell tank. Watts nickel electrolyte contains nickel sulfate, nickel chloride, and boric acid.

Makeup Water: Filtered makeup water was automatically introduced into the electrolyte as needed to replenish water lost through evaporation. This was accomplished through the use of a solenoid valve signaled by a level probe.

Heaters: The electrolyte was pumped to a heated sump to maintain temperature.

Filters: Electrolyte (both anolyte and catholyte) was also pumped through 5 micron polyester filters for particulate removal.

Pipes and Fittings: Pipes and fittings for the pilot-scale cell were either manufactured at Covofinish or designed by Covofinish researchers and manufactured by qualified vendors.

Cementation Unit: Anolyte was circulated through a cementation chamber. Removal of technetium from the anolyte was accomplished using the attraction of technetium to fresh nickel surface.

Data Logging: Various sensors measuring voltage, temperature, pH, and other variables were monitored periodically and documented. Anolyte and catholyte activity were also monitored daily by sampling.

A sketch of the pilot scale nickel electrorefining cell is provided in Figure 3.0.1.

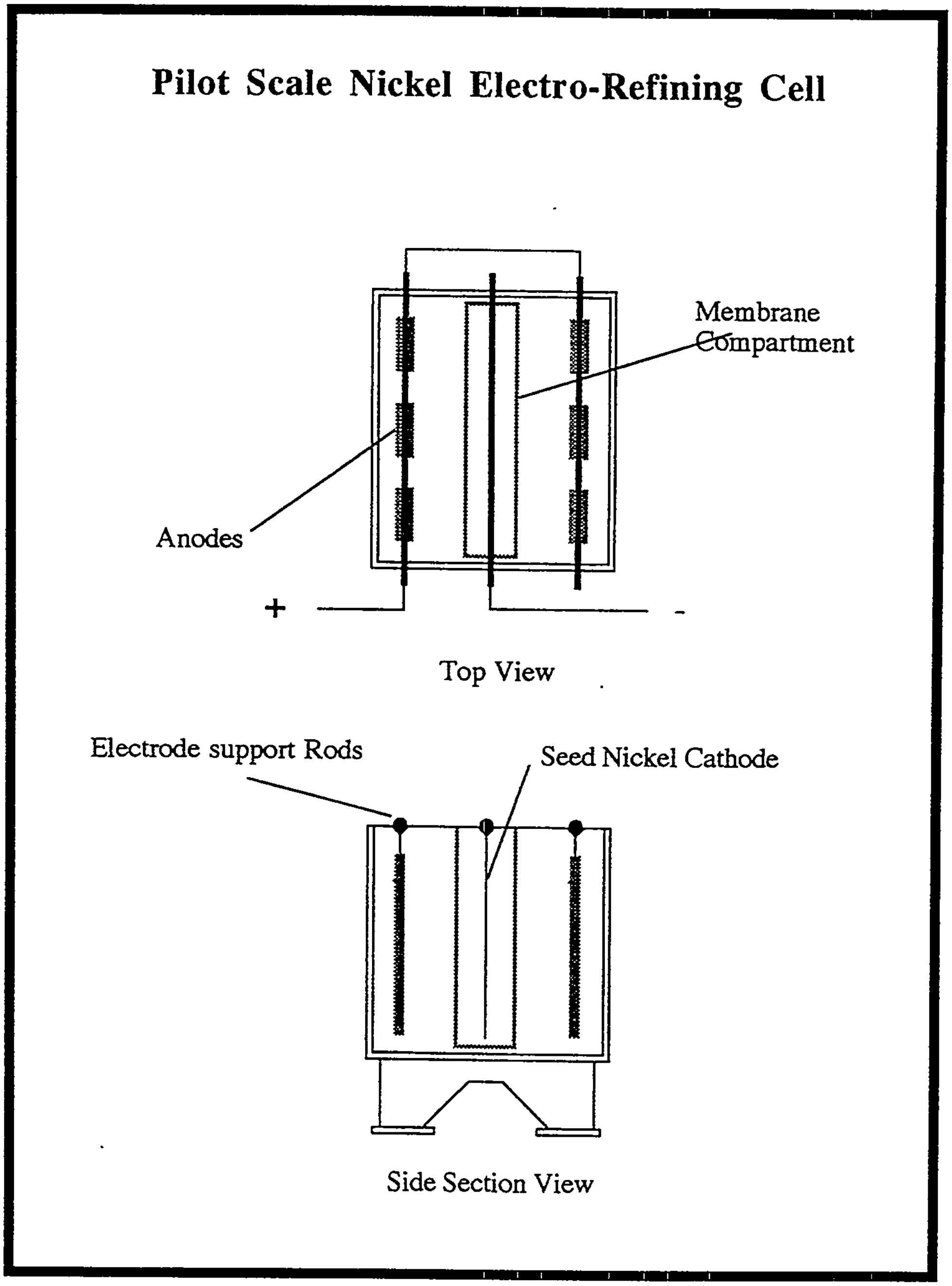


Figure 3.0.1. Sketch of pilot-scale cell.

3.0.6 Pilot Cell Pre-Testing

Covofinish researchers filled the pilot-scale cell with water and performed a trial run of the integrated electrorefining system. The purpose of the trial run was to verify proper operation of valves, heaters, probes, and pumps before initiating electrorefining pretests on the pilot-scale cell. These pretests not only confirmed proof-of-concept but also determined where modifications were needed to further optimize the cell's efficiency prior to shipping the cell to MSC.

Covofinish researchers further optimized current density, cell voltage, and the rate of acid addition to improve cathode quality. During the pretests, Covofinish also optimized parameters on the displacement reaction in the cementation unit which resulted in further improvements. After pre-testing experiments were completed, Covofinish dismantled and shipped the pilot-scale electro-refining cell to MSC for experiments using ^{99}Tc contaminated nickel.

3.0.7 Pilot-Scale Electro-refining at MSC

MSC personnel installed the cell components received from Covofinish, and set up the required piping, power supplies, filters, and pumps needed to run the experiments. The major components of the cell and the cell setup was similar to that at Covofinish, with the following exceptions:

Anodes and Cathodes: At MSC, ten anodes (0.75 in. x 4 in. x 28 in.) were located on either side of one nickel cathode starter sheet (~0.0625 in x 24 in. x 24 in.). The anodes at MSC consisted of two arrays of five nickel slabs. The anodes had been produced by MSC from melt-refined nickel barrier material provided by the K-25 plant. The anodes were cut from a melted (declassified) nickel ingot. These anodes, which contained approximately 250 Bq/g of ^{99}Tc , were located on either side of the cathode .

The electrolyte used in the pilot-scale experiment was a Watt's type electrolyte containing nickel sulfate, nickel chloride, and boric acid. The electrolyte solution was heated to an operating temperature of 140 degrees F. The initial pH of the electrolyte was approximately 4.0. Current supplied to the anodes was adjusted to 160 amps.

The anolyte was circulated continuously through the cationic membrane and then through a cementation unit and a five-micron filter. The catholyte was circulated through a heating chamber and a five-micron filter.

A photo of the pilot-scale cell is shown in Figure 3.0.2.

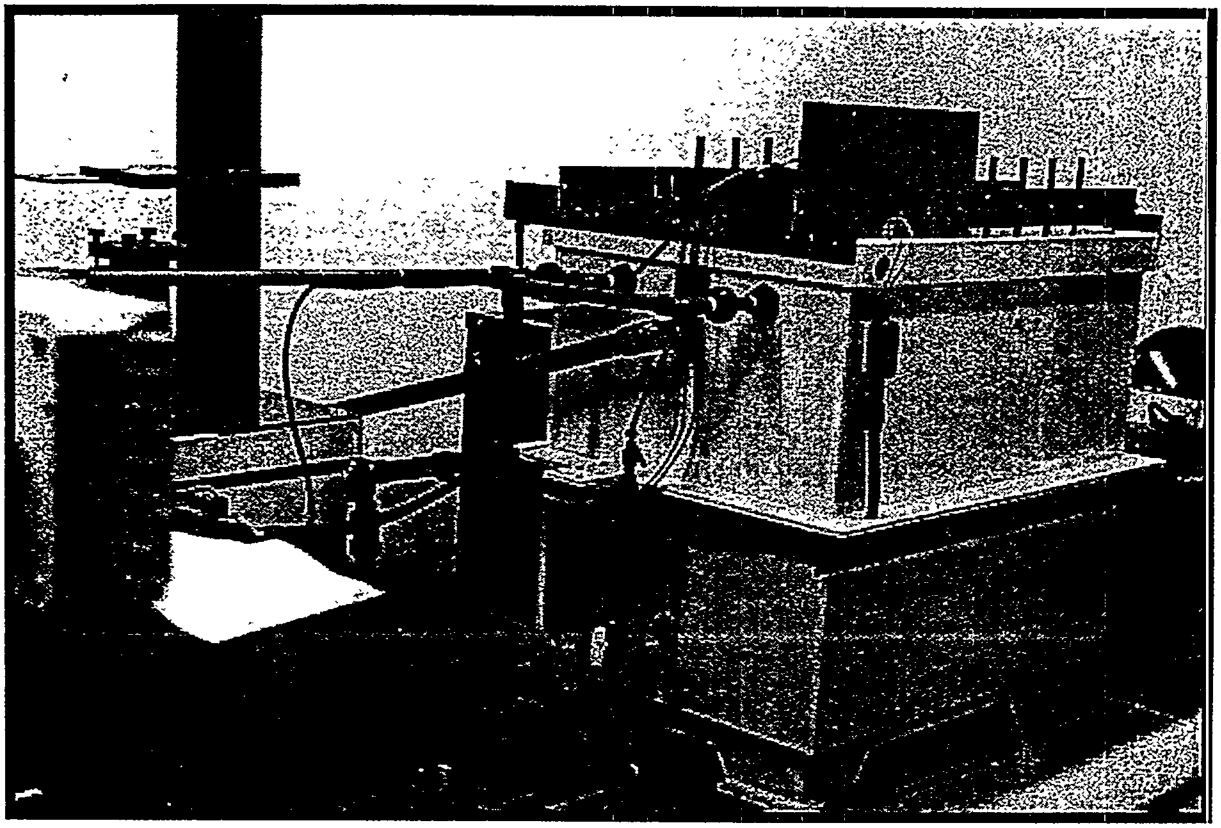


Figure 3.0.2. Photo of pilot-scale electrorefining cell.

Once the pilot-scale cell was operational, Covofinish researchers traveled to the Oak Ridge site to assist MSC personnel in the cell's startup. Like the experiments at Covofinish, the pilot-scale cell was run continuously. Voltage, temperature, and pH were also monitored periodically and recorded. Anolyte and catholyte were sampled daily. Nickel content in the solutions was monitored using Inductively Coupled Plasma (ICP). Nickel cathodes were also monitored daily for quality. Samples of nickel cathode were plated onto small, stainless steel cathode coupons that had been attached to the cell bar and placed in the catholyte. A checklist was developed for daily operations. This included checks of the catholyte drain flow, the pH indicators, filter changeout, data logging, and cell parameter adjustments as needed.

3.0.8 Research Results and Conclusions

The initial test duration was thirteen days from 8/15/96 to 8/27/96. The weight of the starter cathode before operation was 10 lbs.

During the test, the pH of the catholyte was maintained between from 3.6 to 4.8. The anolyte pH ranged from 2.6 and 3.8. Cell current was maintained at 160 amps. At the end of this test, the cathode weight was approximately 125 pounds. The cell was drained and cleaned. Drillings of the cathode were taken and tested using a Packard scintillation counter. In summary, lab analysis indicated a technetium content of less than 1.1 Bq/g.

The second test of the pilot scale electro-refining cell took place from 9/13/96 to 9/30/96 with one cathode change-out on 9/24/96. Since the cell was brought up slowly, full current was not applied until 9/18/96. A metals recovery unit was added to the operation to pull out impurities in the nickel. Again the cell current was maintained at 160 amps. The catholyte pH ranged from 1.8 to 4.8. Anolyte pH ranged from 2.3 to 3.8. Nickel Carbonate was added to the anolyte in an attempt to control the pH on 9/27 with little effect. On the last day of operation, the catholyte pH dropped from 2.7 to 1.8. The decision was made to shut down the cell and inspect the components.

Particulate on the membrane indicated that the membrane needs to be washed off before reuse. This could have caused some interference with nickel ions passing through the membrane. The first cathode from the second test weighed 65 lbs and the second cathode weighed 60 lbs yielding a total of 125 lbs for 12 days or 10.4 lbs/day. Results from cathode samples revealed similar results to the first run with technetium contents around 1 Bq/g.

For more details on methods for determination of isotopic content, see the section titled " Refinement of methods to quantify isotopic elements" in this report and the topical report issued to FETC at the end of Phase II (Advanced Technologies for Decontamination and Conversion of Scrap Metal (9/30/96).

At the end of the testing, a mass balances was documented in order to estimate the material handling and flows required for the full scale cell. A summary of the mass balance is shown in table 3.0.3 below for run 2.

IN	Weight (lbs)	OUT	Weight (lbs)
10 anodes	270	10 spent anodes	157
Cathode 1 starter	10	Cathode 1	65
Cathode 2 starter	10	Cathode 2	60
		Main cell deposits	4.3
		Metals recovery	0.4
		Cementation	0.3
Total	290	Total	287

Table 3.0.3 (Run 2)

Discrepancies in the totals at the bottom of the mass balance are attributed to measurement error and material on the filters, which was not measured. The deposits generated are relatively higher than those anticipated for a full scale unit because iron was introduced into this nickel during the melt as a slag addition. This created deposits that are not anticipated to be a part of a full scale operation.

Challenges during operation of the pilot scale cell included maintaining the optimal pH of the solutions, keeping the 5 micron filters changed to prevent plugging, and keeping the cationic membrane clean to prevent any restriction for the flow of nickel ions from the anode to the cathode. As operation of the cell progressed, these issues were raised and solved resulting in a higher quality product from each test run.

The objectives and goals set for the pilot-scale cell experiments were met and, in some cases, exceeded. Specifically, the pilot-scale research:

- demonstrated that the parameters for the lab-scale electro-refining experiments could be equally as successful in a production environment and
- provided important production data for establishing design criteria and operating parameters needed to develop a full-scale cell.

Knowledge gained and parameters established in the pilot-scale experiments will be applied toward establishing a full-scale electro-refining cell set up at MSC. Experiments with the full-scale cell will further optimize the efficiency, cost-effectiveness, and safety of this system in a production mode at MSC's facilities.

3.1 Full-Scale Electrorefining

In Phase II of this effort, experiments on a lab-scale cell confirmed that technetium could be removed from nickel through electrorefining and cementation processes. A pilot-scale electrorefining system was used to optimize parameters for improved efficiency and to prove applicability on a larger scale. In Phase III, a full-scale electrorefining system was set up to optimize parameters, and specific to electrorefining nickel barrier and to improve system efficiency and cost effectiveness in a production mode. The design, installation and setup, and resulting data from investigations on the full-scale electrorefining cell are discussed in this section.

3.1.1 Developing Design Criteria for the Full-Scale Cell

The criteria for designing a full-scale electrorefining system came from assimilating information from hands-on experience with the 200-gallon, pilot-scale electrorefining cell, and data gathered from industries using electrorefining in a production mode.

3.1.2 Pre-Design Research

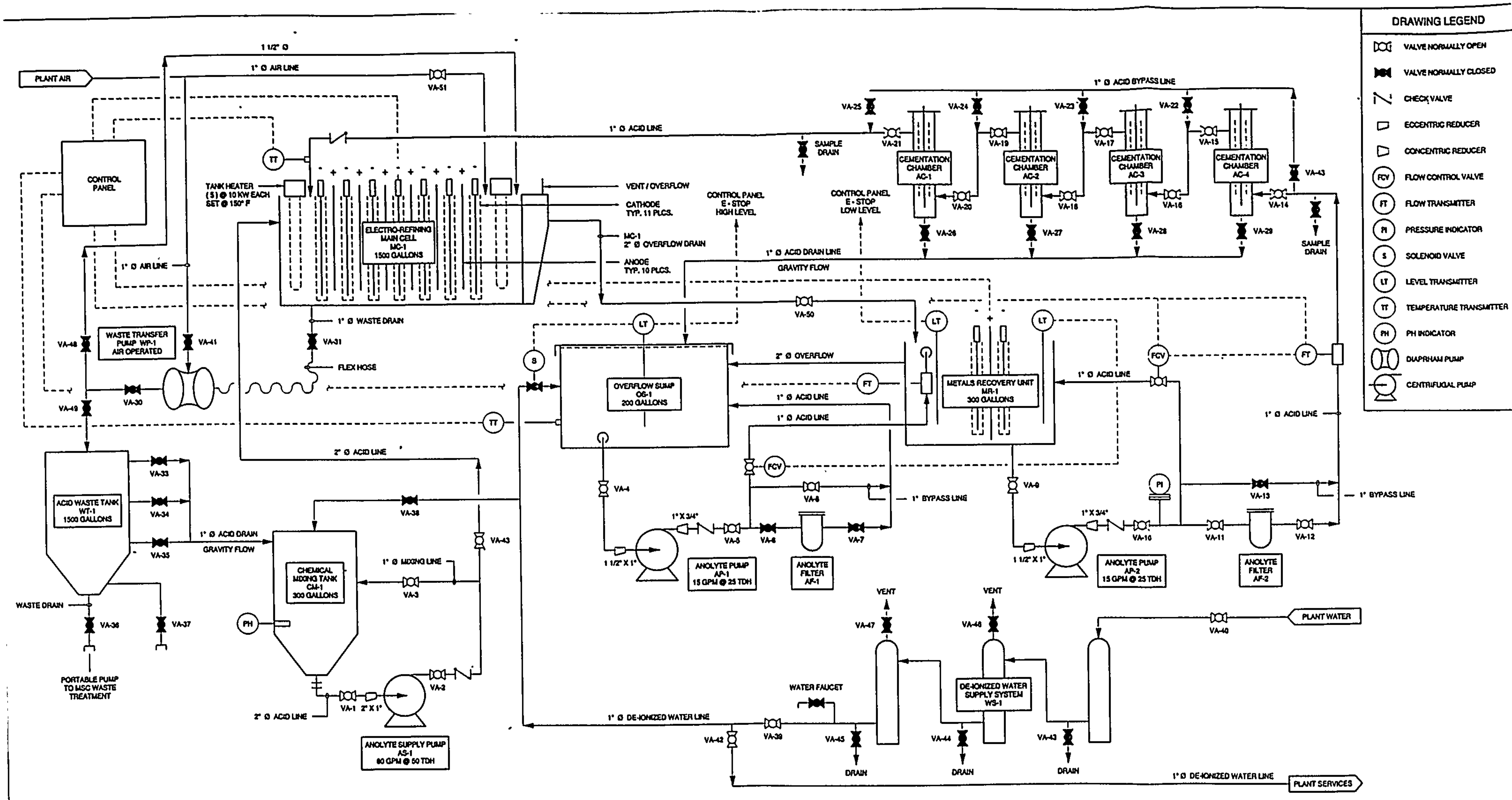
MSC researchers gathered data from various industries that use electrorefining processes in a production mode. MSC personnel traveled to a Phelps Dodge copper refining facility in El Paso, Texas. The purpose of the trip was to gain first-hand production knowledge from an industry with a long history in electrorefining processes.

MSC researchers gained important information relevant to electrorefining such as electrolyte content, electrical distribution, safety issues, material handling, equipment design, and maintenance issues. Although many modifications to basic electro-refining were required to decontaminate the nickel barrier, the trip produced a significant number of usable applications and important considerations, that were incorporated into the design of the full-scale cell at MSC.

One MSC engineer attended a course in Extractive Metallurgy to gain insight into electro-refining as practiced by industry. As part of the course, the class toured several electro-refining facilities including electro-winning and electro-refining operations. This helped to optimize the full scale cell design.

3.1.3 Full-Scale Electrorefining Cell Design

The conceptual design work for the proposed full-scale electrorefining cell began in the fall of 1996 and continued through mid-1997. The full-scale cell was designed to be able to decontaminate up to 160 pounds of nickel per day. Tank size, anode and cathode size and number, current density, and electrochemicals were all considered in the conceptual design. A cementation unit and a metal impurities recovery unit were also incorporated in the design. The process flow is shown in Figure 3.1.1. MSC engineers projected that the full-scale unit would consist of ten anodes and eleven cathodes, each approximately 38-inches by 38-inches by 2 inches. Equipment parameters, process parameters, and process flow considerations were updated regularly to reflect new information gained from the scale up, from advice of nationally recognized metallurgical experts, and from MSC's own pilot-scale experiments.



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REFERENCE DRAWINGS

APPROVALS	
ENGINEERING	DATE
OPERATIONS	DATE
QUALITY	DATE
CLIENT	DATE

CAD FILE E \ENGINEERING\NICKEL RECLAMATION\APPROVED\D751013 FETC ELECTRO-REFINING P&ID REV B DWG
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CUT 1.5 PITCH THREAD RELIEF WITH ROUND NOSE TOOL ON MACHINE CUT THREADS.
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REMOVE BURRS WELD SPLATTER & LOOSE SCALE.

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MANUFACTURING FACILITY
FETC ELECTRO-REFINING
PROCESS AND INSTRUMENTATION DIAGRAM

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In phase III, MSC engineering staff began designing MSC's full-scale electrorefining cell. Lessons learned by the visit to electrorefining production plants and various experiments (including test melts) by MSC scientists, engineers, and production personnel were included in a functional design specification.

3.1.4 Major Components of Full-Scale Cell

The full-scale cell design included the following major components:

Main Tank: The full-scale cell was a 2,000 gallon tank made of polymer concrete. The eleven cathodes and ten anodes were installed in the tank, alternating anode and cathode. A membrane frame was used to support a cationic membrane surrounding each cathode. The cationic membrane separated the anolyte (contaminated solution around the anode) from the catholyte (clean solution around the cathode). The cell was equipped with a push/ pull ventilation system. Ventilation volume consisted of a 6,000 CFM inline scrubber connected to the existing building HEPA filtration system. Power distribution at the cell included a copper bus running down each side along the top of the tank (lengthwise). One buss connected to anodes to provide a positive electrical charge. The buss on the opposite side connected the cathodes to provide a negative electrical charge. The buss on both sides connected to the power supply via water cooled leads.

Metal Impurities Recovery Unit (MRU): Solution exiting the main cell entered the MRU for impurities removal. The MRU included one anode and two internal cathodes. The cathode in the MRU was surrounded by a frame that supported a cationic membrane on both sides. The MRU cell was equipped (on the top of the tank) with a vent that connected it to the same scrubber that ventilated the main cell. After filtration, anolyte entered the top corner of the MRU cell tank and exited at the bottom middle.

Electrolyte Solution: The solution used in the full-scale cell was a Watts-type electrolyte containing nickel sulfate, nickel chloride, and boric acid at a pH level of around 3.0. The solution was mixed in a 300-gallon tank and transferred to the main cell.

Temperature Controls: The electrolyte was heated using five immersion heaters located inside the main cell. The temperature of the electrolyte (both in the main cell and in the heated sump) was monitored by a sensor and displayed on the operators' control panel. The temperature control was set between 145 and 155 degrees F. The electronic controls provided an automatic shut down of the process if the temperature of the electrolyte exceeded 170 degrees F. The temperature of the electrolyte at the metals recovery unit (MRU) was monitored and read from the MRU display.

Power Supplies: Current from the power supply to the main cell, the cementation unit, the metal recovery unit, and the polarity reversing unit, was monitored and controlled from the central operator control panel. Electricity was provided to the ten anodes at a current density of 15-20 amps/ ft^2. The cell was run at 1800 to 3600 amps at about 6 volts.

Cementation Chambers: Cementation chambers, for removal of technetium from solution, were made of polypropylene. Four chambers were arranged in series. The bottom of the chamber was flanged to allow cleanout of sludge as needed, and the top was flanged for accessibility. Each chamber was approximately 8 inches in diameter and 54 inches high. Electrolyte passed from the bottom port, filled the chamber, and exited through the top port.

Overflow Sump: The overflow anolyte from the main cell was originally gravity fed into the 200-gallon, chemical-resistant sump tank. The overflow sump was located under the main electrorefining cell. A level indicator in the sump controlled the outlet pump, turning it off if levels got too low and turning off both anolyte pumps and the makeup water if levels got too high. The overflow from the main cell was redesigned. The overflow sump is now fed by an overflow from the MRU.

Catholyte Recirculation Pump : A portable catholyte recirculation pump was located between the main cell and the catholyte filter. The pump could be turned on manually as needed when the catholyte began to appear cloudy or when particulate was visible. The portable pump would circulate the catholyte from inside any one of the eleven cathode frames (cell chamber), through the catholyte filter, and then return the catholyte to the same chamber. The pump could be used in any of the catholyte chambers.

Anolyte Filters: An anolyte recirculation pump located behind the sump, delivered anolyte from the sump through the first anolyte filter and into the MRU. A second anolyte pump pulled anolyte from the MRU, through the second filter, then through the cementation process and back into the main cell to complete the loop. Both pumps circulated anolyte at 5 gpm and ran continuously, except when shut down by the operators.

Deionized Water Supply System: Deionized water was used during startup for chemical additions and mixing and was also used to make up for the evaporative losses during cell operation. A sensor in the sump signaled low fluid levels. The signal was sent to a solenoid on the deionized water supply system, allowing flow. The makeup water was supplied from the MSC plant water supply and passed through ion-exchange columns to provide deionized water to the system.

Anode/Cathode Rack: The portable anode/cathode racks were made available to allow for rinsing chemicals off the anodes or cathodes as they were removed from the main cell. A pressure washer was used to clean the anodes or cathodes after removal.

Hoist: A one-ton hoist and a steel frame was used for material handling above the electrorefining cell. A spreader beam was specifically designed to enable operators lift one anode or cathode at a time.

Compressed Air Supply: Compressed air was used to aerate the anolyte in the main cell. A pipe was permanently installed in the main cell that extended to the bottom of the anolyte and ran the length of the tank. Compressed air was filtered and "de-oiled" (made oil free) before use in the cell. A manual valve was available to vary the flow.

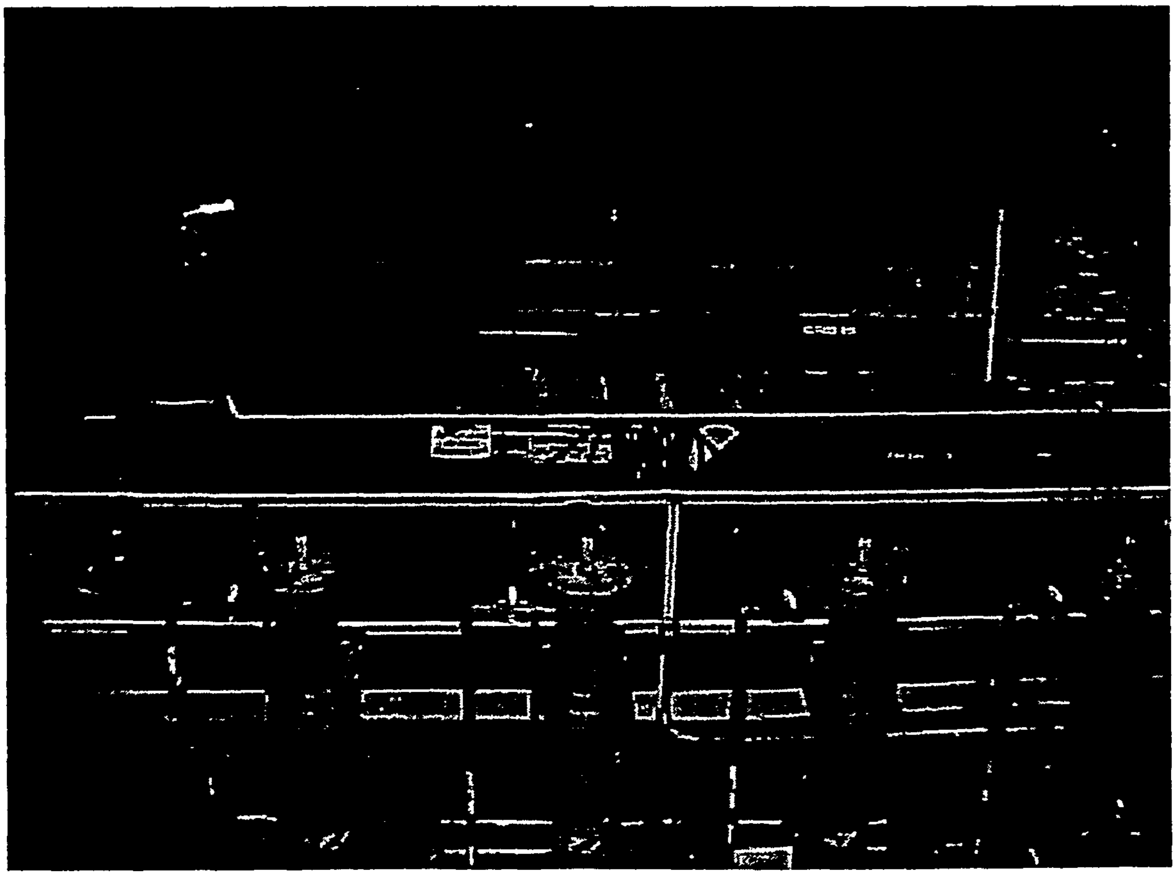
pH Monitor: The pH (in both the anolyte and the catholyte) was monitored in the MRU using a continuous monitor. PH in the main cell was monitored as needed using a portable pH probe. Samples of anolyte and catholyte were taken daily during operation and sent to the lab for compositional analysis and pH.

Ventilation System: Fumes from the main cell and from the MRU were scrubbed by an intermediate scrubber system. The intermediate system scrubbed the acid in the air stream before the air stream reached the building's HEPA ventilation system, protecting the HEPA system from chemical attack inherent in the electrorefining processes.

Piping, Pumps, and Tanks: Piping, pumps, tanks, etc. were constructed of materials suitable for safe containment of the chemicals used in the system.

Phone Dialer: A phone dialer was purchased to automatically contact the operators in case of problems with the system. This allowed continuous 24 hour, 7-days-per-week operation of the cell.

Figure 3.1.2 Photo of Electrorefining cell:



3.1.5 Electrolyte Solution

A Watts-nickel electrolyte solution was used in the nickel electrorefining process. Specifications (concentrations and purities) for the chemical components needed for MSC's specific processes were obtained from specialists in the field. Vendor product specifications were evaluated and costs and solubility data were compared.

Initial optimization efforts included minimizing the presence of any elements that could interfere with the electrorefining process. This was achieved by purchasing chemicals of the highest purity. Reports on the research and analysis of the optimized Watts solution are provided in Appendix 3.1.

Initial plans were to mix acid in a separate mixing tank. The acid temperature could not be maintained in the mixing tank. As a result, acid was mixed in the main electrorefining cell. For this, nickel sulfate, nickel chloride, and boric acid were purchased in solid form and added to the heated cell. As procedures for acid mixing were optimized, it was discovered that procuring nickel sulfate and nickel chloride in liquid form, mixing in small batches, and then transferring to the main cell proved the most efficient method. The MSC mixing plan for Watts nickel electrolyte is shown in appendix 3.1.

3.1.6 Potential Hazards and Safeguards

Safeguards were implemented to address potential hazards inherent in the electrorefining processes. Potential hazards were identified in the following major areas; electrical, power lifting equipment, thermal, and chemical (solids and fumes). Work instructions were prepared for all job duties associated in these (and other) process areas. Employees were given extensive training on the safe operation of all components and machinery associated with their job descriptions as it related to work in the electrorefining production area. Examples of the hazards are shown below are for the one full scale electrorefining cell.

Electrical	Maximum 15 volts D.C., 3600 amps
Chemical/ burns	Heated nickel sulfate, nickel chloride, boric acid. PPE required as well as restricted access to the top of the cell
Fumes	Mainly water vapor, potential for chemical fume, however, results from samples taken by independent source revealed no fumes from cell that are potentially harmful to people. Ventilation system implemented as a precaution
Material handling	Potential for cuts, drops, etc. PPE required. Special handling devices were designed and implemented.

3.1.7 Testing and Commissioning the Electrorefining System

Once the components of the electrorefining system were installed, the integrated system was made ready for testing and commissioning. A functional test and commissioning plan was written for final testing of the entire system before startup. The plan (which incorporated industrial and ALARA safety requirements) proposed to functionally test individual components, subsystems, and the integrated electrorefining process. Production and maintenance employee work instructions were developed for all phases of the electrorefining processes.

Mechanical and electrical controls for individual components of the full-scale electrorefining system (including peripheral units) were tested. Tested components also included individual flow valves, controllers and instrumentation, piping and ductwork, and power supplies. Subsystems, including the chemical mixing system, the ventilation system and scrubbers, the de-ionized water system, the overflow sump, the cementation system, and the monorail system were also tested. Test activities included comparing the drawings and sketches from the functional design to the actual installed system components.

3.1.8 Electrical Design Inspection

Lockwood Green Technologies (LGT) performed a walkdown of the full-scale electrorefining system to observe and inspect the installation for conformance to LGT drawing E001. Finding no major problems with the installation of the electrical devices, LGT inspectors verified that the workmanship was professional and was (with minor discrepancies) in compliance with all licensing codes. Discrepancies noted by LGT personnel were corrected by MSC. LGT also provided engineering support in accordance with the previously approved QA program.

3.1.9 Testing of Integrated System

The integrated electrorefining system was tested under conditions that would be similar to those expected in the electrorefining production processes. The full system test used salt water as a substitute for electrolyte to verify fluid flow and sensor indicators.

A function check was made of the integrated system's monitoring of flow systems, temperature, current, and voltage, pH, and level alarms. Testing with the initial optimized parameters were used as the standards for verifying overall operation of the system as specified in the functional design. Discrepancies found during the system test were red-lined or noted. A punch list was made to ensure that corrections were incorporated in the operations plan.

A safety review was also performed to ensure that the system was safe to operate, based on information obtained during commissioning and functional testing.

3.1.10 Nickel Source for Full-Scale Electrorefining

MSC requested 4,500 pounds of contaminated nickel barrier from the ETTP site to use for testing MSC's full-scale electrorefining cell. Researchers were unable to complete the transfer of this material due to costs to transfer the nickel into fiber (sacrificial) drum and transport the nickel to MSC.

MSC was able to obtain nine contaminated nickel ingots from Paducah, however, with a total weight of approximately nine tons (18,000 pounds). This nickel already had been previously melted during the Cascade Improvement Program/Cascade Upgrade Program (CIP/CUP) from the late 1970's and early 1980's. Full scale testing of electro-refining was conducted on this nickel. For the full-scale cell tests, these nickel ingots were sliced into anode sheets, mounted to hangers, and placed directly into the electrorefining cell. In addition, the cut ingots were used for test melts.

3.1.11 Operations

The full scale cell was started for the first time in June 1998. After one day of operation, the cell was shut down due to cationic membranes sticking to the cathodes, mandating a re-design of the cathode frames. During the month of July, as the cathode frames were under design changes, several tests were carried out on the cell to improve plating quality. Trials were carried out in the cell on a single strip plate. Plating was improved from a "flaking" appearance to a smooth plated product. Parameters such as current, current reversal, and temperature were modified to achieve the desired result. Optimizing parameters to reduce "dendrite growths" on the surface of the cathode was also a subject of testing during this time. The making of a nickel starter sheet by plating on a titanium surface was accomplished during this time. During July, a new prototype membrane frame was placed in the full scale cell. Trials on this new frame and membrane indicated good performance of the assembly. Once proven, additional frames were ordered. In September, the new cathode boxes were installed and tested. The nickel cell ran successfully from Oct. 6 to Oct. 15 when the cell was shut down for cathode change-out. A demonstration for FETC was held on October 13 at MSC.

3.1.12 Lessons Learned

Several challenges were experienced and resolved during the testing and the process demonstration.

<u>Safety</u>: In October 1998, during a shutdown for cathode changeout, a chemical scald injured an electro-refining cell operator. The cell was shut down immediately and the accident investigated. A corrective actions list was developed and implemented. This included adding an additional platform for improved access to the top of the cell. A new instruction was developed and extensive training took place. The Radiation and Industrial Safety Work Permit was reviewed and rewritten. The cell was restarted on 2/18/99.

Membrane Frames: After the June 1998 startup of the full-scale cell, MSC experienced difficulties with membrane materials sticking to the cathodes. The cell ran for only one day. MSC researchers tried various techniques to solve the problem, including adding inflated tubes on the side to stretch (tighten) the membrane and adding stiffeners to the membrane frames. After several unsuccessful attempts to modify the frames, MSC researchers concluded that new, wider membrane frames would be required. A new membrane frame was designed by MSC. One new membrane frame was purchased as a prototype and tested before a purchase order was released for enough membrane frames to fill the cell.

When the new frames, Nafion™ membranes, gaskets, and bolts arrived for the full-scale cell, the cathode boxes were assembled. A few of the PVC bolts broke during assembly and were replaced with coated stainless steel bolts. The new membrane and frame assembly was placed in the full-scale cell. Trials were run at various settings to further optimize plating. The new frame and membrane appeared to function well.

Nickel Plating Quality: While waiting for the new frames, trials continued in the electrorefining cell on a single strip plate. The goal was to optimize the electrodeposition appearance and to set electrorefining parameters. These trials produced continuously-improved results, moving from a plated metal coupon with a "corn-flake" appearance to a plated thin foil on a titanium surface. The thin foil was then peeled off and used as a nickel starter sheet. More nickel was plated onto the starter sheet, producing a strip with uniform deposition on the surface of better quality than was accomplished on the pilot scale.

Difficulties With Flow Controllers: MSC also experienced difficulties with the flow controllers. The controllers lacked the flexibility needed to control the flow in the system. In addition, electrical interference caused the transmitters to send incorrect signals to the controllers. The transmitters were relocated to avoid interference.

Pump failures: Two centrifugal pumps (with mechanical seals) in the acid recirculating loop failed. It was determined that cycling the cell through heating and cooling have caused crystals to form in the pumps causing failure. The start/ stop is inherent in a development process. In a continuous process, this effect would be minimized. Magnetic drive centrifugal pumps were specified, procured, installed and tested. Also, any time there is an interruption to the system that causes cooling of the anolyte, pumps are flushed before shut down to prevent buildup of crystals inside the pump. As of this writing, the new pumps are continuing to operate successfully.

Decontamination of the Nickel: The first tests did not decontaminate the nickel to levels to reach our goal (~1 Bq/ g). Researchers concluded this was probably caused by 1) some of the anolyte splashing over the membrane frame, causing contamination of the catholyte (the new frames remedied this situation); and 2) the cationic membrane that was purchased appeared to be of inferior quality to that used on the pilot-scale cell. MSC purchased Nafion 450 membrane materials to place in the new frames. This also improved decontamination. Over time, decontamination levels improved dramatically.

3.1.13 Results

Chart 3.1.5 shows the contamination levels in the Paducah Ingots before electrorefining. Technetium contamination ranged from 226 Bq/g to 627 Bq/g.

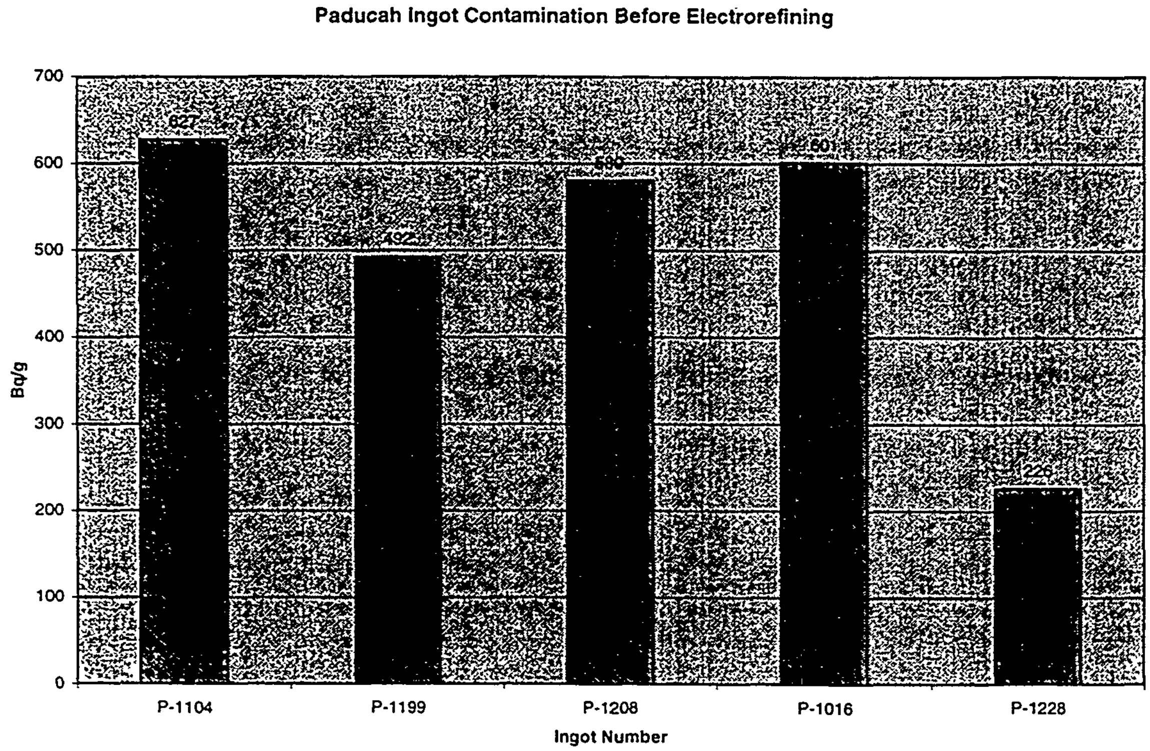


Figure 3.1.5 Paducah Ingot Contamination Levels

After implementation of corrective actions and optimization, the cell was re-started on 2/18/99. First, starter sheets were made by plating out nickel onto a Titanium sheet. After approximately 24 hours, the titanium sheets are pulled from the cell and the nickel starter sheet is "stripped" or separated from the titanium sheet. See photo 3.1.6.

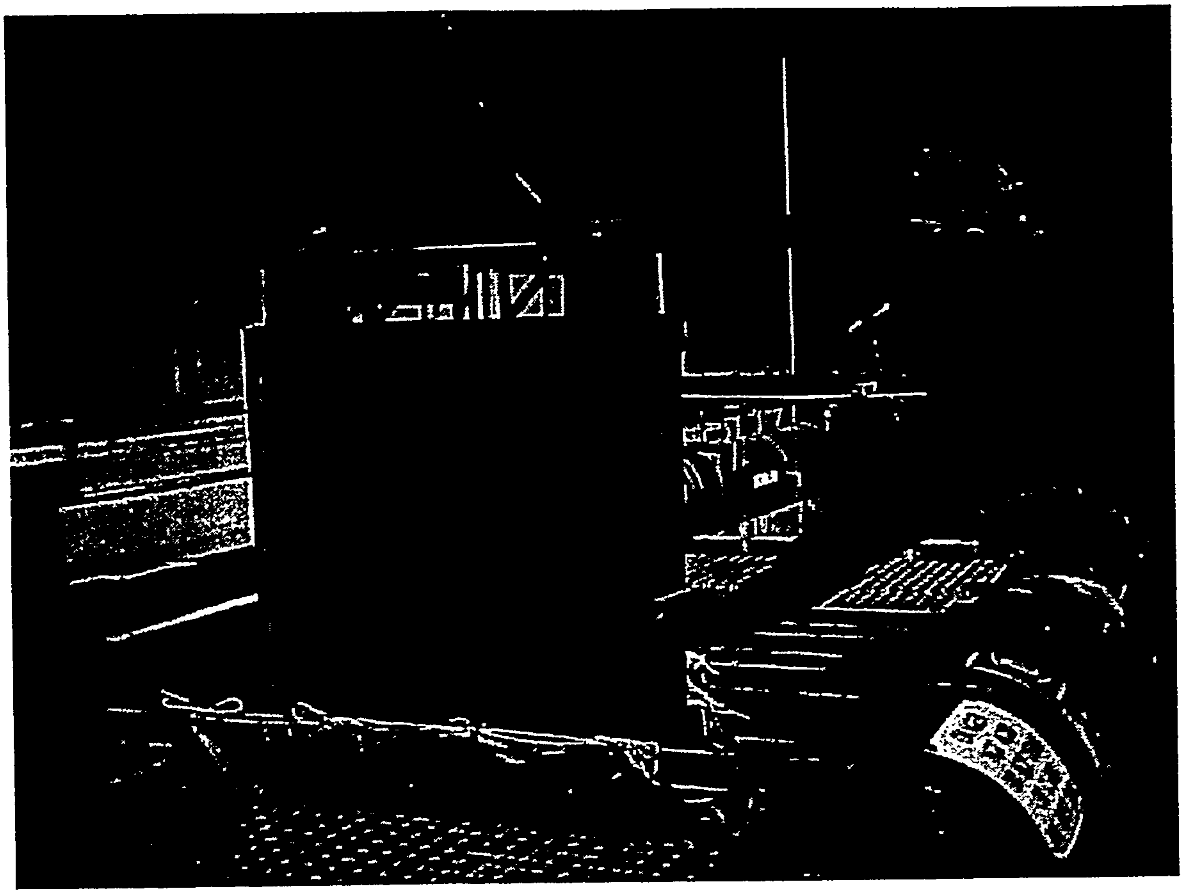


Figure 3.1.6

After the nickel starter sheet is stripped from the titanium sheet, it is placed back into the electrorefining cell to make a nickel cathode. Approximately 14 lbs per day of nickel is plated onto each cathode.

Samples of anolyte and catholyte were taken daily to track levels of technetium in the solutions. These levels are indicators of potential contamination reaching the final product cathode. Figure 3.1.7 shows technetium levels in the anolyte and catholyte over time. Anodes that were left in the cell, which dissolved and contributed contamination to the solution, explains the upward trend of contamination in the anolyte. Also, adjustments were necessary to the anolyte purification circuit before contamination levels were dropped to acceptable levels. This graph demonstrates the learning curve necessary to optimize operating parameters. The catholyte trends were added to review the effect of the cationic membrane. This graph shows that the cationic membrane is an effective barrier between the anolyte and catholyte.

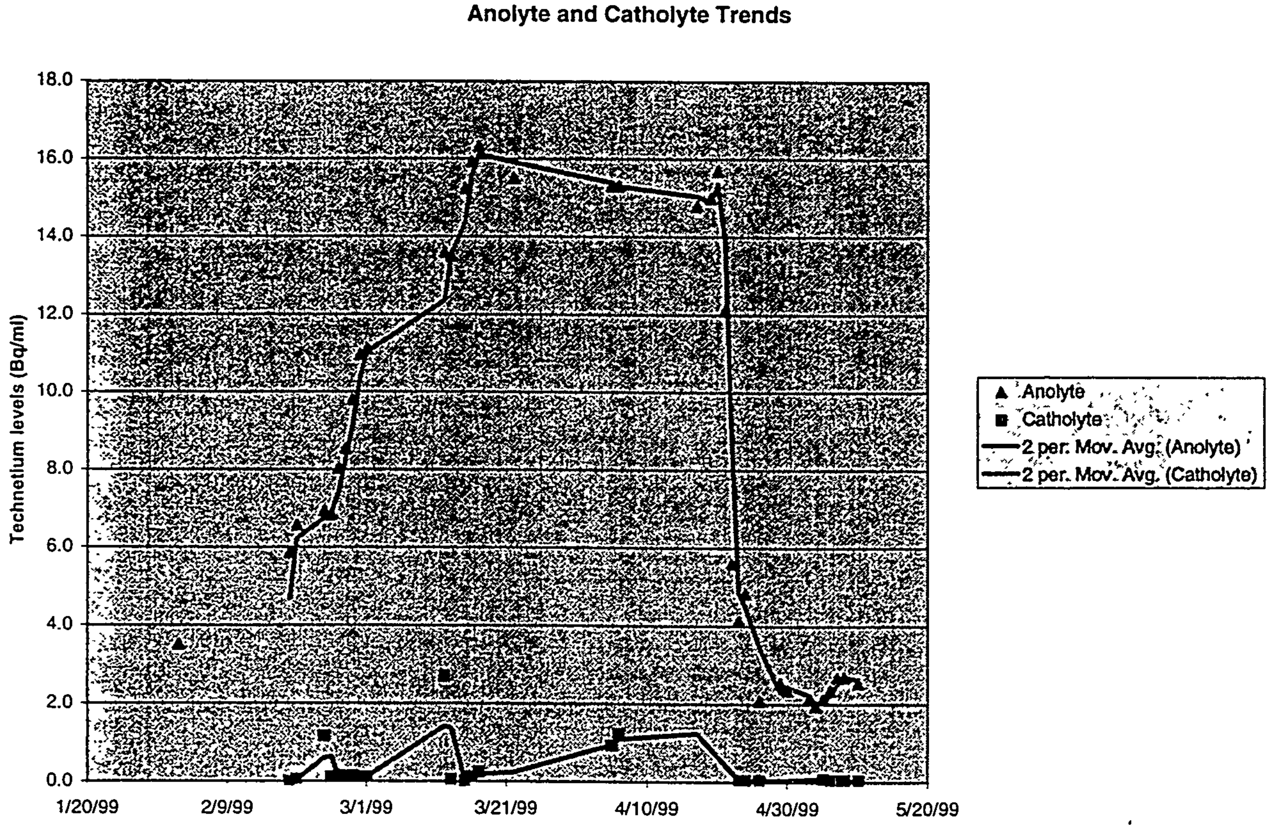


Figure 3.1.7

The technetium levels in the cathode product were also tracked over time as shown in Figure 3.1.8. Again, as parameters were optimized, cathode results were above goal. Once the system was brought under control, results in the range of 1-2 Bq/g were achieved.

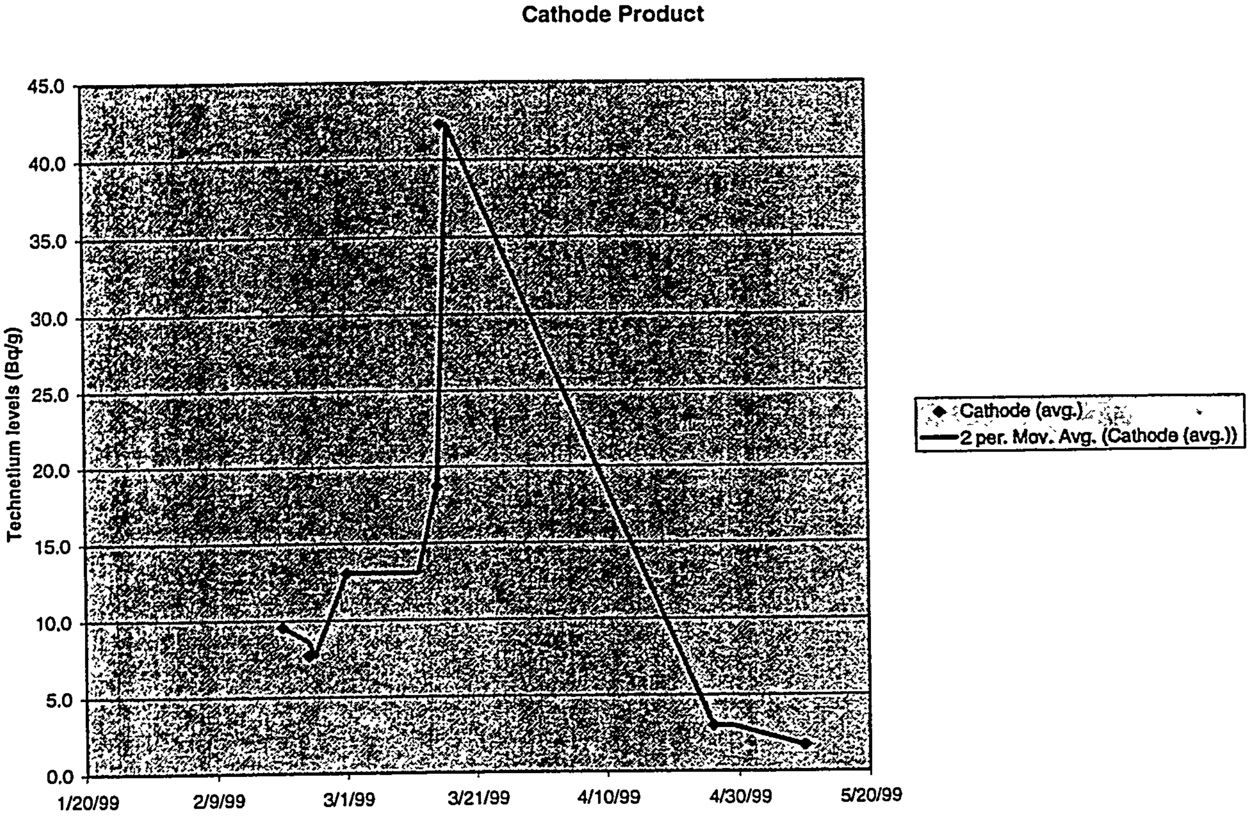


Figure 3.1.8

It was found that tracking anolyte technetium contamination levels is a good indicator of final nickel cathode values. As shown in figure 3.1.9, even though the anolyte levels are tracked in Bq/ml, and values for the cathode are in Bq/g, a correlation between the two are evident. Tracking and controlling anolyte contamination is imperative for acceptable product nickel.

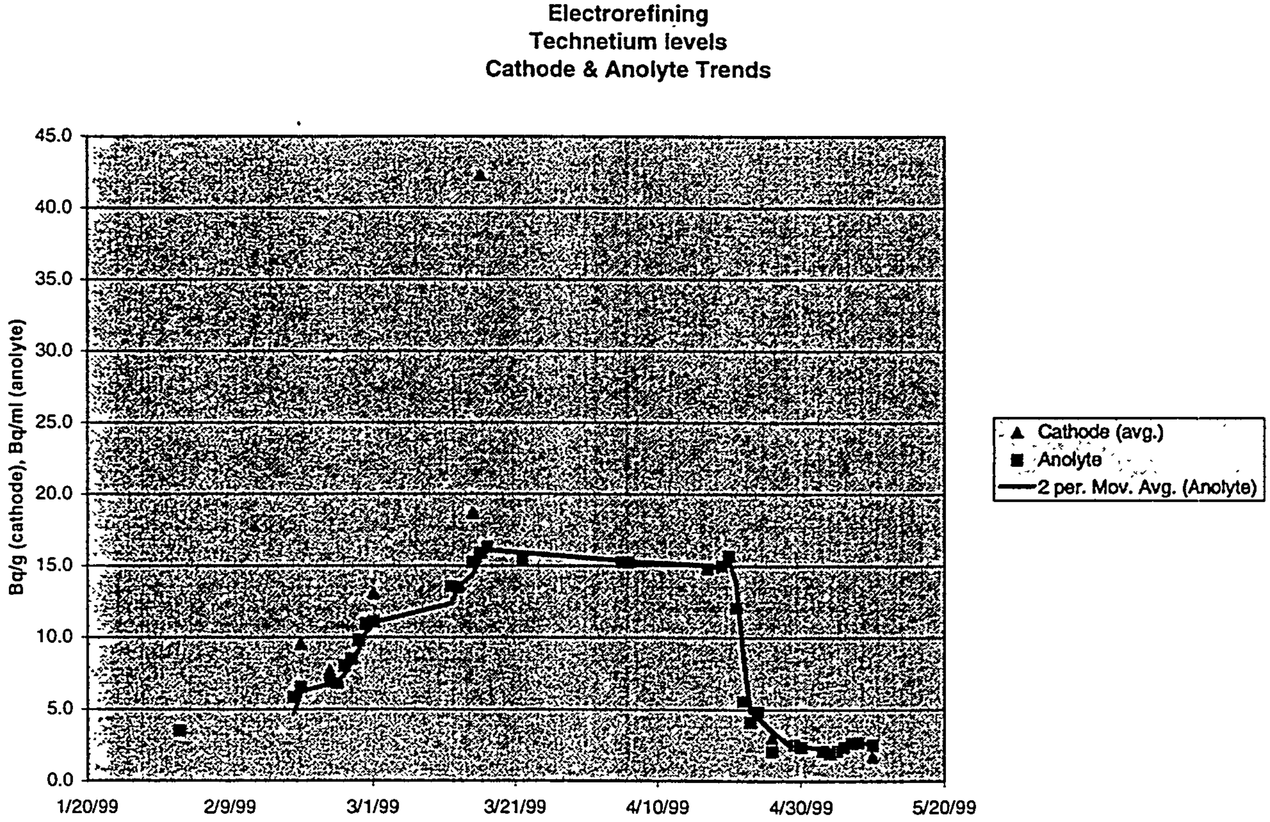


Figure 3.1.9

Latest results as of May 1999:

Contaminated anode	592 Bq/g
Product cathode	1-2 Bq/g
Cementation chamber (where ^{99}Tc is removed)	2315 Bq/g

Figure 3.1.10

Once process parameters were stabilized and optimized, the full-scale electrorefining system worked as anticipated. The cementation units revealed high levels of technetium as expected. Nickel was decontaminated by the full scale cell between 1 and 2 Bq/g which meets our stated goal of decontamination around 1 Bq/g.

As of May 20, 1999, over one ton of barrier nickel was processed through the electrorefining cell. Over one month of actual plating time was logged.

3.1.14 Phase III nickel recovery research costs

Task 311, One full scale electrorefining Cell
Task 312, Anode baskets (direct dissolution)
Task 313, Chemical dissolution

TASKS 311, 312, 313 COST SUMMARY

DATE	TASK #	ENGINEERING LABOR+OH	PRODUCTION LABOR+OH	OTHER SUBCONTRACTOR COST	OTHER DIRECT COST	TOTAL COSTS
1997	311	$ 46,546.79	$ 189,559.79	$ 4,281.71	$ 151,219.66	$ 415,679.56
	312	$ 8,596.24	-	$ 23,165.91	$ 248.74	$ 35,635.47
	313	$ 228.05	$ 190.44	-	-	$ 418.49
TOTAL		$ 55,371.07	$ 189,750.23	$ 27,447.62	$ 151,468.40	$ 451,733.52
1998	311	$ 46,609.10	$ 73,648.54	$ 8,843.12	$ 124,643.55	$ 274,408.05
	312	$ 3,787.69	-	-	$ 94.00	$ 3,896.25
	313	-	-	$ 39,459.26	$ 17,686.24	$ 65,991.62
TOTAL		$ 50,396.80	$ 73,648.54	$ 48,302.38	$ 142,423.79	$ 344,295.92
1999	311	-	$ 9,702.28	-	$ 22,033.48	$ 35,146.55
	312	-	-	-	$ -	-
	313	-	-	-	$ -	-
TOTAL		-	$ 9,702.28	-	$ 22,033.48	$ 35,146.55
GRAND TOTAL		$ 105,767.87	$ 273,101.05	$ 75,750.00	$ 315,925.67	$ 831,175.99

Additional cost detail for the full scale cell is shown in appendix 3.1.

3.1.15 Projections based on one full scale cell:

The following data provides an economic estimate for a scaled up version of the electro-refining cell that was built under a FETC contract and is currently operating at MSC. Economies of scale can be expected from a large plant. However, the estimates for this evaluation are based on equipment, material, and personnel costs associated with the full-scale cell. The assumed economy of scale for this evaluation is a range of 10 to 40 percent. The estimates are given as ranges, based on uncertainty. In determining economic viability of deploying this or any new technology, requires risk-based decision making. The technology has proven to work and in order to determine if it is economical, assumptions have to be made about how a large-scale plant will be built and operated and how the price of nickel will fluctuate over the life of the project. MSC and its Parent Corporation BNFL, INC. performed this risk analysis early before the full-scale project was completed or the technology proven and based on the results entered into a fixed price contract with the DOE to D&D three buildings at the ETTP site. The contract recognized the value of recovering the nickel and the DOE has received a credit for this material. The following is a quote from the DOE/ORO office web page:

"The $238 M contract cost included a credit back to DOE of $55,569,748 for the recyclable material. This amounts to quarterly cost savings of $2,646,178 over 21 quarters for the materials recycled or reused. The recycling activities began in the 4th calendar year quarter of 1998 and will continue throughout the duration of the contract."

Assumptions:
Cost projections are based solely on the performance of the one full scale research cell
These numbers are not based on any operational experience of a production scale electrorefining plant
Costs from the one full scale cell are for electrorefining only and do not include costs for pre-processing nickel
Preprocessing could include pretreatment and melting to delassify nickel and cast into anodes

Electrorefining
Costs

One cell cell totals:

Equipment for one full scale cell (~15' long) cost ~$316,000
Production rate 160 lbs/day

A possible plant size:

20 cells ~30' long, or 40 cells ~15' long
Production rate ~9,000 lbs/day

Projections:

Scaling equipment costs from one cell to a plant size, expected savings as a result of:
- Bulk discount
- Production facility instead of research
- Shared acid regenerative loop between cells
- Shared waste treatment facilities between cells

Could result in equipment costs savings of 10-40% for a potential plant equipment costs of:
40 x $316,000 x (1-(10% to 40%)) = ~$7,600,000 to ~$11,400,000

Operation of the pilot scale cell (technician only) for one year = ~$120,000
Same discounts as assumed above
Estimate savings 10 to 40%
$120,000 (1- (10% to 40%) x 40 cells = ~$2,800,000/ yr to ~$4,300,000/yr

Pre-processing of nickel could cost in the range of $0.75 to $1.50/lb
This assumes existing vacuum induction equipment at MSC can be used for melting and casting

3.1.16 Path Forward

The path forward includes the design and installation of a production scale electrorefining facility in support of the ETTP Three Building D&D Project. This facility, as currently planned, will be located at MSC in Oak Ridge and will have an initial capacity of about 1200 tons/yr. As of this writing, the plant is in final design.

3.1.15 Nickel Melting

For ETTP nickel, melting of nickel would be required to declassify the nickel and prepare anodes for electrorefining. Developing an anode casting process required researching a mold design suitable to produce a shape that would fit directly into the electrorefining cell. Selecting mold materials that could sustain the superheat during casting was part of the design process as well. Developing casting methods included optimizing melting parameters, such as temperature, residual gasses refractory containment, and metal flow.

Designing Molds

Anode molds were sized to create anodes that fit directly into the tank on the full-scale cell. The anode molds were made of ductile iron. Graphite dams were installed at the ends of each mold to provide a fill cavity of 36-in. by 36-in. by 2-in. thick.

Various means of casting molds in a production mode were investigated. One promising option under consideration is to fit the molds into a mold chamber by stacking them in a spiral-staircase configuration in MSC's six metric ton vacuum induction degassing and pouring furnace. Casting and cascading would occur by pouring the molten metal into the top mold and allowing the superheated liquid to cascade down to the bottom molds, as indicated in Figure 3.1.6. Other casting methods, such as casting molds one at a time in MSC's one ton vacuum induction furnace, were also investigated.

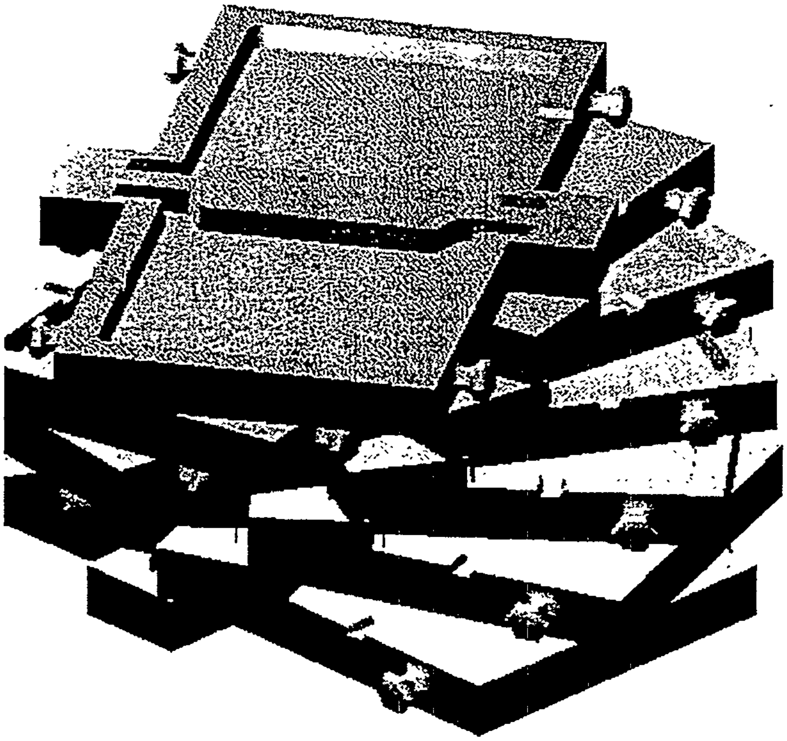


Figure 3.1.6. Spiral stacking of molds for production mode, cascade casting.

Effects of Fluorine on Refractories

In order to produce anodes for electrorefining that mimic commercial electrorefining practices, melting and casting of diffusion plant nickel is required. The life of the oxide refractory used to normally contain molten nickel in a melting process is in question. The presence of fluorine compounds (e.g. (UO_2F_2 and NiF_2) causes corrosive attack to oxide refractories.

Frequent relining would be a major expense in materials and labor. Moreover, the used refractories would constitute a significant volume of radioactive waste that would incur burial costs.

To avoid corrosive attack by fluorine, MSC researchers recommended a replacement of the oxide refractory system with graphite. Melting nickel in graphite would require that the melts take place in vacuum or inert atmosphere. In addition, the molten nickel would become saturated in carbon to about 0.6 weight percent. The effects of high carbon nickel on the casting process and on the anode quality was uncertain. MSC researchers had to devise a practical method to melt nickel in a graphite system. The basic question was, could carbon saturated nickel be cast into acceptable anodes.

To answer that issue, researchers performed 500 pound test melts of nickel in a graphite crucible in the vacuum furnace available in MSC's plant (see figure 3.1.7). After the first casting run several findings occurred. Molten nickel dissolved the graphite crucible sufficient to saturate the metal with carbon. After casting the nickel showed evidence of high gas loading during solidification, as seen by the appearance of a "boiled" surface.

The crucible dissolution was deemed unacceptable. The conclusion was carbon must be present in a free form, and available for dissolution when nickel becomes molten, otherwise the crucible will be attacked.

Small amounts of aluminum were added to stop the evolution of gasses, suspected to be CO, during solidification.

Several more 500 pound test melts were performed attempting to solve the aforementioned issues. To provide free carbon when nickel became molten remained a difficulty. Efforts to apply a sacrificial graphite mortar to the hot face of the graphite crucible were unsuccessful. The crucible was dissolved below the mortar depth even though excess was provided. The aluminum addition coupled with a time/ temperature hold to allow for degassing was successful in stopping gas evolution during solidification.

To date, the high carbon nickel anode has not been used in the cell to determine if there are detrimental effects to the operation. Parameters in the electrorefining cell have been kept as consistent as possible to assure repeatable results. It was deemed prudent not to introduce a high carbon anode into the cell as of this writing

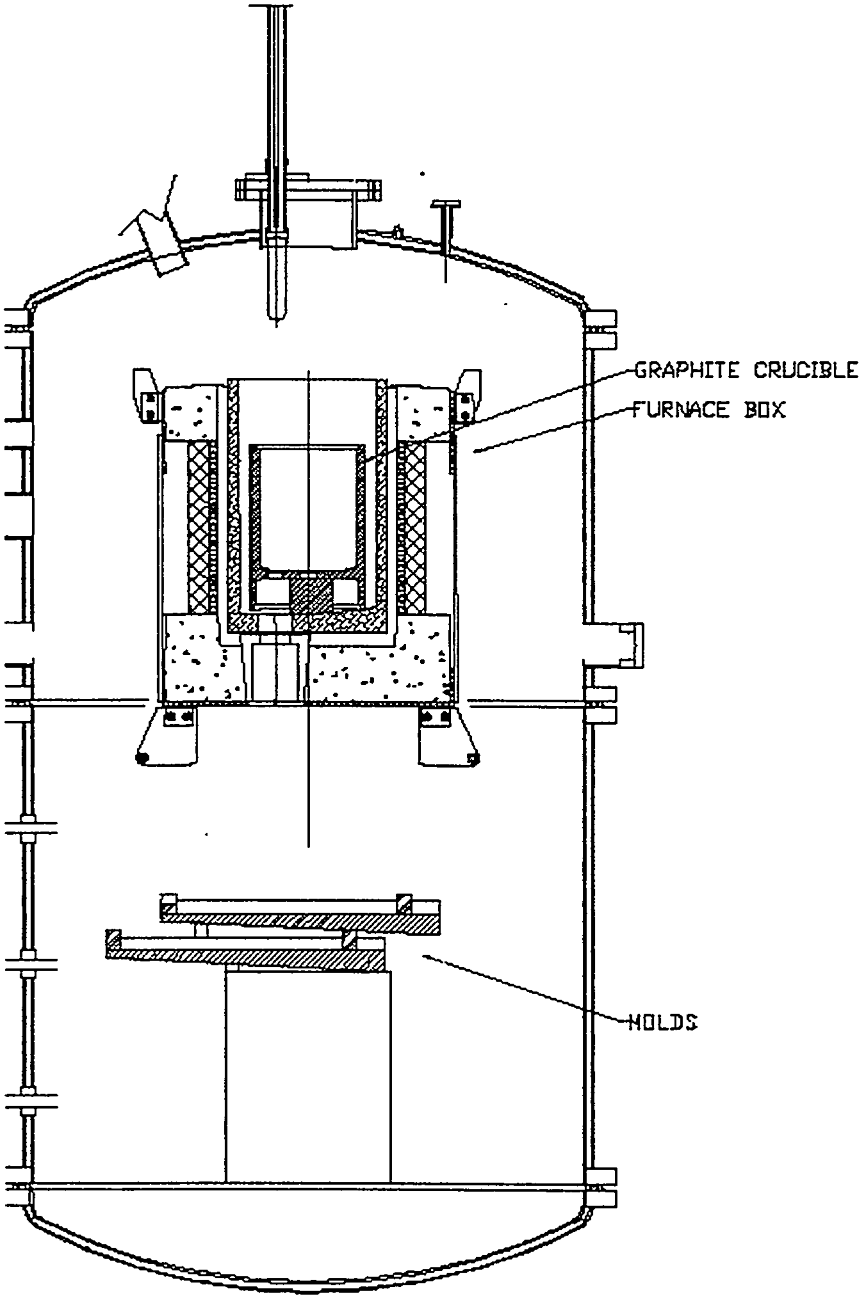


Figure 3.1.7 VIM furnace and crucible setup

Results of literature had indicated carbon to be inimical to nickel casting. Therefore, MSC scientists consulted with several experts in the metallurgical field to determine the specific effects of carbon and its compounds on nickel castings. The overall conclusion from the experts consulted indicated that carbon should be tolerable in the electrorefining cell.

The tolerance of carbon in the cast nickel anodes was further tested in subsequent preliminary melts under multiple variables. Lab analyses showed promising results, but further testing on high-carbon castings is on hold as MSC researchers are currently considering options to remove the fluorine before melting.

3.1.16 Nickel Direct Dissolution

Research by the Colorado School of Mines (CSM)

During Phase III of this research effort, CSM researchers (1) investigated direct dissolution and chemical dissolution as alternatives to melt refining and (2) characterized emissions inherent in the nickel electrorefining processes.

Experiments were performed on nickel electrowinning and nickel electroplating under various conditions and test setups.

One major expense in electrorefining nickel barrier was tied to the melt process. Fluorine compounds, (e.g. (UO_2F_2 and NiF_2), present in the nickel diffusion barrier are now known to attack the oxides in refractory materials and significantly shorten the expected refractory life. In an effort to reduce or eliminate the cost associated with melting nickel, CSM and MSC researchers looked for alternatives to melting. Two options CSM researchers examined were electrowinning (chemical dissolution) and electroplating (direct dissolution). Chemical dissolution involved placing the barrier material into an acid tank, allowing it to dissolve, and then electrorefining the solution to recover the nickel. Direct dissolution involved placing the nickel directly in an electrochemical solution, electro-dissolving the nickel into solution and then electrowinning the nickel.

CSM researchers set up experiments for both options using clean nickel of various sizes and geometries. Both options proved to be successful with dissolving the nickel anode, by direct dissolution being the most promising. Details of CSM's experiments and their research results are provided in Appendix 3.1.

CSM researchers also captured and tested fumes emanating from the surface of the electrorefining cell to determine the types of off-gas and the volume of fumes that could be expected from operating a full-scale cell. The data was used to establish the size and capacity of the ventilation systems that would be needed for a full-scale cell.

Direct Dissolution Experiments on Nickel Barrier

After the successful results of CSMs electrowinning and electroplating experiments, MSC personnel repeated the electroplating (direct dissolution) experiments using actual contaminated nickel barrier in a 100 ml lab-scale cell. MSC researchers had received security clearances and were able to perform these experiments at the ETTP site using classified, contaminated nickel barrier. The purpose of the test was to perform direct dissolution to show nickel can be electro-dissolved and plated. The membrane, used for decontamination purposes was not included in these tests.

The direct dissolution method was chosen because it was faster and more effective than chemical dissolution for dissolving the nickel and plating it out on a cathode.

These lab scale tests found direct dissolution to be a promising method for nickel decontamination. Further, scaled up versions of the experiments would be required to provide a detailed economic assessment of the process and to determine its ability to decontaminate nickel. While initially, direct dissolution promised to be economically attractive, such issues as possible increased process waste, effects on the cationic membrane, and security concerns would have to be addressed. Further design would be required to determine modifications required to the existing cell design to accommodate direct dissolution.

3.2 Full-Scale Laser Cutting Demonstration

3.2.1 Background and Overview

Radioactive scrap metals (RSMs) are received at Manufacturing Sciences Corporation (MSC) for decontamination and recycle where economically feasible. Incoming materials are sorted, and a conveyor moves most of the steel scrap to a tumble grit-blasting unit for mechanical decontamination. A chemical decontamination facility is available for cleaning soft metals such as copper, aluminum, and nickel.

One of the challenges of decontaminating scrap metal is reducing the large pieces to a size that can be accommodated by MSC's automated processing equipment. If, for example, a piece of metal exceeds 2 ft. or is more than 3/4 in. thick, then it must be reduced to a manageable size using either cold-cutting tools, such as band saws or pipe cutters, or (more typically) thermal processes, such as plasma or other oxy-fueled torches. There are several disadvantages to each of these size reduction methods:

1. Manual cold cutting tools and thermal technologies are both very time consuming for setting up as well as executing the cutting operations.
2. Thermal technologies produce fumes and excessive heat. Also, the kerf (the cut width) of conventional thermal technologies is wide, increasing the amount of waste.
3. Workers must wear personal protective equipment, including respirators, to manually perform thermal cutting operations. This is especially unpleasant in environments where the thermal cutting technologies have produced high ambient temperatures.

Automating the cutting processes could increase productivity and reduce worker exposure. Until recently, however, automation has been limited because of the wide diversity in the size, shape, and composition of incoming RSM materials and because the technological advances of cutting tools were too limited to efficiently handle an automated process.

Both of the above limitations were dispelled in a project opportunity made available to MSC. This opportunity is part of the three-building decontamination and decomissioning efforts at the Oak Ridge Gaseous Diffusion Plant (ETTP). There is a large quantity of radioactively contaminated process components, which are of similar geometry and material composition. This large volume of metal makes automating the size-reduction processes both practical and prudent.

To investigate the potential for automation using laser technologies, MSC planned and executed a full-scale laser cutting demonstration using an off-the-shelf, Nd:YAG 2000-Watt laser with fiber-optic beam delivery and a remotely controlled robotic manipulator. The laser-cutting demonstration was designed to meet specific research objectives:

1. To compare, in a full-scale operation, the cutting performance of the laser to the cutting performance of existing technologies,
2. To evaluate the potential for using lasers to automate size-reducing activities when materials are both geometrically and compositionally similar, and
3. To optimize parameters for automated cutting operations in a production mode.

3.2.2 Research Approach

In preparation for the laser-cutting demonstration, MSC researchers began gathering information on cutting operations for oxy-propane and plasma torches. Values were compared on a number of variables, including ventilation (filter clogging issues), cut speeds, secondary waste, ease of operation, and quality of cut. Researchers also gathered information on remote operations, fixturing, material handling, laser safety and containment, personnel training requirements, and special needs for radiological environments. The information gained established the criteria used to identify needed components for an automated laser cutting system and divide the research activities into achievable and manageable steps.

The laser cutting demonstration was divided into four steps. First, the cutting capabilities of a commercially available Nd:YAG laser system were established by initial lab testing performed by the Edison Welding Institute (EWI). Test results determined the parameters to be used in subsequent demonstrations. Second, the Energy Technology Engineering Center (ETEC) in California used the Nd:YAG laser to demonstrate its size reducing capabilities on reactor fuel storage tubes. The laser was then shipped to MSC, along with data and lessons learned from the EWI testing and the ETEC demonstrations.

Third, MSC performed a follow-on, full-scale laser cutting demonstration where the remote laser cutting operations were more fully automated. Finally, a subsequent series of laboratory bench-scale tests were performed to enhance the laser cutting data base in areas identified by the demonstrations.

3.2.3 Pretesting

Initial lab testing of the Nd:YAG laser was performed by the Edison Welding Institute (EWI) under contract to ETEC. The purpose of the pretesting was to ensure that the Nd:YAG could adequately cut the materials projected to be used in the demonstration at MSC.

EWI researchers used a continuous-wave (nonpulsed) 3-kW Nd:YAG laser with a 15-m-long fiberoptic beam delivery system. The laser was able to successfully cut a 1-cm-thick steel pipe over a laser power range of 1000 to 2700 watts. In an investigation involving 41 cuts, EWI researchers experimented with a number of variables to determine their effect on kerf (cut width). These variables included beam power, laser end-effector focal length, assist gas type and pressure, and cutting speeds.

EWI Researchers found that a smaller kerf minimized secondary waste but a wider kerf helped keep molten dross from bridging the cut and rewelding it. Researchers also found that the primary parameters controlling the quality of the cuts were the type and pressure of the assist gas. (Assist gas is the gas that flows across the work area, blowing away from the cutting zone the debris caused by the cutting process.) Researchers also determined that oxygen was the optimum assist gas because it increased the burn and the cutting efficiency of the laser.

The full report of the EWI research is provided as Appendix 3.2.

3.2.4 ETEC Demonstration

ETEC investigated the capabilities of the fiberoptic beam delivery system using remote control technologies. ETEC researchers identified two laser systems as promising candidates for remote cutting applications—the currently available Nd:YAG (neodymium-doped yttrium aluminum garnet) laser (as used in the MSC demonstration) and the COIL (chemical oxygen iodine laser), which offers higher-power laser output.

Using information from the initial EWI lab tests, ETEC performed several bench-scale tests, followed by two deployment demonstrations to evaluate the effectiveness of the available Nd:YAG lasers for size reducing RSM.

The laser resonator, power supply, water chiller, gas assist supply, and operational controls were located in a nonhazardous environment for protection and maintenance accessibility. The laser focusing head and a tube holding fixture were located in a radiologically controlled containment tent that had been fitted with an HEPA-filtered ventilation system. The laser beam was delivered to the focusing head through a 15-m-long fiber-optic cable. ETEC was able to successfully use the 2 kW Nd:YAG laser to size reduce (for disposal) approximately 300, contaminated fuel storage tubes that were 5-in diam by 118-in long.

Each storage tube was positioned horizontally on a fixture. Two longitudinal cuts were made by moving the laser head lengthwise along the tube. One circumferential cut was made by rotating the tube. Each tube was thus sectioned into four approximately equal sections.

A continuous wave laser with a power level of 0.8 kW was used for cutting these thin-walled (1/16"-thick) tubes. Oxygen was used as the assist gas. At a linear cutting speed of about 400 cm/min, the laser was able to size reduce each tube in about 2.5 minutes. This included the time it took to manually load each tube and unload the tube sections. (Only the laser cutting was automated for this demonstration).

ETEC researchers performed a number of additional test cuts using a plasma-arc torch to develop comparative data on laser cutting versus cutting with current technologies. The plasma-arc tests used the identical geometry and cutting procedure that had been used with the laser. Both the laser and the plasma-arc systems had comparable cutting speeds, but the plasma-arc system required greater maintenance. Researchers noted, however, that there were costs savings with the laser cutting system over the plasma arc system because of reduced secondary waste and lower maintenance requirements. Nevertheless, the maintenance requirements for the laser system were significant. One example was multiple filter changeouts caused by higher levels of airborne debris generated by the laser cutting operation.

Additional cost estimates for size reducing much larger quantities of contaminated tubes were also provided by ETEC researchers. Those estimates included the capital equipment costs of the cutting hardware (which was markedly higher for the laser system) and also assumed replacement of the current filter system with self-cleaning filters. Because of capital equipment costs, plasma arc cutting was less expensive for smaller quantities of tubing while laser cutting was less expensive for larger quantities. The break-even point in ETEC's comparisons was about 230,000 tubes. These estimates, however, included manual handling of the tubes rather than robotically controlled automation in a full-scale cutting process.

ETEC researchers concluded that the use of the laser system resulted in significant reductions in secondary waste generation and personnel exposure. While not demonstrated in the task because of the manual tube handling, exposure to personnel was reduced as a by product of the laser's lower maintenance requirements and the system's capability for remote cutting operations.

ETEC researchers further test operated the Nd:YAG laser at their facility before shipping it to MSC; and they preset the laser at the optimum parameters for ensuring a successful demonstration. ETEC also transferred all data and lessons learned to MSC for use in their automated, full-scale laser cutting demonstration. The full ETEC report is provided in Appendix 3.2.

3.2.5 System Components for MSC's Demonstration

Prior to receiving the laser equipment from ETEC, MSC had prepared a functional design specification that covered component acquisition, setup and installation of equipment, and remote control requirements for an integrated and automated system. The major components for the laser demonstration, as identified in the functional design, include the following:

K-25 Converter Shell: A No. 2 converter shell made of 1/2-inch thick nickel plated carbon steel was selected from the K-25 site as the object to be cut during the full-scale laser cutting demonstration. The No. 2 converter shell (Figure 3.1) is representative of the hundreds of similarly shaped and sized converter shells that await removal from the K-25 site. Also, the shell's size (6 ft. in diameter x 12 ft. long) was small enough to be shipped to MSC in one piece. Since the shell had been used only for cooling, it's design was unclassified; therefore, no special handling was required for security purposes. A nondestructive analysis (NDA) was performed on the shell to verify that it could be shipped on MSC's license.

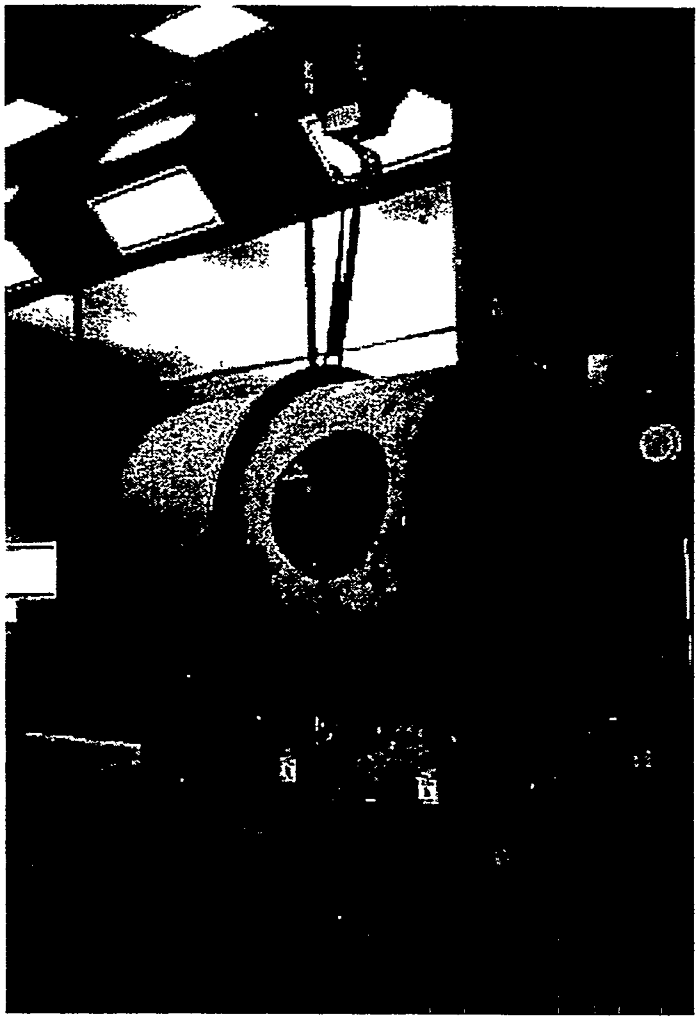


FIgure 3.2.1 No. 2 Converter Shell from ETTP on turning cart.

<u>NdYAG Laser</u>: DOE located a Lumonics Nd:YAG laser and arranged for it to be made available for the laser-cutting demonstrations. The 2000 Watt multiwave laser, which was capable of cutting up to 1/2-inch carbon steel, included a power supply, a chiller, and a fiber optic cable. The fiber-optic cable transported the beam from the power supply to the laser focusing head.

<u>Auto-Focus Cutting Head (AFCH)</u>: The Lumonics AFCH is a non-contact, capacitive clearance measuring end-effector designed for use in robotic cutting applications. In the demonstration, the AFCH was driven by a high-speed DC servo motor. Its copper nozzle tip served as a sensing electrode. Because it could compensate for part and program deviations, the AFCH was used to maintain a distance of 2 mm from the demonstration shell for cutting.

Robotic Manipulator: Both new and used robots, as well as welding manipulators, were evaluated for use in the laser-cutting demonstration. The manipulator selected was a pre-owned Cincinnati Milacron robot, as illustrated in Figure 3.2.2. (A similar robot had been used to size-reduce RSM at the Capenhurst site in the United Kingdom.) The robotic manipulator controlled the cutting head of the laser. The Cincinnati Milacron robot was capable of holding the AFCH to within 1 inch of the surface to be cut and was able to perform a smooth cutting operation. The manipulator also had adequate horizontal and vertical reach to accommodate the 5 ft. in diameter x 12 ft. long shell.

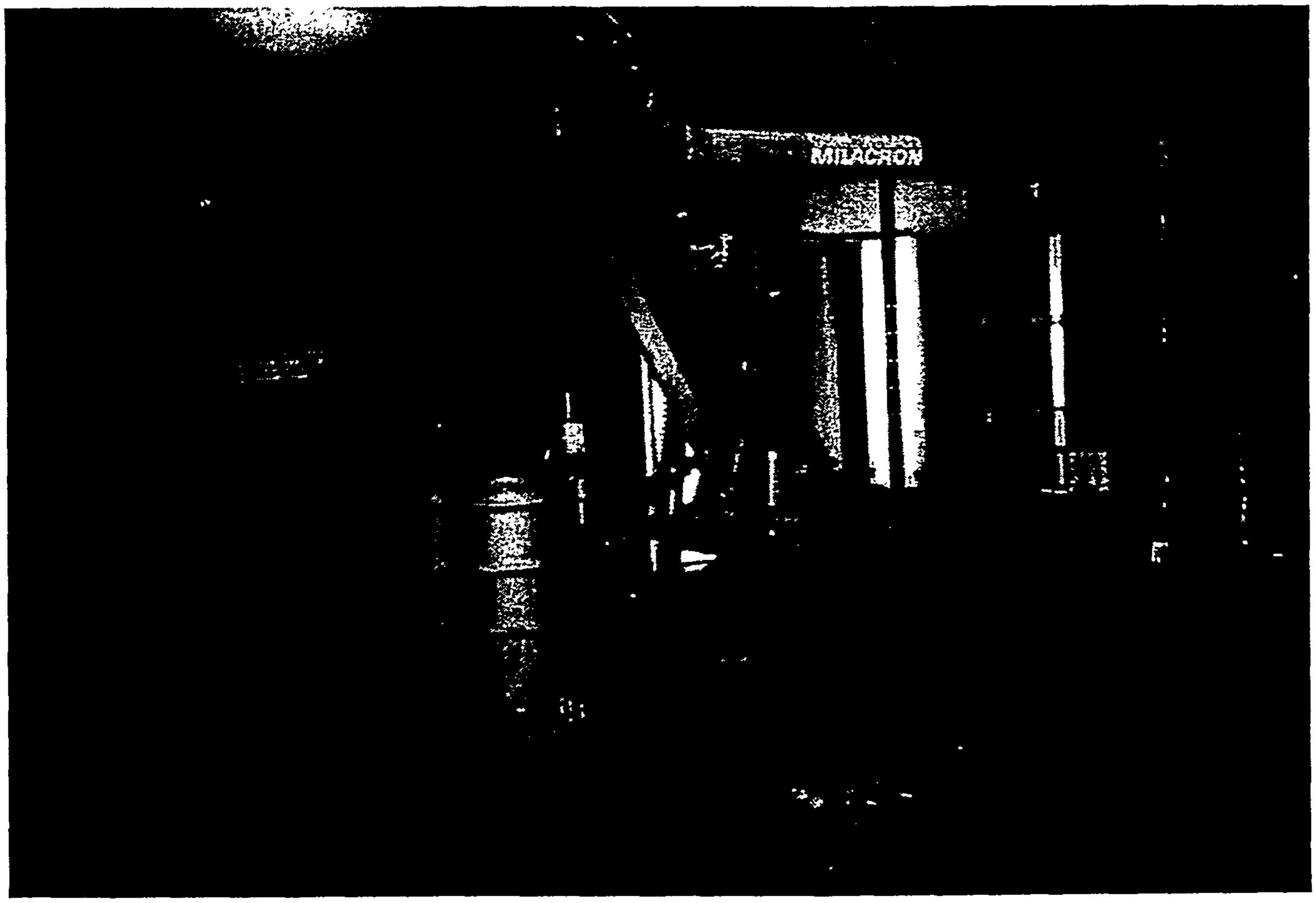


Figure 3.2.2 Cincinnati Milacron robotic manipulator.

Indexing Cart and Turning Rollers: An indexing cart with turning rollers was used to rotate the converter shell in the horizontal plane during cutting operations. (The cart and rollers can be seen in place in Figure 3.2.1)

Light-Proof Room: A prefabricated lightproof room was purchased to house the laser, shell, manipulator, and other equipment needed for the full-scale laser cutting demonstration. The room was equipped with protective windows to allow operators and visitors to safely view the cutting operation without being exposed to hazards from the laser or the robotic manipulator.

Ventilation System: A high efficiency, facility ventilation system was installed in the lightproof room to control airborne contaminants generated by the laser-cutting process.

Programmable Logic Controller (PLC): Each component of the automated cutting system was equipped with independent controls. An Allen Bradley SLL500 system was put in place to interface between the major components and to take control when all components were placed in a "ready" state. Designed with a touch screen and keypad, the PLC provided the operator with overall system control via selected analog variables and digital logic. For the demonstration, independent control of each system was referred to as "manual mode" while the PLC system control was referred to as "automatic mode." Position feedback was accomplished using a regenerative DC drive with tach speed feedback and an encoder mounted on the turning rolls. The equipment allowed the operators to control the speed for a specified distance as indicated by feedback from the encoder. The PLC control system provided the following functions:

- An *alarm monitoring function* alerted operating personnel of abnormal conditions and, if necessary, halted operations in a controlled manner.
- A *control step sequencing* function performed startup, operation, and shutdown routines.
- An *analog and digital control function* provided the necessary interface between the six components to coordinate the equipment operation as one system.
- An *operator interface*, a backlit LCD screen with touch screen capabilities and keypad, allowed the operator to view the control system operation.

Operator's Control Booth: Operations were controllable from a booth that was separated from the laser and other components in the lightproof room. A video camera was mounted in the cutting room such that an operator in the booth could view the cutting operations from a monitor.

3.2.6 System Configuration

A decision matrix was used to compare several layout options for the laser cutting demonstration. Various methods of rotating the converter shell were evaluated to optimize the productivity of the robotic manipulator and the laser.

The converter shell, laser, robotic manipulator, turntable with indexing rollers, waste box, and monorail (along with all associated hardware and power supplies) were housed in the lightproof room. Electrical hardware was purchased to connect the major components and integrate them through the PLC. Minor peripheral equipment needed to perform a successful demonstration were also installed. Water pipes, for example, were installed between the laser and the chiller to provide a continuous cooling supply to the laser.

A cooling block was used on the AFCH to help dissipate heat generated by the laser process and the drive unit on the AFCH. The AFCH was also equipped with oxygen as an assist gas to promote the cutting effect. Nitrogen was used to prevent dirt or oil from forming on the focus lens. Finally, the AFCH was equipped with three crash-detection switches, plus one temperature switch.

Final component configuration was established by MSC engineering, with representatives from the Industrial Systems Design Corporation. Industrial System Design experts provided programming assistance. Representatives from Lockwood Greene Technologies ensured component integration. Final wiring and PLC programming and debugging were completed, hardware was cold tested, and preliminary demonstrations were performed on other materials by MSC's technical staff.

3.2.7 Safety Features

Laser safety was incorporated into every step of the planning process, and a number of safety features were installed in preparation for the laser-cutting demonstration. The lightproof room, which provided total enclosure of the laser cutting operation, had over its viewports UV protection as approved by the Laser Institute of America (LIA) and in accordance with ANSI Z136.1 1993. Installed in the lightproof room (at the room doorway) were interlocks that automatically turned off the laser in case the door to the room were opened. Warning signs and placards were installed to indicate when the laser was in use. A blue warning light was installed to alert personnel in the area that a laser is in use.

The operator's station was completely removed from the work area in the lightproof room to protect workers from exposure to the radioactivity in the RSM, to the laser beam, and to keep them from being injured by the automatically operated robotic manipulator.

3.2.8 Personnel Training

All MSC personnel who were to be involved in either operation, maintenance, or management of the laser-cutting activities received comprehensive training in the operation of remote equipment and in laser safety.

3.2.9 Equipment and Operations Training

Production personnel were fully trained to operate both the laser and the robotic manipulator. After instructional training, the operators began practicing on individual components in the automated system. Initial cutting with the laser was performed manually (for training purposes) without the benefit of the programming that would later automate the system. Practice cuts were made on stainless steel and carbon steel materials—both of which were thinner than the ½-inch carbon steel used for the converter shells.

Operators also received a week of in-house training provided by the vender of the Cincinnati Milacron Robotic Manipulator. As part of this training, operators practiced controlling the robot for cutting at various movements and at differing speeds. In addition, they spent time testing and evaluating methods of rotating the shell during the cutting operations.

3.2.10 Laser Safety Training

MSC identified and trained a Laser Safety Officer (LSO) and a deputy LSO to oversee the safe operation of the laser-cutting demonstration. These officers were given the responsibility and authority to monitor for potential laser hazards and to enforce appropriate controls to assure safe laser operations. The LSO trained operating personnel on laser safety using guidelines from the *American National Standard for Safe Use of Lasers*, American National Standards Institute (ANSI Z136.1-1993) and the *Laser Safety Guide*, 9th edition, prepared by the Laser Safety Committee of the LIA.

The American National Standards material included visual presentations that incorporated key components of the ANSI Z136 laser safety standards. Student workbook (included with the training materials) provided practice questions and exercises to test the trainee's understanding of the materials being presented in each series. The workbook also served as a desk reference.

A number of topics were covered in the laser training which encompassed the safe operation of low, medium, and high-powered lasers and the use of high-powered lasers in manufacturing and laboratory environments. Additional equipment-specific training on the safe use of the Nd:YAG laser was provided by Lumonics representatives.

3.2.11 Set Up For MSC's Demonstration

The Nd:YAG laser and Cincinnati Milacron robotic manipulator were set up in the lightproof room inside MSC's manufacturing facility. The Lumonics Auto-Focus Cutting Head was mounted on the working arm of the robotic manipulator. This special laser head, which delivered the laser beam via the fiber optic cable, provided a means of effectively cutting the converter shell and tracking the surface to compensate for deviations, either in the part itself or in the program. The AFCH enabled the cutting beam to maintain a 1 mm distance from the cutting surface at all times.

The robotic manipulator was positioned with the laser end effector always pointed downward so that the laser beam would not be directed toward the observation ports of the lightproof room.

The converter shell was placed on a cart with automatic indexing rollers that rotated the shell 360 degrees on the horizontal axis.

A PLC was used to control the path of the laser and the size of the piece to be cut. The PLC was also interlocked to the doors and the robot to stop the laser if any set perimeters were out of the desired range.

The 25-ton chiller (shipped with the laser) was located outside the containment facility (the lightproof room). The chiller was housed in a portable out building to protect it from the weather. Pipes connecting the chiller water to the laser were located along the wall and were insulated for maximum cooling efficiency.

A laser expert had been employed as a consultant to assist in the installation and operation of this project. The expert had also instructed MSC personnel on the operation and safeguards of laser operations.

3.2.12 MSC Laser-Cutting Demonstration

The full-scale laser cutting demonstration consisted of three different operations: (1) part handling, (2) laser cutting using a robotic manipulator, and (3) scrap removal.

Part Handling

A track mounted cart supporting the converter shell could be independently moved in and out of the work area using remote control. For the demonstration, the converter shell was placed on the cart mounted indexing rollers and moved into the lightproof room.

Laser Cutting Using Robotic Manipulator

Laser power, assist gas, and cutting speeds were optimized during previous cutting trials on stainless steel, carbon steel of varying thicknesses, and aluminum.

Laser cutting head motions were directed by the PLC, which allowed the operator to move the robotic arm over the full converter shell surface. Using the robotic arm to move the cutting head, operators made axial cuts along the converter shell. Circumferential cuts were made by rotating the converter shell on the remotely controlled turntable.

The automated laser-cutting system removed 30-cm by 30-cm sections of the converter shell. Cutting parameters included a laser power level of 1.8 kW, oxygen assist gas, and a cutting speed of 76 cm/min. The auto-focus enhancement of the laser cutting head improved cutting performance by controlling the cutting head standoff distance.

A photo of the cutting operation is shown in figure 3.2.3.

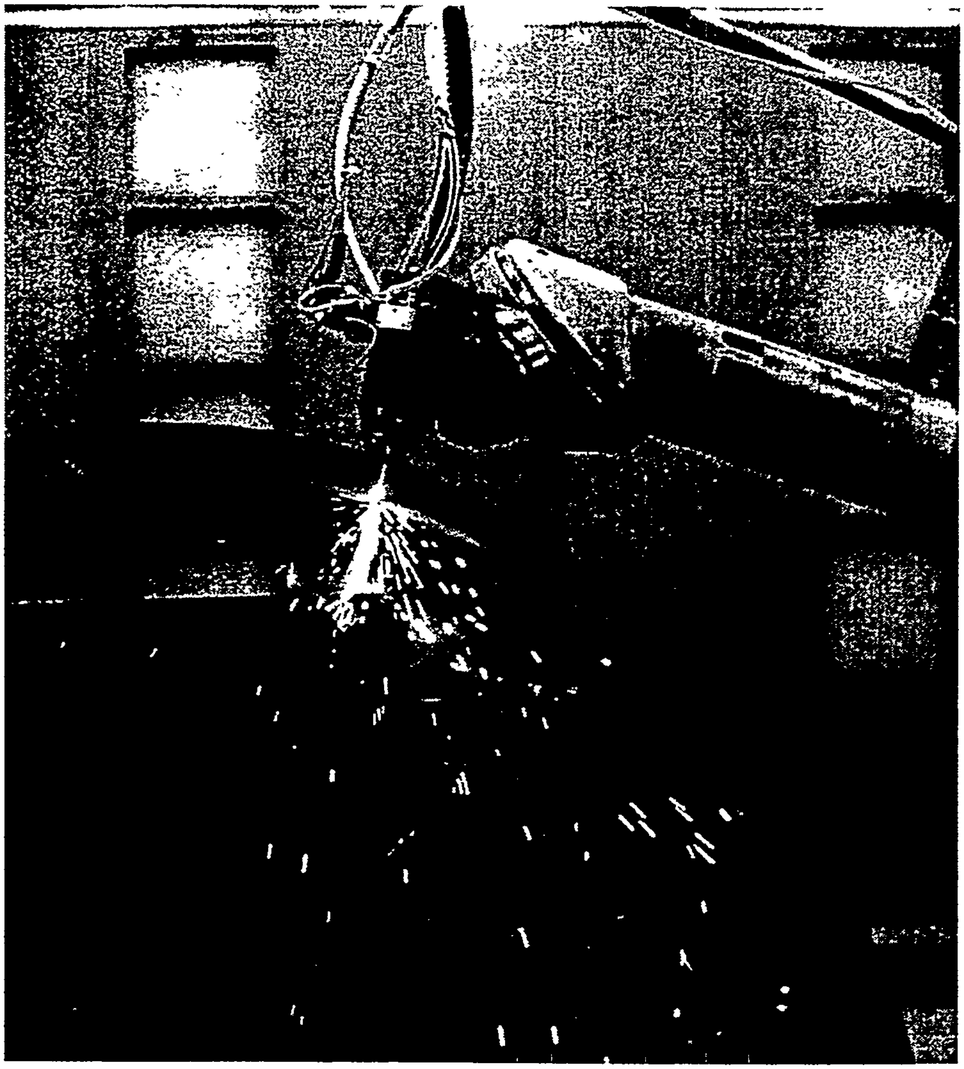
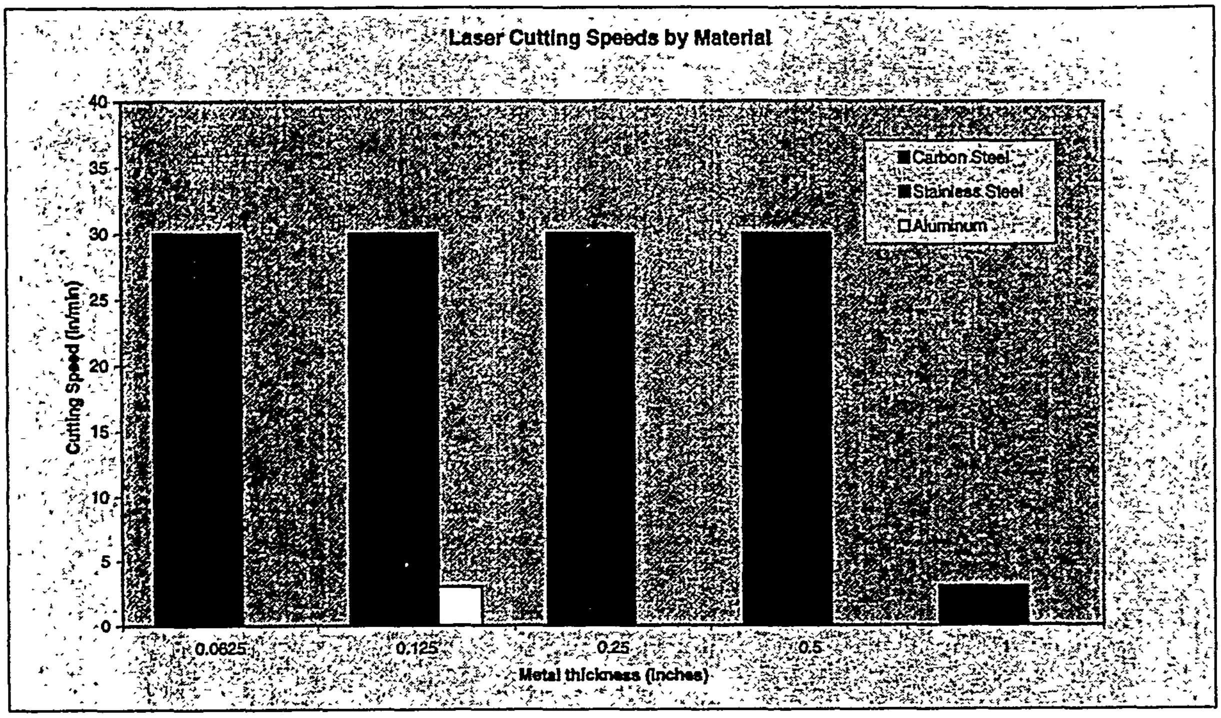


Figure 3.2.3 Laser cutting

Most of the laser cutting was performed using a continuous-wave (CW) laser beam. An exception was aluminum. To cut aluminum, the laser beam had to be delivered in a square wave, pulsed mode. Since aluminum cutting required higher laser power than steel, more instantaneous power was delivered to the cutting site using a pulsed beam. Drawbacks were that the pulsed-beam cutting was uneven. Also, the cutting speed was slower. This was expected due to the reflective properties of aluminum.

Additional laser cutting tests were performed at MSC to investigate the ability of the remote laser system to cut other materials. Results are summarized in Table 3.2.4

Table 3.2.4 Comparative results of laser-cutting experiments and demonstration.



Carbon steel and stainless steel followed the same performance curve. Steels at 1/2-inch or less were cut at speeds around 30-inches per minute, which rivals the speed of plasma cutting. The speed is drastically reduced, however, when cutting aluminum or 1-inch thick carbon steels. Aluminum 1/8 inch thick was cut at 1 inch per minute at 1500 watts square wave and 1-in thick carbon steel was cut at 3 inches per min at 1900 watts. The 1-inch per minute and 3-inches per minute cutting speeds for these materials are too slow to be feasible for size reducing these types of scrap metals in a production mode.

Scrap Removal

The adapted procedures of keeping the laser head pointing downward and restricting the cutting head motion limited the laser beam to linear travel. Therefore, this method allowed the sectioned pieces of the converter shell to drop directly into a waste box when a cut was complete. To eliminate manual handling, a conveyor stand was indexed into the opposite end of the shell and a monorail box was positioned so that the RSM could be removed using remote controls.

3.2.13 Results of MSC's Experiments and Demonstration

There were two notable differences between the cutting performance in the ETEC demonstration and the performance in the demonstration at MSC. First, the amount of airborne contaminate generated at ETEC was much greater than the amount generated at MSC; therefore, the airborne contaminate was more easily managed at MSC. This appears to be a function of the thickness of the material being cut. This relationship was observed in subsequent bench-scale tests as well.

Second, the maintenance requirements for the focusing head were greater at MSC. Frequent cleaning was necessary to remove the deposited airborne debris. Focus head cleaning was not required for any of the other cutting tests. The difference was attributed to the location of the cutting head (directly above the cutting site) in conjunction with the increased amount of cutting performed. This problem was minimized by installing a protective glass plate in front of the focusing lens and directing a dry-nitrogen purge gas through the auto-focus head to blow airborne debris away from the lens assembly. Those two changes reduced maintenance requirements significantly.

Preliminary cuts with the laser were successful. Continued optimization of cutting parameters, such as cutting speed, assist gas pressures, and laser power levels allowed MSC's full-scale laser cutting demonstration to be successful. The 5 foot diameter carbon steel converter shell was size reduced at 1800 watts, at 10 psi oxygen, and at a speed of 10 to 30 inches per minute. The resulting pieces (measuring about 1 ft^2) were captured in the monorail box. As anticipated, the use of the monorail box (the standard scrap metal handling box in MSC's recycling facility) prevented double handling of the size-reduced pieces.

Kerf for plasma-arc cutting was compared to the kerf for laser cutting. The average plasma kerf measured 0.125-inches while the laser kerf measured 0.035 inches for the 1/2-inch thick material. The laser's smaller kerf will result in savings due to the reduction of slag and the change out of HEPA filters

3.2.14 Additional Bench-Scale Testing

The ETEC and MSC laser-cutting demonstrations identified a number of areas where additional laser cutting information would be beneficial for future applications. A supplemental series of laboratory bench-scale tests was performed at EWI (under contract to ETEC) to investigate:

- the quantity of airborne particulate generated during laser cutting,
- the maximum cutting speeds for cutting various thickness of steel pipe, and
- the maximum thickness of steel plate that could be cut with a 3-kW, off-the-shelf Nd:YAG laser using fiberoptic beam delivery.

The results are shown in appendix 3.2.

3.2.15 Conclusions

All test results from the laser-cutting research are being examined to determine the cost-effectiveness of using lasers to reduce the size of the hundreds of converter shells and other RSM from the ETTP three-building D&D project. The demonstration provided the MSC team with a better understanding of the risks involved in laser cutting operations and pointed out the safety measures that must be adhered to when using laser technologies. Other specific challenges drawn from the demonstration include the following:

- The fiber optic cable is delicate and it incurs high maintenance costs
- Using automated equipment with lasers requires extensive programming
- Using an auto-focus head requires regular cleaning
- A considerable amount of time is needed when troubleshooting the entire system

In spite of these drawbacks, MSC's demonstration confirmed that a laser and robotic manipulator could be used to effectively and safely cut converter shells into pieces manageable for automated processing while minimizing radioactive and chemical exposure to personnel. The series of tests described here demonstrate that the Nd:YAG laser can cut both thin and thick components, can be operated under various degrees of remote operation and automation, and can cut up to 1/2 inch-thick steel at high linear cutting rates.

The ETEC demonstration showed the potential for cost savings using remote laser cutting. The MSC demonstration extended the application to thick materials using automated robotics. Subsequent bench scales tests provided further data on cutting capabilities using off-the-shelf laser systems. These various tests and demonstrations provided data useful for establishing operating parameters for production cutting. Test and demonstration data also provided important knowledge that could be directly applied to a production environment.

The relatively expensive laser hardware makes other technologies initially more cost effective for small-scale applications. However, reductions in personnel exposures and minimal maintenance requirements may override cost considerations in hazardous environment applications.

In summary, the successful series of lab and field-deployment tests and demonstrations confirms that current, off the shelf, laser technologies with remote fiberoptic beam delivery systems can be used effectively for automating material or component size reduction in D&D applications.

3.2.16 Path Forward

The project scope for the three-building D&D activities at the ETTP site includes size reduction, decontamination (where required), and disposition of over 126,000 tons of metals and over 1500 converter shells. Lasers should be further evaluated for possible use in this D&D work. High initial capital costs have inhibited their use to date. Also, newly installed equipment in MSC's recycling facility will handle larger components. This increases the size of components that can be accepted into automated cleaning facilities and reduces the need for size reduction activities.

3.3 Develop and Fabricate a New Product from RSM

3.3.1 Background

Recycling contaminated scrap metal saves space from disposal of scrap metals and minimizes the need for non contaminated metals to be used in the nuclear industry where they could become contaminated.

3.3.2 History of RSM Products

MSC has supported DOE in the recycle of contaminated scrap metals. Examples of past products are shown below:

Savannah River – Converted stainless steel piping, ductwork, slug buckets, and heat exchanger parts to over 1,000 stainless steel drums, 55 & 85 gallon size. This contract also included the fabrication of stainless steel boxes and closed head sanitary drums.

Oak Ridge – Converted over 1,000,000 pounds of metal into useful products including over 1,000 carbon steel B-25 boxes, carbon steel drums, stainless steel drums and specialty containers. Also, utilized lead from ORNL and INEL to make stainless steel clad, lead shielded overpack containers for storage and transportation of high level RH-TRU waste from INEL.

Idaho National Engineering Labs – Fabricated 12 shielded boxes for storage of U-233 contained in drums. Each box recycled over 8,000 pounds of contaminated lead.

3.3.3 Detailed analysis of DWPF specification

MSC examined the prospect of producing a finished DWPF canister at it's current facilities. The conclusion was to compete for the long term supply of DWPF canisters a facility and infrastructure would have to be built that would look substantially like that which is available in commercial industry (an integrated steel mill coupled with an ASME code fabrication shop). The driving force and funding for such a facility is not available.

Economies of scale and high demand for stainless steel products drove the cost of austenitic stainless steel plate to a little over a doller per pound. This cost is primarily in the raw materials, not labor. Scrap conditioning, melting furnaces, continuous casters, reheating furnaces, rolling mills, material handling assets, and pickling lines are all designed to reduce the man hours and increase the throughput. Typical throughput is between 1,200,000 to 1,600,000 tons per year. Steel plate in industry requires less than one man hour per ton in a fully tooled, highly integrated steel mill, and a yield from liquid metal to finished plate is in excess of 90%.

ASME certified fabrication shops typical of industry today, are equipped with the appropriate tools and retain the quality infrastructure to meet the requirements of a DWPF canister. Tools such as material handling assets, three roll benders, weld fixturing, and inspection tooling needed to make pressure vessels of all sizes are in place within commercial industry. A quality assurance program commensurate with the needs of the DWPF canisters, which include a certified weld inspector and appropriate NDE equipment as well as ASME code protocol, are in place in commercial shops.

MSC is a low volume specialty melting shop, geared to environmental compliance within a radioactive contol area. The melting machine at MSC, a 6 metric ton Vacuum Induction Degassing and Pouring (VIDP) furnace is capable, with slight modifications, of producing the liquid, high quality steel, but the rest of the facility is not geared for the needs of DWPF. Static cast ingots from the VIDP require manual surface conditioning and have a 60% by weight yield (from cast shape to rollable ingot). The rolling mill at MSC is limited in capacity to about 3 foot widths, and is designed as a nonferrous rolling mill with comparatively low torque output. This limitation causes a need for alternative DWPF designs, that utilize small plate sizes. After hot-rolling, the plate must be pickled to remove rolling scale. The descaling line at MSC is labor intensive and has limited throughput. Steel plate requires about 24 manhours per ton to produce. Coupled with low material yields, waste liabilities, and rolling mill constraints, plate production cannot be cost competitive with clean metal production.

The fabrication process at MSC is not commensurate with ASME code shop. The material handling assets, three roll bender, weld fixturing, and inspection cannot make the commercial industry design DWPF. The infrastructure to accomplish such a highly specialized container is not in place. That infrastructure includes the QA program and trained and qualified people to become an ASME code shop.

MSC alone, cannot make the investment to tool its existing plant to accomplish the task of producing cost effective DWPF canisters. A duplication of an existing commercial industrial base inside a radiation control area would be required. A feasibility study was performed by MSC in 1994 to determine the cost of a production steel facility inside a radiation control boundary. The cost was $140 M.

MSC does not see DWPF as a viable candidate for an RSM product line. Investment by MSC in this concept would not be financially responsible at this time. It is our estimation that the cost to produce the DWPF canister and be competitive within 25% of current canister costs is not feasible.

3.3.4 Products made from RSM

Fundamentally, a product made from RSM should include some or all of the following traits:

- Contain a large volume of RSM

- Not designed for efficient use of material (no need to design for minimization of metals used)
- Minimize processing steps
- Compete as a substitute for, not as a replica of, non-contaminated products

Candidate products requiring sophisticated machining and testing, with low material utilization, have much less chance of ever being successfully deployed. MSC has, and will continue to work with the Metals Recycling group within DOE to determine good candidates for products made from RSM. See the concept paper titled "MSC Recycled Contaminated Metals" in appendix 3.3.

In a letter to FETC in February, 1998, MSC requested to modify the scope and redistribute costs to support electro-refining and anode preparation activities. The results of these efforts are shown in the full scale electro-refining and melting sections of this report.

3.4 Refinement of Methods to Quantify Isotopic Elements

3.4.1 Executive Summary

Phase II efforts narrowed the field of characterization procedures to four that showed the most promise for determining the amount of technetium present in a bulk contaminated metal. The technologies were Inductively Coupled Plasma Mass Spectrometer (ICP Mass Spec), Tennelec LB5100 (Low level alpha/ beta counting), liquid scintillation, and EMPORE™ rad disks. During Phase III, the investigation of these technologies used to quantify ^{99}Tc contained in a bulk contaminated nickel matrix continued.

MSC's technical staff and laboratory technicians determined that liquid scintillation with random verification using ICP mass spec would be the preferred method for radiological detection. It was also decided that the ICP mass spec was the preferred method to provide information of metals concentration. Listed below are the results of the various techniques performed.

3.4.2 Electrorefining Sampling

All samples analyzed for ^{99}Tc by the various techniques were nickel shavings obtained by drilling through a nickel cathode. Immediately after being drilled from the cathode, each set of nickel shavings was allowed to cool, sealed in a plastic bag, and labeled with a number representing the cathode location from which it was drilled. The nickel shavings were stored in the sealed plastic bags until needed for analysis. For each analysis, the appropriate amount of nickel shavings was weighed out, the weight recorded, and the shavings sealed and transported in a plastic bag to the designated equipment or facility where the quantitative analysis of the nickel for ^{99}Tc was performed.

Samples of each cathode produced, anolyte and catholyte samples were taken during operations. This method of sampling allowed MSC to closely monitor the process and monitor the process for pH adjustments or indications of a breach in the cationic membranes.

3.4.3 Statistical Methods

After each analytical technique was employed, results were collected and analyzed using standard statistical methods. Data obtained with each analytical technique were evaluated for normality using the Kolmogorov-Smirnov test and verified by an independent analytical lab.

3.4.4 EMPORE™ Radiation Filter Disks

A technetium-specific ligand contained in the filter traps the technetium on the surface of the disk as a liquid sample is passed through the filter. Three liquid samples were prepared by dissolving the nickel shavings in nitric acid.

After dissolution of the nickel, hydrochloric acid was added to prevent precipitation of iron compounds. Each solution was diluted to a 500 ml final volume and filtered two times through a purchased EMPORE™ radiation filter disk. The filters were dried in a laboratory oven for 15 minutes at 50°C. The radioactivity trapped on the filters was counted using an LB-5100 alpha beta counter. The LB-5100 is a gas flow proportional charged particle detector that uses an argon-methane atmosphere to detect both alpha and beta particles. The results are presented below.

Results

^{99}Tc measurements obtained by Manufacturing Sciences Corporation using EMPORE radiation filter disks and an LB-5100 gross alpha beta counter

Sample designation	Results (decays/min)	Results (pCi/g)	Results (Bq/g)
1	77.90	11.7	0.43
4	40.84	6.1	0.23
5	59.37	8.9	0.33

[a] Each sample contained 3.0 g of nickel shavings.

Results from application of statistical tests **to the measurements reported by Manufacturing Sciences Corporation**

Mean	8.9 pCi/g (0.33 Bq/g)
Standard deviation	2.8 pCi/g (0.10 Bq/g)
Standard error of the mean	1.6 pCi/g (0.06 Bq/g)
Probability that data is normally distributed	0.654

The Kolmogorov-Smirnov test for normality is passed by this data. The probability of 0.654 is well above the 0.200 cutoff value.

Even though the results pass the test for normality, the results appear lower than those presented by other methods, investigation into other techniques for verification was pursued.

3.4.5 Liquid Scintillation Counting

Phase III continued the investigation of liquid scintillation counting that was initiated in Phase II. During Phase II, some results for the amounts of ^{99}Tc detected in the metal samples were variable using scintillation counting. This variability was attributed to the loss of some ^{99}Tc during the hydroxide precipitation of the metal prior to an Eichrom column separation performed before the actual scintillation counting. These inconsistencies in the amounts of ^{99}Tc detected using scintillation counting prompted examination of the entire procedure entailed in employing this technique to quantify ^{99}Tc in a metal matrix. One of the procedures used in the Phase II investigation of liquid scintillation counting is actually a two-part process, a combination of counting and separation techniques.

Typically for the counting process of the liquid scintillation technique, a sample is prepared by dissolving in an acid. One to two milliliters of this solution is added to 15 or 20 ml of a scintillation cocktail in a plastic vial. The vial is then placed between two photomultiplier tubes (PMTs). The radioactive decay of ^{99}Tc produces a beta particle which deposits energy in a molecule of the scintillation cocktail. When the excited molecule returns to a lower energy state, it emits a flash of light. The PMT detects the light flash and turns the signal into an electric pulse. If a flash is seen by both PMTs, located on either side of the sample, it is recorded as a count. However, if a pulse is read from only one of the PMTs, the signal is considered as electronic noise and is not counted. Reducing the noise signal allows this technology to successfully count radioactivity levels to near background levels.

The separation of the nickel from the ^{99}Tc is necessary to ensure that the ^{99}Tc is present in the sample at concentrations sufficient to produce an activity level significantly above the background. However, as demonstrated in Phase III, the separation of the nickel from the ^{99}Tc must be accomplished without loss of ^{99}Tc in order for the count to yield an accurate measure of the quantity of ^{99}Tc.

In Phase III, two independent investigations of liquid scintillation counting were conducted, both utilizing solid phase extraction to separate the nickel matrix from the ^{99}Tc. The organizations contracted to perform the analyses were Thermo NUtech and Analytical Services Organization of Lockheed Martin Energy Systems of Oak Ridge. Both of these evaluations proved that the results from the MSC analytical lab were accurate methods of detection.

Measurements by Thermo NUtech

Batch 1. Four samples of ^{99}Tc-contaminated nickel shavings were given to Thermo NUtech for analysis. There were two samples from each of two locations on cathode #3. Each sample contained about 10 g of nickel shavings. The shavings were digested in nitric acid at a low temperature (not specified). The ^{99}Tc was extracted using Eichrom TCS01 modified resin and the activity was determined using liquid scintillation counting. The results are presented below.

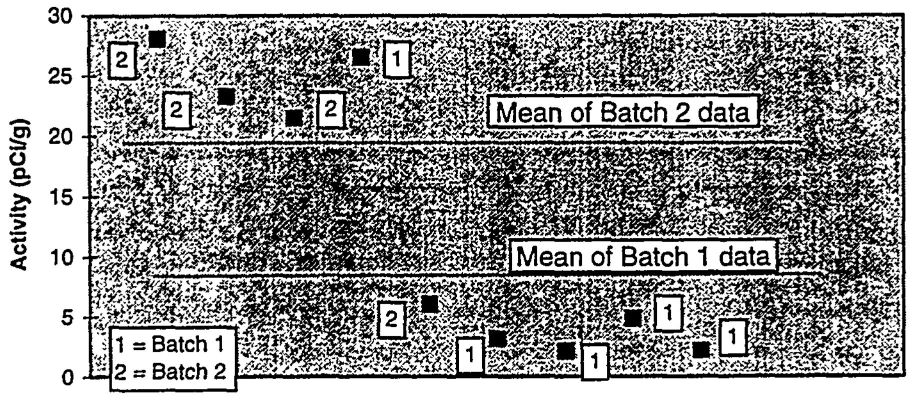
Measurements reported by Thermo NUtech on Batch 1			
Sample Location	Results (pCi/g)	Error (pCi/g)	Minimum detectable activity (pCi/g)
3	3.20 (0.118 Bq/g)	1.22 (0.045 Bq/g)	1.92 (0.071 Bq/g)
3	2.14 (0.079 Bq/g)	1.20 (0.044 Bq/g)	1.95 (0.072 Bq/g)
5	26.50 (0.981 Bq/g)	1.67 (0.062 Bq/g)	1.69 (0.062 Bq/g)
3	4.90 (0.181 Bq/g)	1.10 (0.041 Bq/g)	1.62 (0.060 Bq/g)
5	2.23 (0.083 Bq/g)	1.05 (0.039 Bq/g)	1.69 (0.062 Bq/g)

Results from application of statistical tests to the measurements reported by Thermo NUtech for Batch 1	
Mean	7.794 pCi/g (0.288 Bq/g)
Standard deviation	10.516 pCi /g (0.389 Bq/g)
Standard error of the mean	4.703 pCi /g (0.174 Bq/g)
Probability that data is normally distributed	0.006

With a probability of 0.006, the data fails the Kolmogorov-Smirnov test for normality. The very small probability suggests that there may be a systematic, rather than random, error in the measurements. Visual inspection of the Batch 1 data in Figure 1 reveals one measurement near 27 pCi/g (1 Bq/g) and four much smaller values [the mean of the smaller values is 3.2 pCi/g (0.12 Bq/g)]. The most likely systematic error that would produce the observed measurements would be the loss of ^{99}Tc during sample preparation.

Batch 2. Three 10-g samples of ^{99}Tc-contaminated nickel shavings were digested in nitric acid by the laboratory at Manufacturing Sciences Corporation and were sent to Thermo NUtech for analysis. All three samples were taken from the same location on cathode #3. The shavings were digested in heated nitric acid as specified by Thermo NUtech. The ^{99}Tc was extracted by Thermo NUtech using Eichrom TCS01 modified resin and the activity was determined using liquid scintillation counting. The results are summarized below.

Figure 1. Liquid Scintillation Measurements for ^{99}Tc



Again, the data fails the Kolmogorov-Smirnov test for normality. The results suggest that there may be a systematic, rather than random, error in the measurements. Visual inspection of the Batch 2 data in Figure 1 reveals three measurement around 27 pCi (1 Bq/g) the mean of the three measurements is 24.3 pCi/g (0.898 Bq/g)] and one much smaller value. The most likely systematic error that would produce the observed measurements would be the loss of ^{99}Tc during sample preparation.

Concerned about the validity of the data, MSC chose to continue research on liquid scintillation and sample preparation through other resources. Data provided by Thermo NUtech is provided in Appendix 3.4.

Measurements by Analytical Services Organization

A 10.8 g sample from near the center of nickel cathode #3 was obtained by drilling through the cathode. The shavings were bagged, labeled, and delivered to the analytical laboratory contracted to perform the liquid scintillation counting measurements. The Analytical Services Organization of Lockheed Martin Energy Systems in Oak Ridge, Tennessee was contracted to make the measurements.

The sample received from Manufacturing Sciences Corporation was reduced in size with metal cutters to facilitate digestion. For a preliminary investigation, roughly 0.1 g of samples were digested in Teflon™ beakers that were heated to a low temperature on a hot plate. For the primary investigation, between 0.25 and 0.5 g of sample was placed in a Teflon™ reaction vessel and digested in an open vessel microwave system using both nitric acid and hydrogen peroxide to dissolve the sample. The digestion temperature was controlled to 80±0.1°C. In both the preliminary and primary investigations, the digested samples were diluted to 1M nitric acid and placed in an automated solid phase extraction column. The radioactivity of the extracted samples were counted by liquid scintillation techniques.

Recovery of the digested ^{99}Tc with a Tevaspec extraction resin in a solid phase extraction unit was almost quantitative, ranging from approximately 98 to 102%.

Measurements Reported by Analytical Services Organization			
Sample Location	Results (Bq/g)	Error (Bq/g)	Minimum detectable activity (Bq/g)
Preliminary Investigation			
2	1.15*	0.47	0.53
Primary Investigation			
2	1.01	0.21	0.34
2	0.928	0.21	0.34
2	0.884	0.22	0.35
2	0.847	0.22	0.35
2	0.751	0.21	0.35

*Represents the average of 2 sample runs.

Results from application of statistical tests to the measurements reported by Analytical Services Organization	
Mean	0.884 Bq/g
Standard deviation	0.096 Bq/g
Standard error of the mean	0.043 Bq/g
Reported mean[a]	0.88 Bq/g
Reported error[a]	0.49 Bq/g
Probability that data is normally distributed	0.742

[a]Computed result reported by Analytical Services Organization

With a probability of 0.742, the measurements pass the Kolmogorov-Smirnov test for normality. A probability this large indicates that any errors are likely to be random and there is a large degree of similarity between the distribution of these measurements and a theoretical normal distribution.

The Analytical Services Organization was requested to develop a liquid scintillation counting technique for ^{99}Tc that was accurate to 1-2 pCi/g (0.037-0.074 Bq/g). As seen from the results, this level of detection was not achieved using liquid scintillation counting. The minimum detectable activity was found to be approximately a factor of nine higher than requested, 0.34 Bq/g $\approx 9 \times 0.037$ Bq/g (9.2 pCi/g $\approx 9 \times 1.0$ pCi/g).

Increasing the amount of contaminated nickel metal placed into solution could lower the minimum detectable activity. Decreasing the minimum detectable activity by a factor of nine, would involve the temperature-controlled digestion of large masses of nickel metal with large volumes of nitric acid. Passing even larger diluted volumes through the extraction columns would involve prohibitively long periods of time. It appears that it is not practical to achieve the desired 1-2 pCi/g detection limit with liquid scintillation counting.

Although the minimum detectable value was not met, the samples had a relatively constant value within their analytical uncertainty. There is some fluctuation in the values, especially if the release limit is around 1 Bq/g. However, at these low levels, this is probably attributable to preparation variables including pipetting errors, transferring errors, and weighing errors. Using an automated system maximized the precision of the extraction process.

To validate the liquid scintillation counting results, the activity of the sample was measured by Lockheed Martin Analytical Services Organization using inductively coupled plasma mass spectroscopy. The result from mass spectroscopy was 1.36 Bq/g with an uncertainty of 0.04 Bq/g (1.36±0.04 Bq/g) and a minimum detectable activity of 0.02 Bq/g. The mass spectroscopy measurement is in agreement with the liquid scintillation result (0.88±0.49 Bq/g) within the stated errors. The report provided by the Analytical Services Organization is included in appendix 3.4.

Liquid Scintillation at MSC

When counting a sample in a scintillation cocktail, radiation emissions are converted to light which are seen by a photomultiplier tube of the detector. If a sample contains quenching agents, either color or chemical, the counting efficiency of the system is affected.

If disintegrations per minute (DPM) are being calculated, the sample must be checked and corrected for quench or erroneous results may be determined. By comparing the spectra and number of counts in a quenced and unquenched version of the same sample, a quence curve can be constructed to correct for the loss of activity.

The quench curve correlates the known DPM of a standard to some measured amount of quenching agent. The curve is generated by counting a series of standard samples, each containing the same amount of nuclide, in our case ^{99}Tc and varying amounts of quenching agent, $NiNO_3$. The sample containing no quenching agent generates the highest counts, while the sample with the most quench generates the lowest counts. The efficiency for each sample is calculated and the quench curve is plotted as % efficiency vs amount of quenching material added.

Figure 3.4.1 show the quench correction curve generated using ^{99}Tc and varying amounts of $NiNO_3$ (indicated as tSIE – transformed Spectral Index of the External standard – on the x axis) and the corrected DPM values obtained when using the curve to obtain the number of counts present.

The corrected values indicate that as much as 2.5 ml of a $NiNO_3$ solution at the concentrations to be used for analyzing the Tc-99 can be added to the cocktail with little or no loss in DPM. .

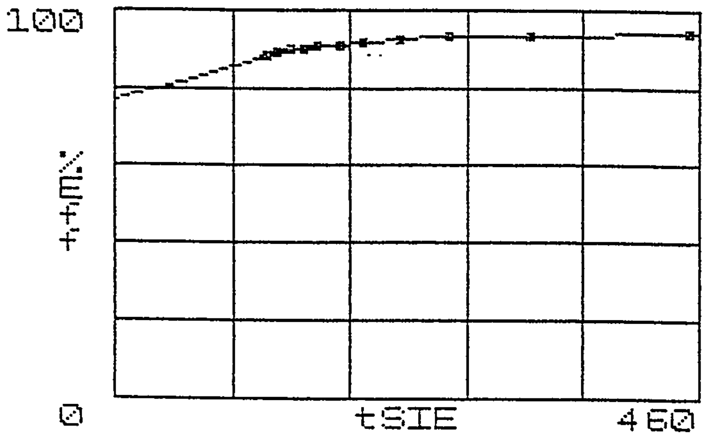
Liquid scintillation at MSC, with random verification by external laboratories has proven to be a successful method for characterization of low levels of technetium in nickel.

```
21 Oct 1998  11:23          ALPHA/BETA - 1.08                    Page #1
Protocol #: 6               Tc-99/Ni                       User  : RSMRP La

Time:  1.00
Data Mode: DPM               Nuclide: TC-99/NI      Quench Set: TC-99 QC
Background Subtract: None

              LL      UL     LCR    2S%      BKG
Region A:    0.0 -   293      0     0.0     0.00
Region B:    2.0 -   293      0     0.0     0.00
Region C:    0.0 -   0.0      0     0.0     0.00

Quench Indicator: tSIE/AEC
   Ext Std Terminator: Count
Ni in Nitric Acid:Nominal 1g/25mL
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal
```



```
tSIE    %Eff
452.85  94.17
326.05  93.21
262.00  93.69
222.04  92.34
193.79  91.83
175.40  90.98
157.38  91.14
145.19  90.27
135.44  89.72
125.28  89.08
116.84  88.24
```

```
S#       SMPL_ID                                     TIME     CPMA      CPMB      DPM1     tSIE
 1 11000dpm Tc-99Std  - No Quench Agent              1.00  10209.1   10183.7   10842.9   451.24
 2    "        "      " 0.25ml NiNO3 Solution        1.00  10435.6   10419.2   11196.3   326.12
 3    "        "      " 0.50ml       "      "        1.00  10476.0   10462.0   11189.7   259.92
 4    "        "      " 0.75ml       "      "        1.00  10321.4   10315.2   11180.7   220.86
 5    "        "      " 1.00ml       "      "        1.00  10259.0   10254.4   11173.0   193.54
 6    "        "      " 1.25ml       "      "        1.00  10025.8   10021.7   11017.6   173.71
 7    "        "      " 1.50ml       "      "        1.00  10038.0   10038.0   11015.6   158.88
 8    "        "      " 1.75ml       "      "        1.00  9864.00   9860.42   10931.8   144.46
 9    "        "      " 2.00ml       "      "        1.00  9785.00   9778.09   10907.0   135.35
10    "        "      " 2.25ml       "      "        1.00  9720.00   9717.52   10910.5   125.47
```

3.4.6 Bulk Activity Approximation

Purpose

A bulk activity method was also researched to determine the concentration of ^{99}Tc in nickel that has been decontaminated. This method would allow a surface survey of nickel to estimate the activity in the bulk. The activity of ^{99}Tc in the decontaminated nickel cathode was 0.88 ± 0.49 Bq/g as measured by liquid scintillation counting. It is desirable, however, to identify a more rapid, less costly measuring technique for estimating the ^{99}Tc activity in the cathodes. These estimates, if correlated to the accurate, more rigorous measurements of activity obtained by liquid scintillation counting, would be a means to approximate the ^{99}Tc activity in each cathode. Currently, the purpose of this technique would be to establish quick methods to estimate levels of contamination for internal purposes only. More rigorous approaches, such as liquid scintillation or ICP Mass Spec, would be required to quantify for potential metals release.

Correlation

A correlation will provide the relationship between the rigorously determined bulk activity and the approximate bulk activity. Determination of the correlation function requires at least three repeatable liquid scintillation measurements from nickel with significantly different levels of bulk activity. A measurement of the approximate bulk activity must be obtained for each of the liquid scintillation measurements. After the data have been collected, the correlation function may be constructed by curve fitting using either graphical or statistical techniques.

Approximate Bulk Activity

The approximate bulk activity of the bulk-contaminated nickel was calculated from the surface activity using the relationship

$$A_{bulk} = A_{surface}/m$$

where m is the mass of the nickel producing the surface activity. This relationship was developed from the definition of specific activity. The surface activity is a measurement easily obtained with readily available surveying instruments. The mass was determined after making the following assumptions.

(1) The ^{99}Tc in the cathode is distributed homogeneously.
(2) The beta particles reaching the surface can be characterized by their average energy.
(3) There is no loss of energy by the beta particles as they pass through the air interface and probe window that are between the surface of the nickel and the detector.
(4) The beta particles are emitted from a volume of nickel with an assumed surface area and a thickness that is the linear distance traveled by a beta particle of average energy.

Theoretical Basis

The mass of nickel from which the beta particles detected at the surface are emitted is given by

$$m_{Ni} = V_{Ni} \bullet \rho_{Ni}$$

where ρ_{Ni} is the density of nickel (8.8 g/cm^3) and V_{Ni} is the volume of the nickel emitting the beta particles detected at the surface. The volume is given by

$$V_{Ni} = x_{Ni} \bullet A$$

where A is the assumed surface area (100 cm^2) and x_{Ni} is the linear distance traveled by the negative beta particle emitted by the ^{99}Tc in nickel.

The linear distance x_{Ni} is defined as

$$x_{Ni} = R/\rho_{Ni} \qquad \text{(Choppin and Rydberg)}$$

where R is the range or density thickness (g/cm^2) of the beta particle in nickel. The value of R for a ^{99}Tc beta particle of average energy (100 keV) is 2.225×10^{-2} g/cm^2, as given in Table 2. Using this value for R, the maximum thickness of nickel that a 100-keV beta will penetrate is

$$x_{Ni} = (2.225\times10^{-2}\ \text{g/cm}^2)/(8.8\ \text{g/cm}^3) = 2.528\times10^{-3}\ \text{cm} \cong 2.53\times10^{-3}\ \text{cm}.$$

The volume, V_{Ni}, was computed to be

$$V_{Ni} = x_{Ni} \bullet A = (2.53\times10^{-3}\ \text{cm}) \bullet (100\ \text{cm}^2) = 2.53\times10^{-1}\ \text{cm}^3.$$

Using the computed volume and the given density, the mass of nickel from which the detected beta particles are emitted is

$$m_{Ni} = V_{Ni} \bullet \rho_{Ni} \cong (2.53\times10^{-1}\ \text{cm}^3) \bullet (8.8\ \text{g/cm}^3) = 2.23\ \text{g}.$$

Average Energy of Beta Particles

Beta decay events do not produce beta particles with a unique energy. Rather, because the available decay energy is shared with the nucleus, the beta particles emerging from beta decay events have a continuous range of energies from the maximum down to zero. The negative beta particles (electrons) emitted in the decay of ^{99}Tc have a maximum energy of 292 keV, approximately 300 keV, cited in *Nuclides and Isotopes, 14th edition.*

Choppin and Rydberg gives the following approximation for a typical beta decay: the average energy of a beta particle is equal to 1/3 the maximum energy. For ^{99}Tc, the average beta particle energy is assumed to be 1/3 of 300 keV, or 100 keV. This average energy was used as the basis for determining the penetration distance of the beta particles and the mass of nickel from which the beta particles are emitted.

Range of Beta Particles in Nickel

There are no readily available tabulated values for R in nickel. There are, however, tabulated values of R for other elements, including copper which has a density approximately equal to that of nickel ($\rho_{Cu} = 8.96$ g/cm^3, $\rho_{Ni} = 8.8$ g/cm^3). For this approximation, it is assumed that $R_{Cu} \cong R_{Ni}$.

The variation of R with the energy of the beta particles in copper is given in Table 1. These tabulated values of R were computed using the continuous slowing down approximation (CSDA). Because the CSDA models the slowing down of the beta in terms of the number of interactions with atomic electron clouds, materials of approximately equal densities would be expected to have roughly equivalent values for R, justifying the assumption that $R_{Cu} \cong R_{Ni}$.

Table 1. Range and computed penetration distance of beta particles in copper

Material	Energy (keV)	Density ρ (g/cm^3)	Range R (g/cm^2)	Penetration Distance $x = R/\rho$ (cm)
Copper	300	8.96	1.26E-01	0.014096
Copper	100	8.96	2.23E-02	0.002483
Copper	90	8.96	1.87E-02	0.002085
Copper ·	80	8.96	1.54E-02	0.001713

Source: ICRU Report 37, *Range of Beta Particles and Positrons*, International Commission on Radioactive Units and Measurements, Volume 37

Linear Distance Traveled by the Beta Particles

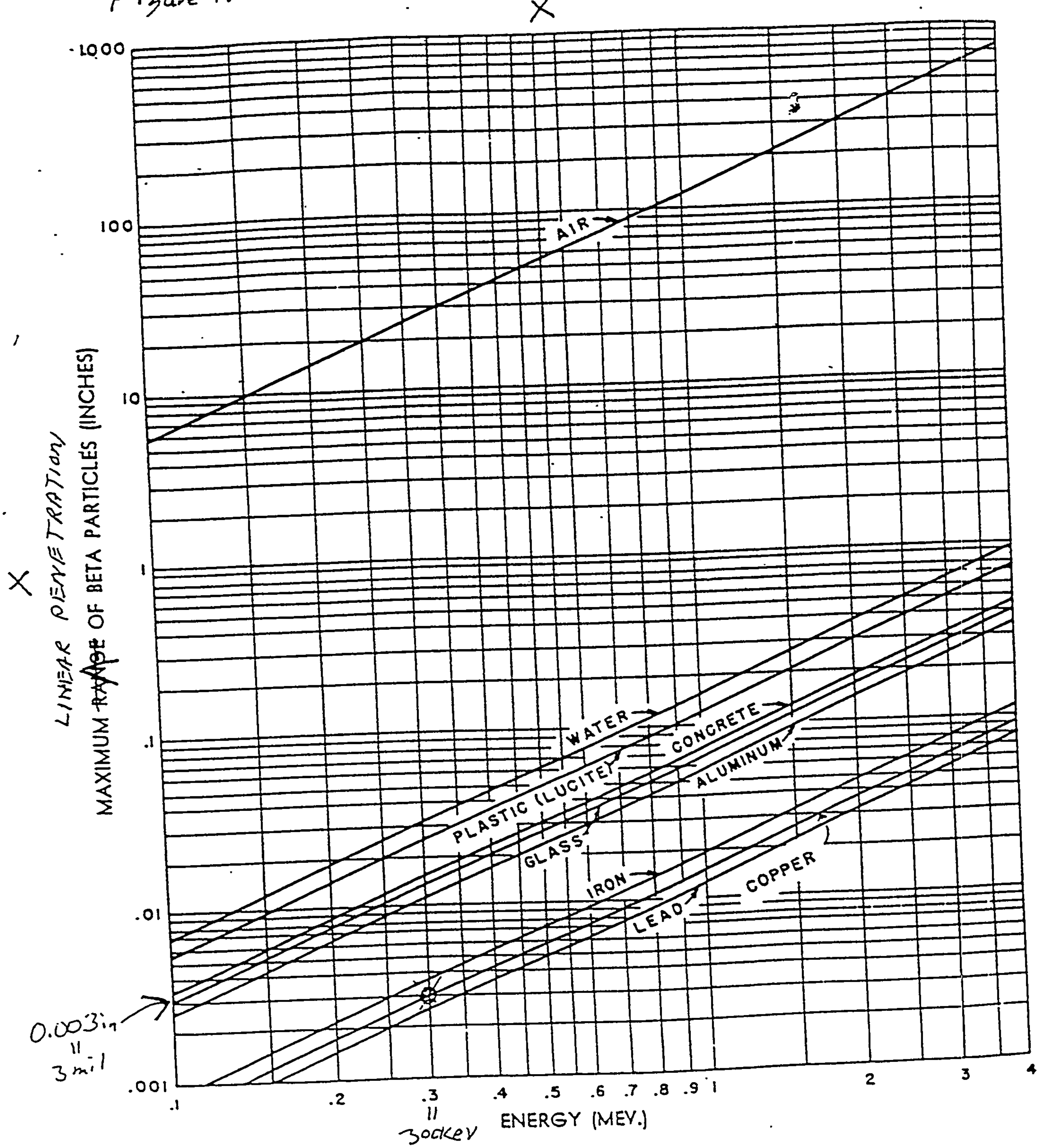
The linear distance traveled by the beta particles through the nickel is given by $x_{Ni} \cong R_{Cu}/\rho_{Ni}$, also referred to as the penetration distance. Table 2 shows the values for the penetration, x_{Ni}, that would result if the range of beta particles in copper were used to determine the penetration of beta particles in nickel. Figure 1 shows the linear penetration as a function of energy from 0.1-4 MeV for materials ranging in density from air to lead.

Table 2. Computed penetration of beta particles in nickel using range values for copper

Material	Energy (keV)	Density ρ (g/cm^3)	Range R (g/cm^2)	Penetration Distance $x = R/\rho$ (cm)
Use copper range values for nickel				
Nickel	300	8.8	1.263E-01	0.014352
Nickel	**100**	**8.8**	**2.225E-02**	**0.002528**
Nickel	90	8.8	1.868E-02	0.002123
Nickel	80	8.8	1.535E-02	0.001744

Source: ICRU Report 37, *Range of Beta Particles and Positrons*, International Commission on Radioactive Units and Measurements, Volume 37 from Radiological Health Handbook compiled and edited by Bureau of Radiological Health, 1970

Figure 1. PENETRATION ABILITY OF BETA RADIATION,



Ni is between Iron & Copper

$X = R/\rho$

R is range.
ρ is density.

The maximum range of beta particles as a function of energy in the various materials indicated. (From SRI Report No. 361, "The Industrial Uses of Radioactive Fission Products". With permission of the Stanford Research Institute and the U. S. Atomic Energy Commission.)

Instrumentation, Surface Activity Measurements, and Statistical Analyses

A Ludlum Model 4389 alpha/beta plastic scintillation probe with a Ludlum Model 2224 scalar was chosen to test the ^{99}Tc in the cathode to calculate the approximate bulk activity. The area of the probe is 100 cm^2; the density thickness of the probe window is 1.2 mg/cm^2. The probe efficiency is 9.4% for beta particles and 17.0% for alpha particles. The area of each measured location on the cathode is greater than 100 cm^2. When the alpha/beta scintillation probe is placed in contact with the cathode, the entire active area of the probe is filled. Data collected during these tests and the corresponding statistical analysis are provided in Appendix 3.4.

The average surface activity of cathode #3 may be calculated from the Set 1 measurements by subtracting the mean background count rate from the mean combined count rate and converting to Becquerels.

$$A = \left((438.3 \pm 24.3) - (427.2 \pm 41.0)\frac{\text{counts}}{\text{minute}}\right)\left(\frac{\text{disintegrations}}{0.094\ \text{counts}}\right)\left(\frac{\text{min}}{60\ \text{sec}}\right)\left(\frac{\text{Bq}}{\frac{\text{disintegrations}}{\text{sec}}}\right)$$

$$A = 1.97 \pm 0.30\ \text{Bq} \cong 2.0 \pm 0.3\ \text{Bq}$$

The average bulk activity of cathode #3 is found by dividing the measured surface activity by the mass of the emitting layer, 2.23g.

$$A_{bulk,cathode\,\#3} = \frac{(1.97 \pm 0.30\,\text{Bq})}{2.23\,\text{g}} = 0.883 \pm 0.13\frac{\text{Bq}}{\text{g}} \cong 0.88 \pm 0.13\frac{\text{Bq}}{\text{g}}$$

Using measurements from set 2, the average surface activity of cathode #3 may be calculated.

$$A = \left((183.25 \pm 15.61) - (170.0 \pm 17.4)\frac{\text{counts}}{\text{minute}}\right)\left(\frac{\text{disintegrations}}{0.094\ \text{counts}}\right)\left(\frac{\text{min}}{60\ \text{sec}}\right)\left(\frac{\text{Bq}}{\frac{\text{disintegrations}}{\text{sec}}}\right)$$

$$A = 2.35 \pm 0.44\ \text{Bq} \cong 2.4 \pm 0.4\ \text{Bq}$$

The average bulk activity of cathode #3 resulting from Set 2 measurements is computed by dividing the average surface activity by the mass of the emitting layer, 2.23g.

$$A_{bulk.cathode\,\#3} = \frac{(2.35 \pm 0.44\,\text{Bq})}{2.23\,\text{g}} = 1.054 \pm 0.197\frac{\text{Bq}}{\text{g}} \cong 1.05 \pm 0.20\frac{\text{Bq}}{\text{g}}$$

Additional measurements were taken (sets 3-5) but were unusable due to inconsistencies that indicated an equipment malfunction. This data is shown in appendix 3.4.

Higher Activity Level

A correlation between the approximate bulk activity of ^{99}Tc-contaminated nickel and liquid scintillation bulk activity requires activity measurements at several levels. The surface activity of cathode #3 is near the background activity. To produce the desired correlation between the approximate bulk activity and the rigorously determined bulk activity, it is necessary to measure the activity of samples with higher levels of ^{99}Tc than those measured in cathode #3. A piece of ^{99}Tc-contaminated nickel barrier was measured that had higher surface activity than the decontaminated nickel. Additionally, the surface activity of a piece of decontaminated nickel, which had been removed from cathode #3, was measured with the alpha-beta scintillation detector.

The probe used for these measurements was 7.5 cm by 16.5 cm, 123.75 cm^2. The contaminated barrier nickel was roughly 8.5 cm by 9.8 cm, 83.3 cm^2, and the piece from cathode #3 was approximately a 10.2 cm square, 104.0 cm^2. The change in probes requires a change in the mass from which the beta particles are assumed to emerge. These corrections are 83.3% for the barrier nickel and 104.0% for cathode #3. The detailed measurements are shown in appendix 3.4. A summary is shown below.

Statistical analysis of measurements of beta particle emissions from barrier nickel and cathode #3 with an alpha/beta scintillation probe

Sample location	Mean (counts/min)	Standard deviation (counts/min)	Standard error of the mean (counts/min)
Barrier nickel	1970.3	39.64	12.54
Cathode #3 (removed piece)	322.9	17.19	5.44
Background	298.7	22.54	7.13

The average surface activity of nickel barrier may be calculated from the measurements and converted to Becquerels. The average bulk activity of the barrier nickel is then calculated by dividing the average surface activity by the mass of the emitting layer, 2.23g with the appropriate geometry correction.

$$A_{bulk.barrier\ nickel} = \frac{(296.38 \pm 18.35\,\mathrm{Bq})}{(0.833 \times 2.23\,\mathrm{g})} = 159.55 \pm 9.88\,\frac{\mathrm{Bq}}{\mathrm{g}} \cong 159.6 \pm 9.9\,\frac{\mathrm{Bq}}{\mathrm{g}}$$

Similarly, the bulk activity of cathode #3 is found to be

$$A_{bulk.cathode\ \#3} = \frac{(4.29 \pm 0.55\,\mathrm{Bq})}{(1.040 \times 2.23\,\mathrm{g})} = 1.85 \pm 0.24\,\frac{\mathrm{Bq}}{\mathrm{g}} \cong 1.9 \pm 0.2\,\frac{\mathrm{Bq}}{\mathrm{g}}$$

Alternate System

The continued difficulties encountered during sets 3-5 measurements prompted a change in the instrumentation. The Ludlum Model 2200 scaler was retained, but the alpha/beta scintillation probe was replaced with a shielded Geiger-Müller tube. The area of the probe is 15 cm^2. This is much smaller than the area of the previously used probe, 100 cm^2.

The efficiency of this new system was determined using a ^{99}Tc source of known activity. The source activity is 13,200 decays/min into 4π steradians. Based on two 10-min counts of the ^{99}Tc source, the average count rate of the source is 1809.5±11.0 counts/min.

The average background was determined from two 900-min counts to be 30,934.5 counts/900 min with a standard deviation of 195.9 counts/900 min and a standard error of the mean equal to 138.5 counts/900 min. This produces a background count rate equal to 34.37±0.22 counts/min.

Correcting the ^{99}Tc source average count rate, 1809.5 counts/min, for the background count rate, 34.37 counts/min, gives 1775.1 net counts/min. Dividing the corrected count rate by the known activity of the source gives the efficiency of the detection system

$$E = \left(\frac{1775.1 \dfrac{\text{counts}}{\text{min}}}{13{,}200 \dfrac{\text{counts}}{\text{min}}} \right) \times 100 = 13.45\%$$

Detailed measurements taken with the shielded Geiger-Muller tube are shown in Appendix 3.4. A summary of the data is shown below.

Statistical analysis of measurements of beta particle emissions from barrier nickel and cathode #3 with a shielded Geiger-Müller tube

Sample location	Mean Total counts	Standard deviation (counts)	Standard error of the mean (counts)
Nickel barrier	34,521.5	276.59	138.30
Cathode #3 (removed piece)	2148.5	55.51	27.76
Cathode #3 (locatiōn 1B)	2082.3	60.18	34.74

The average surface activity of the nickel barrier may be calculated and converted to Becquerels.

$$A = \left(\left(\frac{34{,}521.5 \pm 276.59}{60} \right) - (34.37 \pm 0.22) \frac{\text{counts}}{\text{minute}} \right) \left(\frac{\text{disintegrations}}{0.1345 \text{ counts}} \right) \left(\frac{\text{min}}{60 \text{ sec}} \right) \left(\frac{\text{Bq}}{\frac{\text{disintegrations}}{\text{sec}}} \right)$$

$$A = 67.04 \pm 0.54 \text{ Bq} \cong 67.0 \pm 0.5 \text{ Bq}$$

The average bulk activity of the barrier nickel is then calculated by dividing the average surface activity by the mass of the emitting layer, 2.23g with the appropriate geometry correction.

$$A_{bulk,barrier\ nickel} = \frac{(67.04 \pm 0.54 \text{ Bq})}{(0.15 \times 2.23 \text{ g})} = 200.30 \pm 1.61 \frac{\text{Bq}}{\text{g}} \cong 200.3 \pm 1.6 \frac{\text{Bq}}{\text{g}}$$

The average bulk activity of cathode #3 at location 1B and the excised piece of cathode #3 can be determined in a similar manner.

$$A_{bulk,cathode\ \#3(location\ 1B)} = \frac{(0.042 \pm 0.0015 \text{ Bq})}{(0.15 \times 2.23 \text{ g})} = 0.124 \pm 0.004 \frac{\text{Bq}}{\text{g}} \cong 0.12 \pm 0.01 \frac{\text{Bq}}{\text{g}}$$

$$A_{bulk,cathode\ \#3(removed\ piece)} = \frac{(0.178 \pm 0.015 \text{ Bq})}{(0.15 \times 2.23 \text{ g})} = 0.533 \pm 0.045 \frac{\text{Bq}}{\text{g}} \cong 0.53 \pm 0.05 \frac{\text{Bq}}{\text{g}}$$

The bulk activity of cathode #3 at location 1B is rather low. Computationally, this was caused by the one low count that substantially lowered the average count rate. Additional sampling could produce results for location 1B that are closer to the bulk activity computed for the removed piece of cathode #3.

Results

To determine if surface measurements of ^{99}Tc activity could be related to bulk contamination, measurements were obtained using two surface activity measuring probes: an alpha/beta plastic scintillator and a shielded Geiger-Müller tube. In total 10 sets of measurements were collected. Eight sets of data were collected from samples of decontaminated nickel (cathode #3), and two sets of data were collected from nickel barrier that had not been decontaminated. Three of the sets of measurements of the surface activity of the decontaminated nickel were unusable. The data are presented in the following section.

Measurements of the surface activity of the barrier nickel were performed with an alpha/beta plastic scintillator and a shielded Geiger-Müller tube. One data set was collected with each detector. The bulk activities of the barrier nickel were calculated from the surface activities using the method described in the Approximate Bulk Activity section. The computed bulk activities are as follows:

alpha/beta plastic scintillator	159.6±9.9 Bq/g and
shielded Geiger-Müller tube	200.3±1.6 Bq/g.

For five sets of measurements of the surface activity of decontaminated nickel, the bulk activity of ^{99}Tc was computed. Three sets of measurements were obtained using an alpha/beta scintillation probe and two sets of measurements were obtained using a shielded Geiger-Müller tube. The bulk activities are given in Table 3.

Table 3. Computed bulk activity of decontaminated nickel

Probe type	Bulk activity (Bq/g)
alpha/beta plastic scintillator	0.88±0.13
alpha/beta plastic scintillator	1.05±0.20
alpha/beta plastic scintillator	1.85±0.24
shielded Geiger-Müller tube	0.12±0.01
shielded Geiger-Müller tube	0.53±0.05

The average of the computed bulk activities for the decontaminated nickel is 0.89 Bq/g. This compares well with the average of independently obtained liquid scintillation measurements of the decontaminated nickel, 0.88±0.49Bq/g.

A further study was conducted in the MSC lab in July 1998. A nickel cathode from early trials of the full scale electrorefining cell were sectioned in nine equal portions. Sections were counted using a Ludlum 2224 with a alpha/beta scintillation detector. The counted section was marked and a 100 cm^2 area cut from the quadrant and cut into small pieces. Twenty ml of HNO3 was added to the samples and heated until dissolved. Water was added and the solution evaporated down to 15 ml at 60 degrees C to reduce the nitric acid concentration. The sample was quantitatively rinsed into a 100 ml volumetric flask and diluted to 100 ml with distilled water. One ml of the solution was removed and added to 12 ml of liquid scintillation cocktail and counted for 100 minutes in the Packard 2700 TR counter. Sample A was taken from the front starter sheet from the mother blank and Sample B was taken from the back side. Sample A averaged 5.69 Bq/g using liquid scintillation and Sample B averaged 4.46 Bq/g. Using the 2224 alpha/ beta scintillator, Sample A averaged 0.37 Bq/g and sample B averaged 4.66 Bq/g indicating that the results are not close enough to rely on a surface measurement solely for determination of bulk isotopic content of nickel volumetrically contaminated with technetium. Details of this study are shown in Appendix 3.4.

The results of these studies, obtained with an alpha-beta plastic scintillation detection system, demonstrate the feasibility of using this surface technique only for screening for bulk activity. More rigorous detection methods, such as liquid scintillation are required for reliable isotopic measurements.

3.4.7 Sampling During Electrorefining Operations

Details of results obtained during operation of the one full scale electrorefining cell are shown in section 3.1 (Full-scale electrorefining). Examples of laboratory results are shown in appendix 3.4.

REFERENCES

Choppin and Rydberg, *Nuclear Chemistry: Theory and Applications*, 278-280, Pergamon Press, Oxford, U.K., 1980.

ICRU Report 37, *Stopping Powers for Electrons and Positrons*, International Commission on Radioactive Units and Measurements, Volume 37, Bethesda, Maryland.

Meyer, *Data Analysis for Scientists and Engineers*, 17-18, John Wiley & Sons, New York, 1975.

Meir and Zünd, *Statistical Methods in Analytical Chemistry*, 49-54, John Wiley & Sons, New York, 1993.

NCRP Report No. 58, *A Handbook of Radioactivity Measurements Procedures*, 307-311, National Council on Radiation Protection and Measurements, Bethesda, Maryland, 1985.

Nuclides and Isotopes, 14th edition, General Electric Company, Nuclear Energy Operations, San Jose, California, 1989.

Tsoulfanidis, *Measurement and Detection of Radiation*, 57-68, McGraw-Hill Book Company, New York, 1983.

Conclusions

- Electro-refining technology developed in this project has demonstrated the capability, on a full scale, to remove technetium from diffusion plant nickel barrier to 1-2 Bq/g.
- Direct dissolution has proven promising on a lab scale but requires additional research for scale up activities.
- An automated NdYAG laser cutting system is capable of size reducing a ½ inch thick converter shell from the gaseous diffusion plant. High initial capital costs have prevented its further use in decommissioning activities at MSC.
- Liquid Scintillation is the method of choice for quantifying low levels of technetium in nickel verified on a random basis by Inductively Coupled Plasma Mass Spectrometer.
- Detailed evaluation of the DWPF product specification revealed that the cost to produce the DWPF canister from RSM at MSC's facility and be competitive within 25% of current canister costs is not feasible at this time.

#2

Appendix 3.1

Watts Solution for Electro-refining of Nickel
Specification and Costs

March 6, 1998

Elizabeth H. Terry

FETC 4020-10401-31100

Watts Solution for Electro-refining of Nickel
Specifications and Costs

Information was collected to determine specifications and costs of the chemical components for a Watts solution to be used in the electro-refining process to recover nickel from barrier nickel contaminated with ^{99}Tc. The findings support the following recommendations.

(1) The proposed concentrations of chemical components for the initial make-up of the Watts solution are 40 oz/gal $NiSO_4 \cdot 6H_2O$ (nickel sulfate hexahydrate), 8 oz/gal $NiCl_2 \cdot 6H_2O$ (nickel chloride hexahydrate), and 6 oz/gal H_3BO_3 (boric acid).
(2) The nickel salts used in the preparation of the Watts solution should be purchased in the form of concentrated liquids rather than crystalline solids.
(3) Nickel salts of the highest purity available in liquid form should be used to prepare the Watts solution.
(4) Southeastern Chemical, Inc. of Chattanooga, Tennessee is the preferred vendor to supply concentrated liquid nickel salts of high purity.

Concentrations. The recommended concentrations of nickel salts and boric acid are the Dremco ideal concentrations shown (boldfaced) in Table 1. These concentrations are within the range of other reference values given in the table. The concentrations suggested by Lockwood Greene are not recommended due to the low level of chlorides. A higher concentration of chloride ions generally results in more rapid corrosion of the anode. Increasing the rate of anode corrosion results in more nickel ions moving into the solution per unit time. This will allow the overall movement of nickel ions from anode to cathode to occur at a faster rate, assuming that all other system parameters are optimal.

Note that these concentrations are recommended as starting points in the optimization process. It will be necessary to fine tune the recommended concentrations to achieve optimal concentrations of the chemical components for the Watts solution as other operating parameters are optimized.

Having established the concentrations of nickel sulfate, nickel chloride, and boric acid for the initial make up of the Watts solution, calculations (Attachment A) were performed to determine the amounts of components required to achieve the established concentrations. Specification sheets (Attachment B) provided information necessary for the calculations. The preparation of a 1,500-gallon batch of Watts solution having the recommended concentrations requires these components:

	Using Crystalline Solids of Nickel Salts	Using Concentrated Liquids of Nickel Salts
nickel sulfate	3,750 pounds	746 gallons
nickel chloride	750 pounds	125 gallons
boric acid	563 pounds	563 pounds
DI water	1,190 gallons	580 gallons
sulfuric acid (pH adjustment)	to be determined	to be determined
sodium hydroxide (pH adjustment)	to be determined	to be determined

The concentrated liquid nickel salts contain nickel sulfate at 133 g/L (17.8 oz/gal) and nickel chloride at 178 g/L (23.5 oz/gal) both as nickel metal.

Physical states. The nickel salts as hexahydrates may be purchased as crystalline solids or as concentrated liquids with the nickel salts in solution. Comparing costs of nickel salts for the crystalline solids and concentrated liquids (presented in Tables 2 and 3), the solids cost slightly less than the liquids. The maximum savings in choosing solids over liquids is about $340 for nickel sulfate and $240 for nickel chloride. These saving are insignificant compared with the costs associated with handling and mixing solids. Additionally, there is the cost of producing 1,190 gallons of DI water needed to put the crystalline solids into solution in comparison with only 580 gallons of DI required with the concentrated liquids. All factors considered, liquid form is the better choice.

Purity. Optimization of the electro-refining process requires that interfering elements be minimized. Potential sources of interfering elements are the chemical components of the Watts solution. By purchasing chemicals of the highest purity available, the minimization of interfering elements is achieved.

Examination of Tables 4-7 shows that generally the nickel salts in concentrated liquid form contain fewer impurities than the crystalline solids. Therefore, nickel salts in concentrated liquid form are a better choice for minimizing interfering elements.

Of the vendors surveyed, Tables 4 and 5 reveal that Southeastern Chemical supplies the highest purity liquid nickel salts. The concentrations of impurities range from a factor of 5 to an order or magnitude lower for Southeastern's products as compared to products of Gumm and VanWaters & Rogers.

After MSC has established criteria for the purity of the nickel product, Southeastern Chemical working with Titan International, the manufacturer of the nickel salts, will attempt to supply nickel salts that meet MSC's criteria with no increase in cost. A certificate of analysis indicating concentrations of contaminants will be provided with each batch.

Suppliers and Costs. Direct comparison of the prices shown in Table 3 for a 1,500-gallon batch is valid because the nickel metal concentrations are equivalent (see Attachment C) in each type of liquid nickel salt sold by all the vendors. Following the recommendations given in items (1)-(4) using prices from Table 3, the total cost of the concentrated liquid nickel salts is $7,372 (including shipment to MSC) if purchased from Southeastern Chemical.

If purchased from Frederick Gumm Chemical, the lowest total price for the nickel salts is $6,057 (FOB Gastonia, NC), $1,300 less than the price quoted by Southeastern Chemical. Despite the $1,300 cost difference, Southeastern Chemical is the preferred vendor for these reasons: (1) the purity of Southeastern's nickel salts is substantially higher and (2) Gumm was slow to respond to inquiries and appeared disinterested in meeting specific needs.

Of all the vendors contacted, Southeastern Chemical responded the most promptly to requests for specifications. Additionally, Southeastern's sales staff demonstrated a thorough knowledge of chemicals used for electroplating, experience in mixing Watts solutions, and a willingness to provide technical support to MSC.

Concentrations of Watts Solution

Table 1. Range of reference Watts solution concentrations

Reference	Concentrations [oz (av)/gal]		
	$NiSO_4 \cdot 6H_2O$	$NiCl_2 \cdot 6H_2O$	H_3BO_3
Dremco[b] (ideal)	40 (300 g/l)	8 (60 g/l)	6 (45 g/l)
Dremco[b] (range)	32-42 (240-315 g/l)	6-12 (45-90 g/l)	6-7 (45-52 g/l)
Lockwood Greene[a]	37 (280 g/l)	6 (44 g/l)	5 (40 g/l)
Dennis and Such[c]	20-53 (150-400 g/l)	3-11 (20-80 g/l)	2-7 (15-50 g/l)
Draper[d]	41 (310 g/l)	7 (50 g/l)	5 (40 g/l)
Graham[e]	40 (300 g/l)	6 (45 g/l)	4-5 (30-37 g/l)

[a] Crotzer, H., Lockwood Greene Technologies, Oak Ridge, Tennessee, letter report to V. MacNair, Manufacturing Sciences Corporation, November 25, 1997.

[B] Robinson, D. J., Dremco, Inc., Phoenix, Arizona, facsimile transmission to S. Sarten, Manufacturing Sciences Corporation, January 6, 1998.

[C] Dennis, J. K. and T. E. Such, *Nickel and Chromium Plating*, 2nd Edition, Butterworths, London, 1986.

[D] Brugger, R., *Nickel Plating*, Robert Draper Ltd., Teddington, England, 1970.

[E] Graham, A. K., *Electroplating Engineering Handbook*, 3rd Edition, Van Nostrand Reinhold Company, New York, 1971.

The ideal concentrations proposed by Dremco, as shown in Table 1, were used to calculate the quantities of chemicals required to make up a 1,500-gallon batch of Watts solution. These quantities and the resulting costs are presented in Tables 2 and 3.

To determine the total cost for preparing the Watts solution, costs must be added to those presented in Tables 2 and 3. These additional costs include the preparation of deionized water and the purchase of sulfuric acid and sodium hydroxide required for pH adjustment. Preparation of the Watts solution from crystalline nickel compounds would require approximately 1,190 gallons of deionized water. Preparation of the Watts solution from liquid nickel solutions would require approximately 580 gallons of deionized water. The quantities of sulfuric acid and sodium hydroxide for pH adjustment must be determined based upon performance of the electro-refining cell.

Watts Solution
Prices and Suppliers of Chemicals

Table 2. Crystalline Solids + Approximately 1,190 Gallons of DI Water

Vendor[a]	$NiSO_4 \cdot 6H_2O$ ($/lb)	Quantity per batch (lbs./1,500 gal.)	Cost[b]
Frederick Gumm Chemical	1.33	3,750	$4,987.50 + shipping (FOB Gastonia, NC)
Southeastern Chemical	1.60	3,750	$6,000.00 + shipping (FOB Chattanooga, TN)
Van Waters & Rogers	1.60	3,750	$6,000.00 + shipping (FOB Rock Hill, SC)
Dremco	no quote	no quote	no quote
Vendor[a]	$NiCl_2 \cdot 6H_2O$ ($/lb)	Quantity per batch (lbs./1,500 gal.)	Cost[b]
Southeastern Chemical	1.65	750	$1,237.50 + shipping (FOB Chattanooga, TN)
Van Waters & Rogers	1.68	750	$1,260.00 + shipping (FOB Rock Hill, SC)
Frederick Gumm Chemical	1.71	750	$1,282.50 + shipping (FOB Gastonia, NC)
Dremco	no quote	no quote	no quote
Vendor[a]	H_3BO_3 ($/lb)	Quantity per batch (lbs./1,500 gal.)	Cost
Southeastern Chemical	0.61	563	$ 343.43 + shipping (FOB Chattanooga, TN)
Van Waters & Rogers	0.81	563	$ 456.03 (includes delivery)
Dremco	1.00	563	$ 563.00 + shipping (FOB Nashville, TN)
Frederick Gumm Chemical	no quote	no quote	no quote

[a] Additional vendor information is found in Attachment D.

[b] Price will increase or decrease with the price of nickel which changes daily.

For pH adjustment

H_2SO_4	$0.0865/lb with a $35 drum deposit (source: Roy Flynn on 1/14/98)
NaOH	$0.1850/lb with a $35 drum deposit (source: Roy Flynn on 1/14/98)

Watts Solution
Prices and Suppliers of Chemicals (continued)

Table 3. Liquids + Approximately 580 Gallons of DI Water

Vendor[a]	$NiSO_4$ solution ($/gallon)	Quantity per batch (gal./1,500 gal.)	Cost[b]
Frederick Gumm Chemical	6.72	746	$5,013.12 + shipping (FOB Gastonia, NC)
Southeastern Chemical	8.18	746	$6,102.28 (includes delivery)
Van Waters & Rogers	8.50	746	$6,341.00 + shipping (FOB Rock Hill, SC)
Dremco	9.00	746	$6,714.00 + shipping (FOB Nashville, TN)
Vendor[a]	$NiCl_2$ solution ($/gallon)	Quantity per batch (gal./1,500 gal.)	Cost[b]
Frederick Gumm Chemical	8.35	125	$1,043.75 + shipping (FOB Gastonia, NC)
Dremco	10.00	125	$1,250.00 + shipping (FOB Nashville, TN)
Southeastern Chemical	10.16	125	$1,270.00 (includes delivery)
Van Waters & Rogers	10.50	125	$1,312.50 + shipping (FOB Rock Hill, SC)

[a] Additional vendor information is found in Attachment D.

[b] Price will increase or decrease with the price of nickel which changes daily.

For pH adjustment

H_2SO_4	$0.0865/lb with a $35 drum deposit (source: Roy Flynn on 1/14/98)
NaOH	$0.1850/lb with a $35 drum deposit (source: Roy Flynn on 1/14/98)

Table 4. Impurities in Concentrated Liquid Nickel Sulfate

Values taken from specification sheets in Attachment B	Suppliers		
	Frederick Gumm	Van Waters & Rogers	Southeastern
	Manufacturers		
	Palm[a]	Atotech	Titan[b]
cobalt	not detected	not specified	< 1 ppm typical[c]
iron	3 ppm max	10 ppm max < 5ppm typical	0.9 ppm measured
copper	2 ppm max	2 ppm max < 1 ppm typical	0.4 ppm measured
zinc	1 ppm max	1 ppm max < 1 ppm typical	not detected
chromium	5 ppm max	5 ppm max < 1 ppm	1.4 ppm measured
manganese	1 ppm max	5 ppm max < 5 ppm typical	not analyzed
cadmium	< 1 ppm max	1 ppm max < 1 ppm typical	0.2 ppm measured
lead	< 1 ppm max	3 ppm max < 1 ppm typical	not detected

Table 5. Impurities in Concentrated Liquid Nickel Chloride

Values taken from specification sheets in Attachment B	Suppliers		
	Frederick Gumm	Van Waters & Rogers	Southeastern
	Manufacturers		
	Palm[a]	Atotech	Titan[b]
cobalt	120 ppm max	not specified	< 1 ppm typical[c]
iron	10 ppm max	20 ppm typical	0.1 ppm measured
copper	1 ppm max	10 ppm max < 10 ppm typical	not detected
zinc	1 ppm max	10 ppm max < 10 ppm typical	0.1 ppm measured
chromium	10 ppm max	10 ppm max < 10 ppm typical	0.6 ppm measured
manganese	not specified	not specified	not specified
cadmium	1 ppm max	not specified	not specified
lead	1 ppm max	10 ppm max < 5 ppm typical	0.3 ppm measured

[a] The specifications for the concentrated liquids offered by Dremco are the same as those obtained from Palm,Frederick Gumm's manufacturer. However, Dremco sells them for a higher price than Frederick Gumm (see Table 3).

[B] After MSC has established criteria for the purity of the nickel product, Southeastern working with Titan have agreed to supply nickel salts that meet MSC's criteria with no increase in cost.

[c] This value does not appear on the Titan specification sheets. It was obtained during a telephone conversation on Tuesday, February 24, 1998, with the Titan representative, Ellen Jansen.

Table 6. Impurities in Crystalline Solid Nickel Sulfate

Values taken from specification sheets in Attachment B	Suppliers: Frederick Gumm Manufacturers: Palm	Suppliers: Van Waters & Rogers Manufacturers: Atotech	Suppliers: Southeastern Manufacturers: OMG
cobalt	150 ppm max	not specified	150 ppm max < 10 ppm measured
iron	10 ppm max	15 ppm max 10 ppm typical	10 ppm max < 1 ppm measured
copper	5 ppm max	5 ppm max < 5 ppm typical	5 ppm max < 1 ppm measured
zinc	5 ppm max	5 ppm max 2 ppm typical	5 ppm max < 1 ppm measured
chromium	10 ppm max	1 ppm max < 1 ppm typical	10 ppm max < 5 ppm measured
manganese	5 ppm max	3 ppm max 2 ppm typical	not analyzed
cadmium	1 ppm max	1 ppm max < 1 ppm typical	1 ppm max < 1 ppm measured
lead	5 ppm max	2 ppm max 1 ppm typical	1 ppm max < 1 ppm measured

Table 7. Impurities in Crystalline Solid Nickel Chloride

Values taken from specification sheets in Attachment B	Suppliers: Frederick Gumm Manufacturers: Palm	Suppliers: Van Waters & Rogers Manufacturers: Atotech	Suppliers: Southeastern Manufacturers: Titan
cobalt	200 ppm max	200 ppm max	200 ppm typical
iron	100 ppm max	100 ppm max	100 ppm typical
copper	20 ppm max	20 ppm max	20 ppm typical
zinc	20 ppm max	20 ppm max	20 ppm typical
chromium	not specified	not specified	not specified
manganese	not specified	not specified	not specified
cadmium	50 ppm max	50 ppm max	not specified
lead	20 ppm max	20 ppm max	20 ppm typical

ATTACHMENT A

Calculation of Weights/Volumes of Watts Solution Components

Recommended concentrations for Watts solution taken from Table 1 (boldfaced) and given below.

$$\left.\begin{array}{l} ①\ \frac{40\,oz\,(av)}{gal}\ NiSO_4\cdot 6H_2O \\ ②\ \frac{8\,oz\,(av)}{gal}\ NiCl_2\cdot 6H_2O \\ ③\ \frac{6\,oz\,(av)}{gal}\ H_3BO_3 \end{array}\right\} \text{Basis} = 1500\ \text{gals total makeup Watts solution}$$

Calculations to obtain weights (lbs) of each chemical component needed to achieve the recommended concentrations:

① $NiSO_4\cdot 6H_2O$: $\frac{40\,oz}{gal}$ = recommended conc.

$$\frac{40\,oz}{gal} = \frac{x_1}{1500\,gals} \Longrightarrow x_1 = 60{,}000\,oz \left|\frac{1\,lb}{16\,oz}\right. = \boxed{3750\ \text{lbs in } 1500\ \text{gals}}$$

② $NiCl_2\cdot 6H_2O$: $\frac{8\,oz}{gal}$ = recommended conc.

$$\frac{8\,oz}{gal} = \frac{x_2}{1500\,gals} \Longrightarrow x_2 = 12{,}000\,oz \left|\frac{1\,lb}{16\,oz}\right. = \boxed{750\ \text{lbs in } 1500\ \text{gals}}$$

③ H_3BO_3: $\frac{6\,oz}{gal}$ = recommended conc.

$$\frac{6\,oz}{gal} = \frac{x_3}{1500\,gals} \Longrightarrow x_3 = 9{,}000\,oz \left|\frac{1\,lb}{16\,oz}\right. = \boxed{\begin{array}{l}563\,lbs\\ \text{in } 1500\\ gals\end{array}}$$

Calculations to obtain the volumes (gal
concentrated liquid nickel salt solutio
to be purchased:

$NiSO_4 \cdot 6H_2O$: 3750 lbs (see first page of this Attachment) in 15
purchased conc. = 5.03 $\frac{lbs}{gal}$ (see Attachment

$$3750\ lbs \ \Big|\ \frac{gal}{5.03\ lbs} =$$

746 gals concentrate
liquid nickel sulfat
hexahydrate to b
purchased

$NiCl_2 \cdot 6H_2O$: 750 lbs (see first page of this Attachment) in 15
purchased conc. = 6.00 $\frac{lbs}{gal}$ (see Attachment

$$750\ lbs \ \Big|\ \frac{gal}{6.00\ lbs} =$$

125 gals concentrate
liquid nickel chlori
hexahydrate to b
purchased

Dremco, Inc
Specialized Products for Electrowinning
1550 E Missouri Ave, Ste 302, Phoenix AZ 85014
Tel. 602-234-8908, Fax 602-234-8909

Fax Transmission

Date: 1/6/98 1:30 PM

To: Mr Steve Sarten
Manufacturing Science
Phone: 423-481-0455
Fax: 423-481-3142 Pages: 1

Dear Steve

Subject: Supply of Watts Nickel Bath Chemicals

The Watts Nickel bath is comprised of three principal components,

component	conc. range	ideal conc.
$NiSO_4$	32-42 oz / gal	40 oz / gal = 300g/l
$NiCl_2$	6-12 oz / gal	8 oz / gal = 60g/l
Boric Acid	6-7 oz / gal	6 oz / gal = 45g/l

For your 1500 gal batch, you could purchase these components in liquid form, as follows:

component	vol. req'd	cost	
$NiSO_4$	~~600 gal~~ 746	$5400	$9/gal
$NiCl_2$	~~140 gal~~ 125	$1400	$10/gal
Boric Acid	~~600 lbs~~ 563	$600	$1/lb
Total Cost		$7400	

The solutions are available in drums, fob Nashville.

Sincerely,

Dr. Douglas J. Robinson

746 gals
125 gals
563 lbs
} obtained using the ideal concs. from above (40, 8, 6)

---

600 gals
140 gals
600 lbs
} obtained using
32 oz/gal $NiSO_4 \cdot 6H_2O$
7 oz/gal $NiCl_2 \cdot 6H_2O$
6.4 oz/gal H_3BO_3

rounded up from 563?

page 12

Crystalline Nickel Salts

Volume DI H_2O needed for 1500gals. solution

Chemical Component	Density (lb/gal)	Weight(lbs)* in 1500gals. solution	Volume(gals)* in 1500gals. solution
$NiSO_4 \cdot 6H_2O$	17.3	3750	217
$NiCl_2 \cdot 6H_2O$	16.7	750	45
H_3BO_3	11.9	563	47

$$**\text{Volume}_1 = \frac{\text{Weight}}{\text{Density}} = \frac{3750\,\text{lbs}}{17.3\,\text{lb/gal}} = 217\,\text{gals.}$$

$$\text{Volume}_2 = \frac{750\,\text{lbs}}{16.7\,\text{lbs/gal}} = 45\,\text{gals}$$

$$\text{Volume}_3 = \frac{563\,\text{lbs}}{11.9\,\text{lbs/gal}} = 47\,\text{gals}$$

Σ Volumes$_{1-3}$ = 309 gals. = volume of chem. components

Volume of H_2O = 1500gals − 309gals.
= 1191 gals ≈ 1190 gals. H_2O for 1500gals. solution make up

* See 1st 2 pages of this attachment.

Concentrated Liquid Nickel Salts
Volume DI H_2O needed for 1500gals. Solution

Chemical component	Volume* (gals) needed for 1500gals. solution
$NiSO_4 \cdot 6H_2O$	746
$NiCl_2 \cdot 6H_2O$	125
H_3BO_3**	47

$\Sigma V's = 918 \text{ gals} \approx 920 \text{ gals.}$

H_2O needed $= (1500 - 920)$ gals.

$=$ 580 gals. DI H_2O needed for 1500 gals. solution makeup

* See 3rd page of this attachment

** Occupied volume of H_3BO_3. See 5th page of this attachment.

ATTACHMENT B

Price Quotes and Specification Sheets

Dremco, Inc
Specialized Products for Electrowinning
1550 E Missouri Ave, Ste 302, Phoenix AZ 85014
Tel. 602-234-8908, Fax 602-234-8909

Fax Transmission

Date:	1/6/98	1:30 PM
To:	Mr Steve Sarten Manufacturing Science	
Phone:	423-481-0455	
Fax:	423-481-3142	Pages: __1_

Dear Steve

Subject: Supply of Watts Nickel Bath Chemicals

The Watts Nickel bath is comprised of three principal components,

<u>component</u>	<u>conc. range</u>	<u>ideal conc.</u>
NiSO4	32-42 oz / gal	40 oz / gal
NiCl2	6-12 oz / gal	8 oz / gal
Boric Acid	6-7 oz / gal	6 oz / gal

For your 1500 gal batch, you could purchase these components in liquid form, as follows:

component	vol. req'd	cost
NiSO4	600 gal	$5400
NiCl2	140 gal	$1400
Boric Acid	600 lbs	<u>$600</u>
Total Cost		**$7400**

The solutions are available in drums, fob Nashville.

Sincerely,

Dr. Douglas J. Robinson

Dremco Inc.
Specialized Products for Electrowinning and Electrorefining
17020 N. 32nd Street, Phoenix Az, 85032
Tel 602-787-8105, Fax 602-787-8106
Email: dremco@syspac.com
Dr. Douglas J Robinson, President

Fax Transmission From: * 602-234-8909* No. of pages on this fax =
January 22, 1998 4:58p

Libbie Terry
FAX: 423-481-3142

Dear Libbie,

* Re: Spec Sheets*

Please find attatched the spec sheets that you requested on Boric Acid, Nickel Sulfate and Nickel Chloride.

Sincerely,

Dr. Douglas J Robinson

Manufacturing Specification for

LIQUID NICKEL SULFATE

CONCENTRATION: 133.0 to 135.0 g/L (17.8 oz/gal) by weight nickel metal

APPEARANCE: Sparkling clear, dark green liquid

CONTAMINANTS: mg/L by weight, maximum

Cobalt	none detected
Cadmium	<1
Calcium and Magnesium	50
Chromium	5
Copper	2
Iron	.3
Lead	<1
Manganese	1
Zinc	1
Insoluble matter	none detected

pH: 3.0 to 4.0

SPECIFIC GRAVITY: 1.339 to 1.345

METHODS: Assay by EDTA titration.

Appearance by visual inspection.

Metallic contaminants by AA spectro-photometry and/or anodic polarography.

Sulfides by cathodic polarography from +0.2 to 1.0 volts.

Insoluble matter by filtration through a No. 42 Whatman filter.

INSPECTION LEVEL: Each production batch.

Manufacturing Specification for

LIQUID NICKEL CHLORIDE

CONCENTRATION: 178 - 181g/L (23.5oz/gal min) by weight nickel metal.
13.3% minimum as Nickel+Cobalt (Ni+Co)
6 lbs/gal as $NiCl_2 \cdot 6H_2O$

APPEARANCE: Sparkling clear, dark green liquid.

CONTAMINANTS: mg/L by weight, maximum

Arsenic	5	Cadmium	1
Calcium	60	Chromium	10
Cobalt	120	Copper	1
Iron	10	Lead	1
Magnesium	20	Nitrates	<10
Tin	5	Vanadium	5
Zinc	1	Insolubles	non detected

pH: 2.0 - 3.0

% FREE ACID: <1.0

SPECIFIC GRAVITY: 1.330-1.350

METHODS: Assay by EDTA titration
Appearance by visual inspection
Metallic contaminants by Flame AAS and/or anodic polarography
Insolubles by filtration through a No. 42 Whatman filter.

INSPECTION LEVEL: Each production batch

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Purchasing Specification for

56% Boric Acid

Item/Property	Product Specification
B_2O_3, %	56.3 - 56.8
Equivalent H_3BO_3, %	99.9-100.9
SO_4	<= 450
Cl, ppm	<= 18
Iron, ppm	<= 6

A CERTIFICATE OF ANALYSIS MUST ACCOMPANY EACH SHIPMENT.

FREDERICK GUMM CHEMICAL CO., INC.
2098 CHESPARK DRIVE
GASTONIA, NORTH CAROLINA 28052
(704) 865-2800 OUTSIDE N.C. 1-800-541-7878

FAX #: (704) 866-9006

FACSIMILE TRANSMISSION COVER SHEET

DATE: 1-14-98

ATTENTION: Libby

COMPANY:

FROM: Cynthia

TOTAL # OF PAGES: 6 **(including cover sheet)**

REMARKS:

page 21

Gumm

January 14, 1998

MANUFACTURING SCIENTIST

Attn: Libby Terry

RE: Pricing for nickel sulfate and chloride

Dear Libby,

Per our conversation the pricing we discuss is as follows:

NICKEL SULFATE LIQUID	55gal dr	$6.72/gal
NICKEL SULFATE CRYSTAL	55lb bag	$1.33/lb
NICKEL CHLORIDE LIQUID	55gal dr	$8.35/gal
NICKEL CHLORIDE CRYSTAL	44lb bag	$1.71/lb

*Also accompanying this quotation are the specification sheets for each item quoted.

If you should have any further questions, please give me a call at 1-800-541-7878.

Regards,

FREDERICK GUMM CHEMICAL CO.

Cynthia Costner

Cynthia Costner
Computer Operator

CC/s

** NOTE THE PRICE OF NICKEL CHANGES DAILY- PRICE COULD INCREASE OR DECREASE **

FREDERICK GUMM CHEMICAL COMPANY, INC.
538 Forest Street, Kearny, New Jersey 07032 • 1-201-991-4171
Fax: 201-991-5855

PALM
INTERNATIONAL, INC.

PALM INTERNATIONAL, Inc.	MANUFACTURING SPECIFICATION
SPECIFICATION #: PMS-1	ISSUE DATE: 12-1-92
APPROVED BY:	REVISION DATE: 07-21-97
TITLE:	REVISON #: 5

Manufacturing Specification for

LIQUID NICKEL SULFATE

CONCENTRATION: 133.0 to 135.0 g/L (17.8 oz/gal) by weight nickel metal

APPEARANCE: Sparkling clear, dark green liquid

CONTAMINANTS: mg/L by weight, maximum

Cobalt	none detected
Cadmium	<1
Calcium and Magnesium	50
Chromium	5
Copper	2
Iron	3
Lead	<1
Manganese	1
Zinc	1
Insoluble matter	none detected

pH: < 3.0 to 4.0

SPECIFIC GRAVITY: 1.339 to 1.345

METHODS: Assay by EDTA titration.

Appearance by visual inspection.

Metallic contaminants by AA spectro-photometry and/or anodic polarography.

Sulfides by cathodic polarography from +0.2 to 1.0 volts.

Insoluble matter by filtration through a No. 42 Whatman filter.

INSPECTION LEVEL: Each production batch.

PALM
INTERNATIONAL, INC.

PALM INTERNATIONAL, Inc.	MANUFACTURING SPECIFICATION
SPECIFICTATION #: PMS-4	ISSUE DATE: 07-19-94
APPROVED BY:	REVISION DATE: 07-21-97
TITLE:	REVISION #: 3

Manufacturing Specification for

LIQUID NICKEL CHLORIDE

CONCENTRATION: *178 - 181g/L (23.5oz/gal min) by weight nickel metal*
13.3% minimum as Nickel+Cobalt (Ni+Co)
6 lbs/gal as $NiCl_2 \cdot 6H_2O$

APPEARANCE: *Sparkling clear, dark green liquid.*

CONTAMINANTS: mg/L by weight maximum

Arsenic	5	Cadmium	1
Calcium	60	Chromium	10
Cobalt	120	Copper	1
Iron	10	Lead	1
Magnesium	20	Nitrates	<10
Tin	5	Vanadium	5
Zinc	1	Insolubles	non detected

pH: 2.0 - 3.0

% FREE ACID: <1.0

SPECIFIC GRAVITY: 1.330-1.350

METHODS: Assay by EDTA titration
Appearance by visual inspection
Metallic contaminants by Flame AAS and/or anodic polarography
Insolubles by filtration through a No. 42 Whatman filter.

INSPECTION LEVEL: Each production batch

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PALM INTERNATIONAL, Inc.	PURCHASING SPECIFICATION
SPECIFICATION #: PPS-1	ISSUE DATE: 07-19-94
APPROVED BY:	REVISION DATE: 07-07-97
TITLE: Q.C. Manager	REVISION #: 1

Purchasing Specification for

NICKEL SULFATE CRYSTALS

Item/Property	Product Specification
% Ni	22.0 min
% Insoluble	0.015 max
ppm Co	150 max
ppm Fe	10 max
ppm Cu	5 max
ppm Zn	5 max
ppm Ca	10 max
ppm Mg	50 max
ppm Mn	5 max
ppm Na	20 max
ppm Cl	5 max
ppm Cr	10 max
ppm Al	25 max
ppm Cd	1 max
ppm Pb	5 max

CERTIFICATE OF ANALYSIS MUST ACCOMPANY EACH SHIPMENT.

I VERIFY ALL MATERIALS SUPPLIED BY ____________________ WILL MEET PALM'S PURCHASING SPECIFICATION.

NAME:____________________ TITLE:____________________

page 25

PALM
INTERNATIONAL INC.

PALM INTERNATIONAL, Inc.	PURCHASING SPECIFICATION
SPECIFICATION #: PPS-2	ISSUE DATE: 07-30-92
APPROVED BY:	REVISION DATE: 11-17-97
TITLE: QC Manager	REVISION #: 4

Purchasing Specification for

NICKEL CHLORIDE CRYSTAL

FORMULA: $NiCl_2 \cdot 6H_2O$

ASSAY: 24.0 to 25.5 percent by weight as nickel

MAXIMUM ALLOWABLE CONTAMINANTS:

Cadmium	0.0050 %
Calcium	0.0050 %
Cobalt	0.0200 %
Copper	0.0020 %
Iron	0.0100 %
Lead	0.0020 %
Zinc	0.0020 %
Insoluble matter	less than 0.05 %

CERTIFICATE OF ANALYSIS MUST ACCOMPANY EACH SHIPMENT.

I VERIFY ALL MATERIALS SUPPLIED BY ______________________ WILL MEET PALM'S PURCHASING SPECIFICATION.

NAME:____________________ TITLE:____________________

page 26

FAST FAX

Facsimile Transmittal Cover Sheet

FROM:
SOUTHEASTERN CHEMICAL, INC.
1512-A Sholar Avenue
Chattanooga, TN 37406
Fax: 423-698-5152
Ph: 423-622-5154

To Manufacturing Sciences Date 1/14/98

Attention Libby Terry Page 1 of 1

From Sandi Guess

Subject Quotation

Message Dear Ms. Terry,

Per your request, we are pleased to offer the following quotation for chemicals for your new nickel bath. These prices are good for the month of January only due to nickel fluctuation.

NICKEL SULFATE	50 lb. bag	$1.60/lb.
NICKEL CHLORIDE	44 lb. bag	$1.65/lb.
BORIC ACID	50 lb. bag	$0.61/lb.

FOB: Chattanooga, TN

We appreciate the opportunity to offer this quotation. If we can be of further service, please feel free to contact us at any time.

Sincerley,
SOUTHEASTERN CHEMICAL, INC.
Sandi Guess
Sandi Guess

page 27

FAST FAX

Facsimile Transmittal Cover Sheet

FROM:
SOUTHEASTERN CHEMICAL, INC.
1512-A Sholar Avenue
Chattanooga, TN 37406

Fax: 423-698-5152
Ph: 423-622-5154

To Manufacturing Sciences Date 2/26/98

Attention Libby Terry Page 1 of 1

From Sandi Guess

Subject Quotation

Message Dear Ms. Terry,

We are pleased to offer the following quotation for liquid nickel sulfate and chloride. These prices are FOB: Delivered to your plant.

LIQUID NICKEL SULFATE
Packaging: 55 gl.drum
PRICE: $8.18/gal.

LIQUID NICKEL CHLORIDE
Packaging: 55 gl.drum
PRICE: $10.16/gal.

We hope you will find the above of interest. If we can be of further service, please feel free to contact us at any time.

Sincerely,
SOUTHEASTERN CHEMICAL, INC.
Sandi Guess
Sandi Guess

page 28

FAST FAX

Facsimile Transmittal Cover Sheet

FROM:
Southeastern Chemical, Inc.
1512 - A Sholar Avenue
Chattanooga, TN 37406

Fax: 423-698-5152
Ph: 423-622-5154

To MANUFACTURING SCIENCES Date 2/20/98

Attention LIBBY TERRY Page 1 of 5

From SANDI GUESS

Subject TYPICAL ANALYSIS Ni SULFATE & Ni CHLORIDE

Message

page 29

TITAN
INTERNATIONAL, INC.

Liquid Nickel Sulfate

Electroless Grade

Certificate of Analysis

Lot No: 8000LS

Nickel,	g/L	133.31
pH,		3.03
Specific Gravity,		1.345
Cadmium,	mg/L	0.2
Calcium,	mg/L	12.9
Chromium,	mg/L	1.4
Cobalt,	mg/L	ND
Copper,	mg/L	0.4
Iron,	mg/L	0.9
Lead,	mg/L	ND
Magnesium,	mg/L	2.7
Zinc,	mg/L	ND
Insoluble matter,		ND

January 20, 1998

205 Chester Avenue, Suite 100 Moorestown, New Jersey 08057
609-786-1147 Fax: 609-786-3978

page 30

TITAN
INTERNATIONAL, INC.

Liquid Nickel Chloride

Certificate of Analysis

Lot No: 8011LC

Nickel,	g/L	179.38
Nickel,	% by wt.	13.4729
Specific Gravity		1.345
pH		2.31
Iron,	ppm	0.1
Copper,	ppm	ND
Zinc,	ppm	0.1
Chromium,	ppm	0.6
Lead,	ppm	0.3

February 18, 1998

Cobalt never > 1ppm ~~or 1mg/l~~ or 1mg/l } on phone 2-24-98

information from
Ellen Jansen
800-435-4644
at Titan

205 Chester Avenue, Suite 100 Moorestown, New Jersey 08057
609-786-1147 Fax: 609-786-3978

page 31

OMG P.O. Box 111, Franklin, PA. 16323 Phone: 814-432-2125 Fax: 814-432-7645

OM GROUP, INC.
Kokkola Chemicals Oy
Mooney Chemicals, Inc.
Vassel S.A.

November 11, 1997

PALM COMMODITIES INTERNATIONAL
1289 BRIDGESTONE PARKWAY

LA VERGNE TN 37086

Attention: RECEIVING - QUALITY CONTROL

CERTIFICATE OF ANALYSIS

Date Shipped . . . . : 11-12-97
Product. . . . . . . : 22% NICKEL SULFATE CRYSTALS
Customer P.O. Number.: 13317
Customer Number. . . : 043406 Order Number: 42629
Quantity Shipped . . : 630 BAGS
Customer Product Code: 2040048
OMG product code . . : 00571 K2

ITEM/PROPERTY	CUSTOMER SPECIFICATION	BATCH/LOT # N727202-14 K2	BATCH/LOT # N727202-15 K2	BATCH/LOT # N727202-17 K2	BATCH/LOT # N727202-18 K2	BATCH/LOT # N727203-1 K2	BATCH/LOT # N727203-2 K2
Date Produced		07/21/97	07/21/97	07/21/97	07/21/97	07/22/97	07/22/97
Date Expires		07/21/02	07/21/02	07/21/02	07/21/02	07/22/02	07/22/02
Revision Date	03-03-97						
% Nickel	22.1 Min.	22.3	22.3	22.3	22.3	22.3	22.3
% Insoluble	0.015 Max.	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
ppm Cobalt	150 Max.	<10	<10	<10	<10	<10	<10
ppm Iron	10 Max.	<1	<1	<1	<1	<1	<1
ppm Copper	5 Max.	<1	<1	<1	<1	<1	<1
ppm Zinc	5 Max.	<1	<1	<1	<1	<1	<1
ppm Calcium	10 Max.	<1	<1	<1	<1	<1	<1
ppm Magnesium	50 Max.	5	5	5	5	5	5
ppm Sodium	25 Max.	<10	<10	<10	<10	<10	<10
ppm Lead	1 Max.	<1	<1	<1	<1	<1	<1
ppm Chromium	10 Max.	<5	<5	<5	<5	<5	<5
ppm Cadmium	1 Max.	<1	<1	<1	<1	<1	<1

The material designated in this certificate has been manufactured in accordance with standard manufacturing procedures and meets all specifications contained in the above purchase order.

Manufactured at: OMG - Kokkola Chemicals Oy, Kokkola, Finland

James M. Daugherty - Quality Assurance Supervisor
Phone: (814) 432-2125 Ext. 102

OMG Franklin Plant
[illegible]
[illegible]

Inorganic Metallic Salts Metal Powders Metal Carboxylates

TITAN
INTERNATIONAL, INC.

Product: Nickel Chloride Hexahydrate

TYPICAL ANALYSIS

ELEMENT	PERCENTAGES
Nickel	24.0 %
Lead	.002 %
Zinc	.002 %
Copper	.002 %
Iron	.01 %
Cobalt	.02 %
Insolubles	.05 %

page 33

January 14, 1998

Van Waters & Rogers Inc.
A ROYAL PAKHOED COMPANY

Three Riverside Lane
Chattanooga, TN 37406-1725
PHONE (423) 696-0231
FAX (423) 624-4190

Libby Terry
Manufacturing Sciences Corp.
804 Kerr Hollow Road
Oak Ridge, TN 37830

Dear Libby,

Thank you for the opportunity to quote on the following chemicals. I hope you will find our prices to be competitive and we will be given the chance to provide Manufacturing Sciences with the same quality service and expertise that our customers have come to depend on. If you have any further questions or would like to place an order please contact me.

Product	Package	Price	
Nickel Sulfate Spec. 101Granular	50# bags	1.60/lb	FOB: Rock Hill, SC
Nickel Sulfate Liquid	55 gl dr	8.50/gl	FOB: Rock Hill, SC
Nickel Chloride Crystal	20 kil bg	3.70/ki	FOB: Rock Hill, SC
Nickel Chloride Liquid	55 gl dr	10.50/gl	FOB: Rock Hill, SC
Boric Acid Tech Gran	50# bags	.81/lb	Delivered

Sincerely,

Priscilla Pennington

Priscilla Pennington
Sales Representative

Van Waters & Rogers Inc.
subsidiary of **Univar**

3 RIVERSIDE LANE
CHATTANOOGA, TN 37406
PHONE (615) 698-0231
(800) 233-0693
FAX (615) 624-4190

fax transmittal

to:	Libby
fax:	
from:	Priscilla Pennington
date:	2/23/98
re:	nickle chloride & sulfate
pages:	(including cover sheet) 9

NOTES:

We are the first choice.
We anticipate and provide the best in customer-valued distribution services.

FEB 23 '98 12:34 FR VWR-CHATTANOOGA 423 624 4190 TO 14234813142 P.01

page 35

Sheet No. NI-SUL-L
Revision: 4/24/97

Technical Information

NICKEL SULFATE LIQUID

Atotech Nickel Sulfate, Liquid, EN/EG was specifically developed to meet the needs of the electroless nickel and electrolytic nickel industry where very low impurity levels are required. It is produced from high purity origin nickel powder which guarantees a high purity and consistent product.

Specifications:

	Specification	Typical
$NiSO_4 \cdot 6H_2O$	5.0 lb/gal	5.0 lb/gal
Ni + Co as Ni	10.0 % min.	10.25 %
Fe	10 ppm max.	< 5 ppm
Cu	2 ppm max.	< 1 ppm
Pb	3 ppm max.	< 1 ppm
Mn	5 ppm max.	< 5 ppm
Cr	5 ppm max.	< 1 ppm
Zn	1 ppm max.	< 1 ppm
Cd	1 ppm max.	< 1 ppm
Se	1 ppm max.	< 1 ppm
As	1 ppm max.	< 1 ppm
Bi	1 ppm max.	< 1 ppm
Ca	10 ppm max.	< 7 ppm
Ca + Mg as Ca	50 ppm max.	<50 ppm

Properties & Product Characteristics

Clear green liquid

Density:	Approx.	11 lbs/gal
pH:	Liquid	4.0 - 5.0
Particulate matter in Electropure only:		<2 microns

atotech

ATO

Atotech USA Inc., 1750 Overview Drive, P.O. Box 12000, Rock Hill, SC 29731-2000
Telephone: 803-817-3500 • Fax: 803-817-3666

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Packaging:

55 Gallon Fiber Drum
5 Gallon Polyethylene inner cubes in an outer liner
4 Drums per pallet; 36 cubes per pallet.

NOTE: Drums to be shipped and stored at temperatures above 60°F to avoid possibility of crystallization taking place.

WASTE DISPOSAL

This material must be disposed of in accordance with all applicable federal, state, and local regulations. Consult the MSDS for additional regulatory information.

GENERAL SAFETY PRECAUTIONS

Avoid direct contact with this material. Do not inhale associated mist, vapors, and/or dust. As applicable, keep exposure below the limits recommended by OSHA, ACGIH, the manufacturer, and others. Wash contaminated clothing before reuse. Always comply with the Hazard Communication Standard, 29CFR1910.1200; emergency showers and eyewashes must be available.

It is recommended that the plating chemistry product(s) referred to in this Technical Information sheet be used: (1) in accordance with the provided in product specific MSDS; and (2) in compliance with all appropriate requirements and guidelines established by OSHA, NIOSH, ACGIH, NFPA, and others.

FIRST-AID RECOMMENDATIONS

Types of Exposure	Recommendations
Eye	Flush with water for at least 15 minutes.
Skin	Wash thoroughly with soap and water.
Inhalation	Remove to fresh air immediately.
Ingestion	Give water or milk (if conscious and not having convulsions).

REVIEW MSDS BEFORE USING THIS PLATING CHEMISTRY AND FOR PRODUCT SPECIFIC INFORMATION. A precautionary approach should be used when there is potential for chemical exposure – this includes minimizing exposure potential, rapid decontamination, and medical follow-up.

NOTE: A Material Safety Data Sheet (MSDS) for this product is available on request from Atotech USA Inc., Customer Service/Sales Support Group, 1750 Overview Drive, P.O. Box 12000, Rock Hill, SC 29731-2000.

TELEPHONE NUMBERS

Customer Service/Sales Support Group: (803) 817-3538 (MSDS requests)
Product Safety Department: (803) 817-3649 (regulatory inquiries and emergencies)

TO PLACE AN ORDER: 1-800-PLATING
T350

page 37

Sheet No: Nickel Chloride Liquid
Revision: 3/9/93

Technical Information

NICKEL CHLORIDE LIQUID

Atotech Nickel Chloride Liquid was specifically developed to meet the needs of the nickel plating industry where very low impurities are required. It is produced from a high purity virgin nickel powder which guarantees a high quality and consistent product.

Specifications

	Specification	Typical*
$NiCl_2 6H_2O$	6 lbs/gal minimum	6.05 lb/gal
$Ni + Co (as Ni)_2$	-	13.3% by wt.
Fe*	-	20 ppm
Cu*	10 ppm maximum	<10 ppm
Zn*	10 ppm maximum	<10 ppm
Cr*	10 ppm maximum	<10 ppm
Pb*	10 ppm maximum	< 5 ppm

* Basis Nickel Chloride Hexahydrate Crystal having the formula $NiCl_2 6H_2O$

Properties & Product Characteristics

Clear green liquid

pH	2.0 - 2.5
Specific Gravity	1.35
Density	11.2 lbs/gal

"IMPORTANT NOTICE REGARDING THIS INFORMATION:
The statements, technical information, and recommendations contained in this document are based on tests and data that are believed to be reliable. Further, as the actual use of our products by others is beyond our control, no guarantee of any kind is made as to the effects of such use, or the results to be obtained, whether the use is made in accordance with the recommendations or suggestions contained herein or otherwise. This document is not contractual and NOTHING HEREIN CONSTITUTES A REPRESENTATION OR WARRANTY THAT THE GOODS DESCRIBED ARE FIT FOR A PARTICULAR PURPOSE OF A CUSTOMER or that their use does not conflict with any existing patent rights. The exclusive source of any warranty and of any other customer rights whatsoever is on the Atotech Invoice. Also, since this data sheet may be provided by electronic media, Atotech cannot guarantee the accuracy or originality hereof. Any alterations made to this document other than by Atotech corporate headquarters is expressly prohibited."

atotech
ATO

Atotech USA Inc., 1750 Overview Drive, P.O. Box 12000, Rock Hill, SC 29731-2000
Telephone: 803-817-3500 • Fax: 803-817-3666

NICKEL CHLORIDE LIQUID

Packaging

5 gallon cubitainer
55 gallon drum

WASTE DISPOSAL

This material must be disposed of in accordance with all applicable federal, state, and local regulations. Consult the MSDS for additional regulatory information.

GENERAL SAFETY PRECAUTIONS

Avoid direct contact with this material. Do not inhale associated mist, vapors, and/or dust. As applicable, keep exposure below the limits recommended by OSHA, ACGIH, the manufacturer, and others. Wash contaminated clothing before reuse. Always comply with the Hazard Communication Standard, 29CFR1910.1200; emergency showers and eyewashes must be available.

It is recommended that the plating chemistry product(s) referred to in this Technical Information sheet be used: (1) in accordance with the information provided in product specific MSDS; and (2) in compliance with all appropriate requirements and guidelines established by OSHA, NIOSH, ACGIH, NFPA, and others.

FIRST-AID RECOMMENDATIONS

Types of Exposure	Recommendations
Eye	Flush with water for at least 15 minutes.
Skin	Wash thoroughly with soap and water.
Inhalation	Remove to fresh air immediately.
Ingestion	Give water or milk (if conscious and not having convulsions). Remove to fresh air immediately.

REVIEW MSDS BEFORE USING THIS PLATING CHEMISTRY AND FOR PRODUCT SPECIFIC INFORMATION. A precautionary approach should be used when there is potential for chemical exposure -- this includes minimizing exposure potential, rapid decontamination, and medical follow-up.

NOTE: A Material Safety Data Sheet (MSDS) for this product is available on request from Atotech USA Inc., Customer Service/Sales Support Group, 1750 Overview Drive, P.O. Box 12000, Rock Hill, SC 29731-2000.

TELEPHONE NUMBERS

Customer Service/Sales Support Group: (803) 817-3538 (MSDS requests)
Product Safety Department: (803) 817-3549 (regulatory inquiries and emergencies)

TO PLACE AN ORDER: 1-800-PLATING

page 39

Sheet No: NICKEL SULFATE CRYSTAL
Revision: 4/17/96

Technical Information

NICKEL SULFATE CRYSTAL

Atotech Nickel Sulfate, Crystal, Electropure® Spec 103 was specifically developed to meet the needs of the electrolytic and electroless nickel industry where very low impurity levels are required. It is produced from high purity virgin nickel powder which guarantees a high purity and consistent product. Nickel Sulfate Crystal Spec 101 is of Belgium origin and meets most nickel plating applications.

Specifications:

	Specification ELECTROPURE™ (Spec 103):	Typical	Specification Spec 101:	Typical
Ni+Co as Ni	22.0% min.	22.3%	> 22.0% min.	22.1%
Co	-	-	<200 ppm	50 ppm
Fe	15 ppm max.	10 ppm	< 10 ppm	2 ppm
Cu	5 ppm max.	<5 ppm	< 5 ppm	2 ppm
Pb	2 ppm max.	1 ppm	< 5 ppm	2 ppm
Mn	3 ppm max.	2 ppm	-	-
Cr	1 ppm max.	<1 ppm	-	-
Zn	5 ppm max.	2 ppm	< 5 ppm	2 ppm
Cd	1 ppm max.	<1 ppm	< 5 ppm	2 ppm
Se	1 ppm max.	<1 ppm	-	-
As	1 ppm max.	<1 ppm	-	-
Bi	1 ppm	<1 ppm	-	-
Ca	50 ppm	20 ppm	-	-
Mg	50 ppm	35 ppm	-	-
Na	-	6 ppm	-	-
Ca+Mg as Ca	0.20% max.	0.02%	-	-
A - AE as SO_4	0.08% max.	0.05%	-	-
Acidity as H_2SO_4	0.01% max.	0.01%		
H_2O Insol.	0.01% max.	< 0.01%	<100 ppm	<60 ppm

"IMPORTANT NOTICE REGARDING THIS INFORMATION:
The statements, technical information, and recommendations contained in this document are based on tests and data that are believed to be reliable. Further, as the actual use of our products by others is beyond our control, no guarantee of any kind is made as to the effects of such use, or the results to be obtained, whether the use is made in accordance with the recommendations or suggestions contained herein or otherwise. This document is not contractual and NOTHING HEREIN CONSTITUTES A REPRESENTATION OR WARRANTY THAT THE GOODS DESCRIBED ARE FIT FOR A PARTICULAR PURPOSE OF A CUSTOMER or that their use does not conflict with any existing patent rights. The exclusive source of any warranty and of any other customer rights whatsoever is on the Atotech invoice. Also, since this data sheet may be provided by electronic media, Atotech cannot guarantee the accuracy or originality hereof. Any alterations made to this document other than by Atotech corporate headquarters is expressly prohibited."

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ATO

Atotech USA Inc., 1750 Overview Drive, P.O. Box 12000, Rock Hill, SC 29731-2000
Telephone: 803-817-3500 • Fax: 803-817-3656

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Properties & Product Characteristics

Small green crystals, free flowing

Density:	100% through 4 Mesh 95% on 35 Mesh

PACKAGING:

Multiwall Bag - 50 lbs. Net
40 lb. bags per pallet

WASTE DISPOSAL

This material must be disposed of in accordance with all applicable federal, state, and local regulations. Consult the MSDS for additional regulatory information.

GENERAL SAFETY PRECAUTIONS

Avoid direct contact with this material. Do not inhale associated mist, vapors, and/or dust. As applicable, keep exposure below the limits recommended by OSHA, ACGIH, the manufacturer, and others. Wash contaminated clothing before reuse. Always comply with the Hazard Communication Standard, 29CFR1910.1200; emergency showers and eyewashes must be available.

It is recommended that the plating chemistry product(s) referred to in this Technical Information sheet be used: (1) in accordance with the information provided in product specific MSDS; and (2) in compliance with all appropriate requirements and guidelines established by OSHA, NIOSH, ACGIH, NFPA, and others.

FIRST-AID RECOMMENDATIONS

Types of Exposure	Recommendations
Eye	Flush with water for at least 15 minutes.
Skin	Wash thoroughly with soap and water.
Inhalation	Remove to fresh air immediately.
Ingestion	Give water or milk (if conscious and not having convulsions). Remove to fresh air immediately.

REVIEW MSDS BEFORE USING THIS PLATING CHEMISTRY AND FOR PRODUCT SPECIFIC INFORMATION. A precautionary approach should be used when there is potential for chemical exposure -- this includes minimizing exposure potential, rapid decontamination, and medical follow-up.

NOTE: A Material Safety Data Sheet (MSDS) for this product is available on request from Atotech USA Inc., Customer Service/Sales Support Group, 1750 Overview Drive, P.O. Box 12000, Rock Hill, SC 29731-2000.

TELEPHONE NUMBERS

Customer Service/Sales Support Group: (803) 817-3538 (MSDS requests)
Product Safety Department: (803) 817-3549 (regulatory inquiries and emergencies)

TO PLACE AN ORDER: 1-800-PLATING

Sheet No: NI-CL-C
Revision: 6/22/94

Technical Information

NICKEL CHLORIDE CRYSTAL

Atotech Nickel Chloride Crystal is of French origin, produced by Eramet - SLN. It is a high quality and consistent product suitable for use in nickel plating applications, manufacture of Synthetic ink, colorant for glass and oil well treating.

Specifications:

	Nickel Chloride Hexahydrate Guaranteed
Ni	> 24.0%
Co	< 0.02%
Fe	< 0.01%
Cu	< 0.002%
Pb	< 0.002%
Zn	< 0.002%
Cd	< 0.005%
Insols	< 0.05%

Properties & Product Characteristics

Light green coarse powder	Green Hydrated Crystal
Density Approx. 6.5 lbs/gal	Density Approx. 10 lbs/gal

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Atotech USA Inc., 1750 Overview Drive, P.O. Box 12000, Rock Hill, SC 29731-2000
Telephone: 803-817-3500 • Fax: 803-817-3686

Packaging: Multiwall bag with polyethylene liner 50 lbs. net.

WASTE DISPOSAL

This material must be disposed of in accordance with all applicable federal, state, and local regulations. Consult the MSDS for additional regulatory information.

GENERAL SAFETY PRECAUTIONS

Avoid direct contact with this material. Do not inhale associated mist, vapors, and/or dust. As applicable, keep exposure below the limits recommended by OSHA, ACGIH, the manufacturer, and others. Wash contaminated clothing before reuse. Always comply with the Hazard Communication Standard, 29CFR1910.1200; emergency showers and eyewashes must be available.

It is recommended that the plating chemistry product(s) referred to in this Technical Information sheet be used: (1) in accordance with the provided in product specific MSDS; and (2) in compliance with all appropriate requirements and guidelines established by OSHA, NIOSH, ACGIH, NFPA, and others.

FIRST-AID RECOMMENDATIONS

Types of Exposure	Recommendations
Eye	Flush with water for at least 15 minutes.
Skin	Wash thoroughly with soap and water.
Inhalation	Remove to fresh air immediately.
Ingestion	Give water or milk (if conscious and not having convulsions).

REVIEW MSDS BEFORE USING THIS PLATING CHEMISTRY AND FOR PRODUCT SPECIFIC INFORMATION. A precautionary approach should be used when there is potential for chemical exposure – this includes minimizing exposure potential, rapid decontamination, and medical follow-up.

NOTE: A Material Safety Data Sheet (MSDS) for this product is available on request from Atotech USA Inc., Customer Service/Sales Support Group, 1750 Overview Drive, P.O. Box 12000, Rock Hill, SC 29731-2000.

TELEPHONE NUMBERS

Customer Service/Sales Support Group: (803) 817-3538 (MSDS requests)
Product Safety Department: (803) 817-3549 (regulatory inquiries and emergencies)

TO PLACE AN ORDER: 1-800-PLATING

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ATTACHMENT C

Equivalence of Nickel Metal Concentrations in Liquid Nickel Salts

Concentrations of liquid $NiSO_4 \cdot 6H_2O$ as obtained from spec. sheets (Attachment B) and shown below as Ⓐ, Ⓑ, and Ⓒ.

Ⓐ $= 17.8 \frac{oz}{gal}$ = Ni metal conc. (Dremco, Gumm)

Ⓑ $= 133 \frac{g}{l}$ = Ni metal conc. (Dremco, Gumm, Southeastern).

Ⓒ $= 5.0 \frac{lbs}{gal}$ = $NiSO_4 \cdot 6H_2O$ conc. (Van Waters)

Calculations to show equivalence of Ⓐ, Ⓑ, and Ⓒ:

$$Ⓐ = \frac{17.8\ oz}{gal} \Big| \frac{1\ lb}{16\ oz} \Big| \frac{454\ g}{1\ lb} \Big| \frac{1\ gal}{3.79\ l} = \frac{133\ g}{l} = Ⓑ$$

Ⓒ

element	atomic wt. $\left(\frac{lb}{lbmole}\right)$
Ni	58
S	32
O_4	4X16=64
$6H_2$	6X2X1=12
$6O$	+ 6X16=96

compound wt. $NiSO_4 \cdot 6H_2O$ = 262 lb/lb mole

Ni fraction of $NiSO_4 \cdot 6H_2O = \frac{58}{262}$

x = concentration of $NiSO_4 \cdot 6H_2O$ in purchased solution

$$\frac{58}{262} = \frac{17.8 \frac{oz}{gal}}{x} \longrightarrow x = 80.4 \frac{oz}{gal} NiSO_4 \cdot 6H_2O \Big| \frac{1\ lb}{16\ oz}$$

$$x = 5.03 \frac{lbs}{gal} = Ⓒ$$

∴ Ⓐ, Ⓑ, and Ⓒ have equivalent concs. of Ni met

Concentrations of liquid $NiCl_2 \cdot 6H_2O$ as obtained from spec sheets (Attachment B) and shown below as Ⓐ, Ⓑ, and Ⓒ.

$\textcircled{A} = 23.5 \frac{oz}{gal}$ = Ni metal conc. (Dremco, Gumm)

$\textcircled{B} = 178 \frac{g}{l}$ = Ni metal conc. (Dremco, Gumm, Southeaster

$\textcircled{C} = 6.0 \frac{lbs}{gal} = NiCl_2 \cdot 6H_2O$ conc. (Dremco, Gumm, Van Wate

Calculations to show equivalence of Ⓐ, Ⓑ, and Ⓒ:

$$\textcircled{A} = \frac{23.5\,oz}{gal} \left| \frac{1\,lb}{16\,oz} \right| \frac{454\,g}{1\,lb} \left| \frac{1\,gal}{3.79\,l} \right. = 176 \frac{g}{l} \approx \textcircled{B}$$

Ⓒ element	atomic wt. $\left(\frac{lb}{lb\,mole}\right)$
Ni	58
Cl_2	$2 \times 35.5 = 71$
$6H_2$	$6 \times 2 \times 1 = 12$
$6O$	$6 \times 16 = 96$

compound wt. $NiCl_2 \cdot 6H_2O = 237\ lb/lb\ mole$

Ni fraction of $NiCl_2 \cdot 6H_2O = \frac{58}{237}$

x = concentration of $NiCl_2 \cdot 6H_2O$ in purchased solution

$$\frac{58}{237} = \frac{23.5 \frac{oz}{gal}}{x} \longrightarrow x = 96 \frac{oz}{gal} NiCl_2 \cdot 6H_2O \left| \frac{1\,lb}{16\,oz} \right.$$

$$x = 6.0 \frac{lbs}{gal} = \textcircled{C}$$

∴ Ⓐ, Ⓑ, and Ⓒ have equivalent concentrations of Ni metal. page 46

Crystalline $NiSO_4 \cdot 6H_2O$ product supplied by all vendors contacted is 22% nickel by weight (see Attachment B).

Crystalline $NiCl_2 \cdot 6H_2O$ product supplied by all vendors contacted is 24% nickel by weight (see Attachment B).

For pure $NiSO_4 \cdot 6H_2O$ compound (see 1st page of this attachment)

$$\text{Ni fraction of compound} = \frac{58}{262} = 0.22 = 22\%$$

For pure $NiCl_2 \cdot 6H_2O$ compound (see 2nd page of this attachment)

$$\text{Ni fraction of compound} = \frac{58}{237} = 0.24 = 24\%$$

Attachment D

Watts Solution
Suppliers of Chemicals

Dremco, Inc
1550 E Missouri Ave, Ste 302
Phoenix, Arizona 85014
Tel. 602-234-8908, Fax 602-234-8909
Contact: Doug Robinson

Frederick Gumm Chemical Company, Inc.
Gastonia, North Carolina
1-800-541-7878
Contact: Cynthia Costner

Southeastern Chemical, Inc.
1512-A Sholar Avenue
Chattanooga, Tennessee 37406
Tel. 423-622-5154, Fax 423-698-5152
Contacts: Sandi Guess and Ben Johnson (800-376-7113)
    Titan International (manufacturer for Southeastern)
    Tel. 800-435-4644
    Contact: Ellen Jansen

Van Waters & Rogers, Inc.
Three Riverside Lane
Chattanooga, Tennessee 37406
Tel. 800-233-0633, Fax 423-624-4190
Contact: Priscilla Pennington

Vendors which were contacted, but did not provide information as of 3/05/98.

King Supply Company
Illinois
847-698-4564
Contact: Dave Tony

P B & S Chemical Co. Inc.
Knoxville
423-523-7171
Contact: Sheila

Southern Industrial Chemicals, Inc.
1450 Marietta Boulevard, Northwest
Atlanta, Georgia 30318
Tel. 800-394-9770
Contact: Robert Allen

Instructional Guide
Preparation of Watts Solution for Electrorefining

Equipment and Chemicals Required

300-gallon chemical mixing tank made of stainless steel with a kynar lining.
Marker for the tank that indicates these levels: 97 gallons and 250 gallons
Ventilation for tank
Immersion-type heater with the capacity to heat and maintain the temperature of the 300-gallon tank at 55-60°C (131-140°F), constructed of materials that will not react with the Watts solution.
Temperature indicator, immersion type
pH indicator for continuous monitoring of pH
Deionized (DI) water
Concentrated liquid hydrated nickel sulfate [($NiSO_4 \cdot 6H_2O$) see Table 1]
Concentrated liquid hydrated nickel chloride [($NiCl_2 \cdot 6H_2O$) see Table 1]
Nickel sulfate and nickel chloride are EPA-listed hazardous wastes due to their nickel content. See MSDSs for environmental information: spill response and recommended disposal. Do not store nickel sulfate and nickel chloride near acids. They can react with acids to generate hydrogen gas.
Crystalline solid boric acid (H_3BO_3) to buffer the solution (see Table 1)
There are no special storage requirements for boric acid. See MSDS for environmental information: spill response and recommended disposal.
Hydrometer to monitor solution density
Chemical composition analyzer (i.e., ion-selective electrodes)
Personal protective clothing and equipment (see item 1. following and MSDSs for details)

Table 1. Characteristics, quantities, and concentrations of the Watts solution components

Component	Appearance and odor	Quantity in a 250-gallon batch	Concentration in Watts solution
Nickel sulfate ($NiSO_4 \cdot 6H_2O$)	green liquid with characteristic odor	124 gallons	40 oz (av)/gal
Nickel chloride ($NiCl_2 \cdot 6H_2O$)	clear green liquid with characteristic odor	21 gallons	8 oz (av)/gal
Boric acid (H_3BO_3)	white powder with no odor	94 pounds	6 oz (av)/gal
Deionized water (H_2O)		97 gallons	

Method of Preparation

1. While handling and mixing the liquid nickel salts and boric acid, take safety precautions. Wear appropriate protective clothing and equipment to prevent skin and eye contact and inhalation. Do not wear contact lenses. Local exhaust is recommended. Do not breath dust, mist, or fumes generated by handling or using these products. Use with adequate ventilation. Use NIOSH-approved dust respirator if ventilation is inadequate. Keep containers closed when not in use. Avoid prolonged or repeated skin contact. Wash skin thoroughly after handling nickel compounds. A dermatitis known as "nickel itch" may result from skin sensitization. See MSDSs for pertinent information concerning the properties, safe handling, health effects and specific first aid procedures for nickel sulfate, nickel chloride, and boric acid.
2. Add 97 gallons of DI water to the chemical mixing tank.
3. Turn on the heater and heat the water to 60°C (140°F). Adjust the settings of the heater throughout the mixing process to maintain the temperature in the range of 55-60°C (131-140°F). The temperature must be maintained at approximately 60°C–not lower than 55°C. This temperature is crucial. The boric acid will come out of solution at lower temperatures.
4. Turn on the agitator.
5. While agitating the solution and monitoring the temperature, slowly add 94 pounds of boric acid. If the temperature increases above 60°C (140°F), stop adding the boric acid. Resume the addition when the temperature decreases to 60°C.
6. While agitating the solution and monitoring the temperature, slowly add 124 gallons of nickel sulfate.
7. While agitating the solution and monitoring the temperature, slowly add 21 gallons of nickel chloride.
8. Turn off the agitator.
9. If needed, add additional DI water into the tank to make the total volume 250 gallons.
10. Turn on the agitator.
11. Check the temperature and adjust the heater to attain and maintain a temperature in the range of 55-60°C (131-140°F).
12. Mix the solution for 20 minutes while monitoring and maintaining the temperature in the specified range.
13. Turn off the agitator and immediately take a sample of the solution. Restart the agitator. Visually inspect the solution sample. The color of the solution should be green. All solids in the sample should be dissolved. If the solids are not dissolved, repeat steps 12 and 13 until the solids are dissolved. If the solids are dissolved, proceed to step 14.
14. When the solids are dissolved, check the specific gravity of the solution with the hydrometer. The specific gravity should be between 1.2 and 1.35. Record the specific gravity. If the specific gravity of the sample is within this range, proceed to step 15. If the specific gravity is not within the stated range, the chemistry of the solution must be adjusted with the addition of chemicals.
15. Check the pH of the solution as shown on the pH indicator. The pH should be approximately 3. If the pH is outside the range 3 to 3.5, the pH will be adjusted after the solution is pumped to the electrorefining cell.
16. Send the sample of solution to be analyzed for chemical composition. When this analysis is completed, ensure that the results are recorded.

17. Turn off the agitator and the heater.
18. Transfer the solution to the holding tank of the electrorefining cell where the pH will be adjusted if necessary with sulfuric acid and sodium hydroxide.
19. For each batch of solution that is mixed, record the pH, specific gravity, and the chemical composition.

100 gallon Mix Plan
Watts Nickel

Using 38 gallons of Hot D/I Water mix 33 lbs. of Boric acid until the Boric dissolves.
Mix into the boric solution 46 gallons of Nickel Sulfate.
Mix into the same container 10 gallons of Nickel Chloride.
This should be approximately 100 gallons use additional D/I water to obtain 100 gallons if necessary.

50 gallon Mix Plan
Watts nickel

Using 19 gallons of Hot D/I Water mix 17 lbs. of Boric acid until the Boric dissolves.
Mix into the boric solution 25 gallons of Nickel Sulfate.
Mix into the same container 5 gallons of Nickel Chloride.
This should be 50 gallons of Watts Nickel Electrolyte or just a little over in a 55-gallon drum.

300 gallon Mix Plan
Watts Nickel

Transfer 100 gallons of Hot D/I water into the Main Cell.
Add 94 lbs. of Boric acid slowly until it dissolves.
Add 120 gallons of Nickel Sulfate.
Add 20 gallons of Nickel Chloride.
Using Air agitation mix thoroughly while introducing the acids.
Repeat (4) times or until proper level is maintained. Top off the cell with additional acid from MRU and Sump or with D/I water.

Comments and Notes
Preparation of Watts Solution from Crystalline SOLIDS

1. The specifications given in the Instruction and Instructional Guide are based on 250 gallons of Watts solution mixed in a 300-gallon tank. Six batches will have to be mixed to prepare enough Watts solution for the 1500-gallon electrorefining cell. The quantities of chemical components specified are based on a Watts solution with these concentrations: 40 oz (av)/gal nickel sulfate, 8 oz (av)/ gal nickel chloride, and 6 oz (av)/gal boric acid.

2. It is assumed that the temperature of the mixing tank is maintained between 55 and 60°C (131 to 140°F). If this temperature is not maintained in the mixing tank or in the electrorefining cell, the boric acid will not remain dissolved.

3. Note carefully that the instructions state 94 pounds as the quantity of boric acid to be mixed with 100 gallons of water. The solubility of boric acid at 50°C (131°F) is about 1.07 pounds per gallon of water[a]—meaning that 100 gallons of water will only hold 107 pounds of boric acid at 50°C (131°F) under optimal conditions. At 60°C (140°F), 100 gallons of water will hold 123 pounds of boric acid[b]. Therefore, it is imperative that no more than 100 pounds of boric acid be added to each 250-gallon batch with a temperature exceeding 50°C (131°F) while agitating.

4. Note: the holding tank of the electrorefining cell (or any other place the solution is kept) should be heated to approximately 60°C (140°F). If this tank is not heated, the solids will come out of solution.

5. Adjustment of the solution chemistry while it is in the mixing tank, referred to in steps 14 and 15 of the instructional guide, should be addressed in more detail after some experience is gained with the solution chemistry.

6. After all the equipment is installed for the mixing process, the instructional guide should be checked for consistency and modified as required.

---

[a] Interpolation from graph based upon data presented in CRC *Handbook of Chemistry and Physics*, 54th edition and MSDS for boric acid (attached).

[b] *Lange's Handbook of Chemistry*, 14th edition.

April 10, 1998

Solubility calculations

From attached graph, solubility of boric acid at 50°C = $\frac{12.8\,\text{g}\ H_3BO_3}{100\,\text{mL}\ H_2O} = \frac{1.067\,\text{lb}\ H_3BO_3}{\text{gal}\ H_2O}$.

Using the data given above, the maximum mass of boric acid that can be dissolved in 100 gallons of water at 50°C is given by $\frac{1.067\,\text{lb}\ H_3BO_3}{\text{gal}\ H_2O} \times (100\text{gal}\ H_2O) = 107\,\text{lb}\ H_3BO_3$.

Therefore, the 94 lb of boric acid needed can be dissolved in the specified 100 gal of water at 50°C.

From 14th edition of *Lange's Handbook of Chemistry*, the solubility of boric acid at 60°C = $\frac{14.8\,\text{g}\ H_3BO_3^{*}}{100\,\text{mL}\ H_2O} = \frac{1.234\,\text{lb}\ H_3BO_3}{\text{gal}\ H_2O}$.

Using the boric acid solubility data for 60°C, the maximum mass of boric acid that can be dissolved in 97 gallons of water at 60°C is $\frac{1.234\,\text{lb}\ H_3BO_3}{\text{gal}\ H_2O} \times (100\,\text{gal}\ H_2O) = 123\,\text{lb}\ H_3BO_3$.

Clearly, the required 94 lb of boric acid will remain in solution at 60°C.

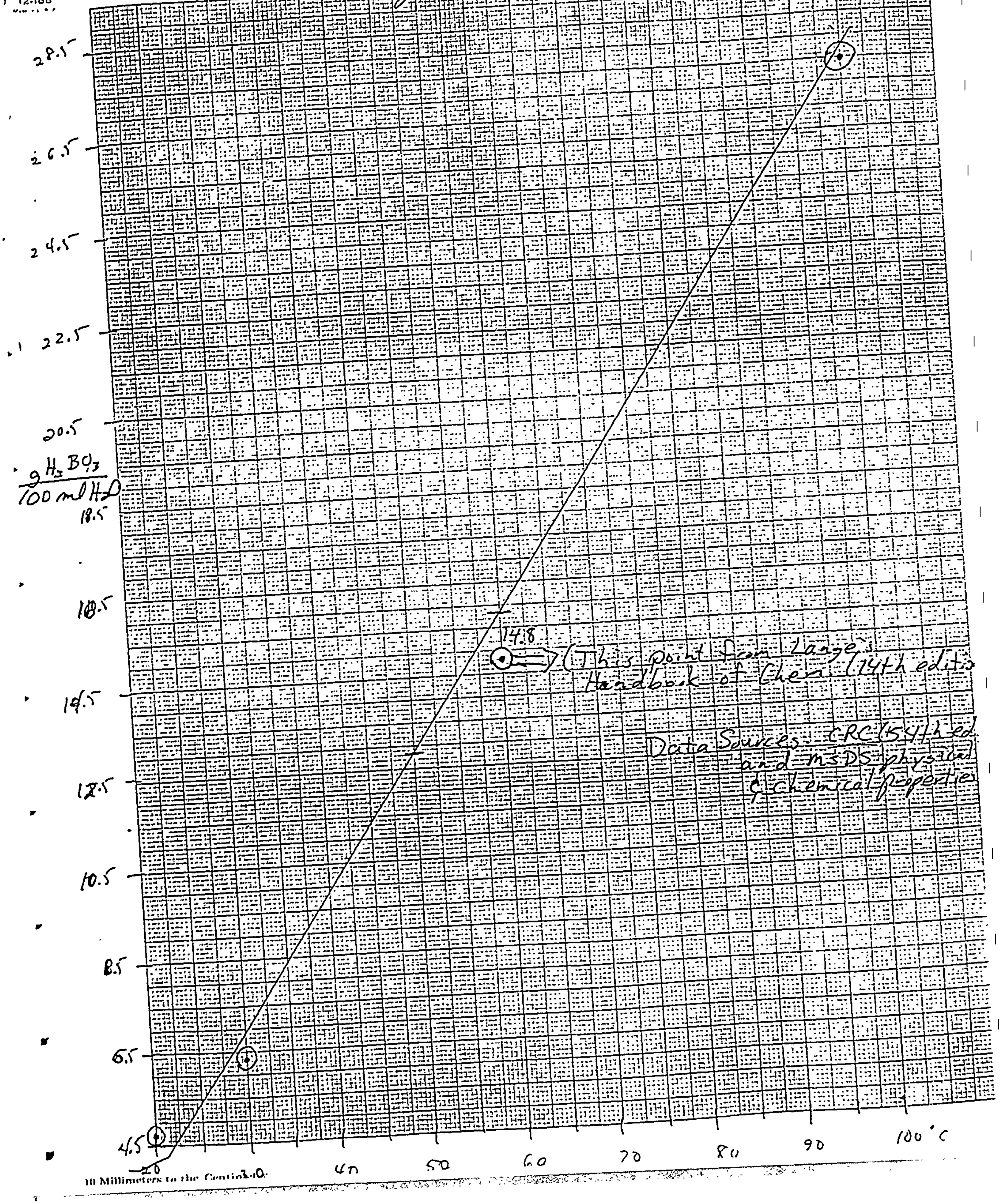
Conversion factor to go from $\frac{\text{g}}{100\text{mL}}$ to $\frac{\text{lb}}{\text{gal}}$ is

$$\frac{1\ \text{g}}{100\,\text{mL}}\left[\frac{1\ \text{lb}}{454\ \text{g}}\right]\left[\frac{1000\ \text{mL}}{1\ \text{L}}\right]\left[\frac{3.785\ \text{L}}{1\ \text{gal}}\right] = \frac{0.0834\,\text{lb}}{1\text{gal}}$$

---

*The solubility value from the graph was 15.8 g/100 mL. The lower, more conservative value of 14.8 from *Lange's Handbook* was used in an attempt to assure that the mixing would be accomplished successfully.

Solubility H_3BO_3 in H_2O



(This point from Lange's Handbook of Chem. (14th editio

Data Sources: CRC (54th ed. and MSDS physical & chemical properties

ELECTROWINNING

OF

NICKEL

X-Ray diffraction of the cathode deposit (Experiment dated: Jan. 9, 1998)

Experimental conditions
Anode material - Platinum coated tungsten wire
Cathode material - Stainless Steel
Anode to cathode distance - 2.5 inches
Current Density applied - 1.31 $A/dm^2 \cong 20\ A/ft^2$

Appearance of the cathode deposit

- *Nickel deposited is brownish metallic gray in color*
- *The nickel deposit is cracked at places and lifted up from the cathode surface*

Objective : To identify the species that co-deposited with Nickel.

Results The summary of the results of XRD of the deposit is given below:

No. of Peak	2 theta	d-space	Intensity	Species
1	44.599	2.0301	100	Ni, (Fe)
2	51.917	1.7598	29	Ni
3	76.481	1.2445	17	Ni
4	92.982	1.0621	17	Ni
5	98.510	1.0167	8	Fe
6	116.103	0.9078	1	Fe

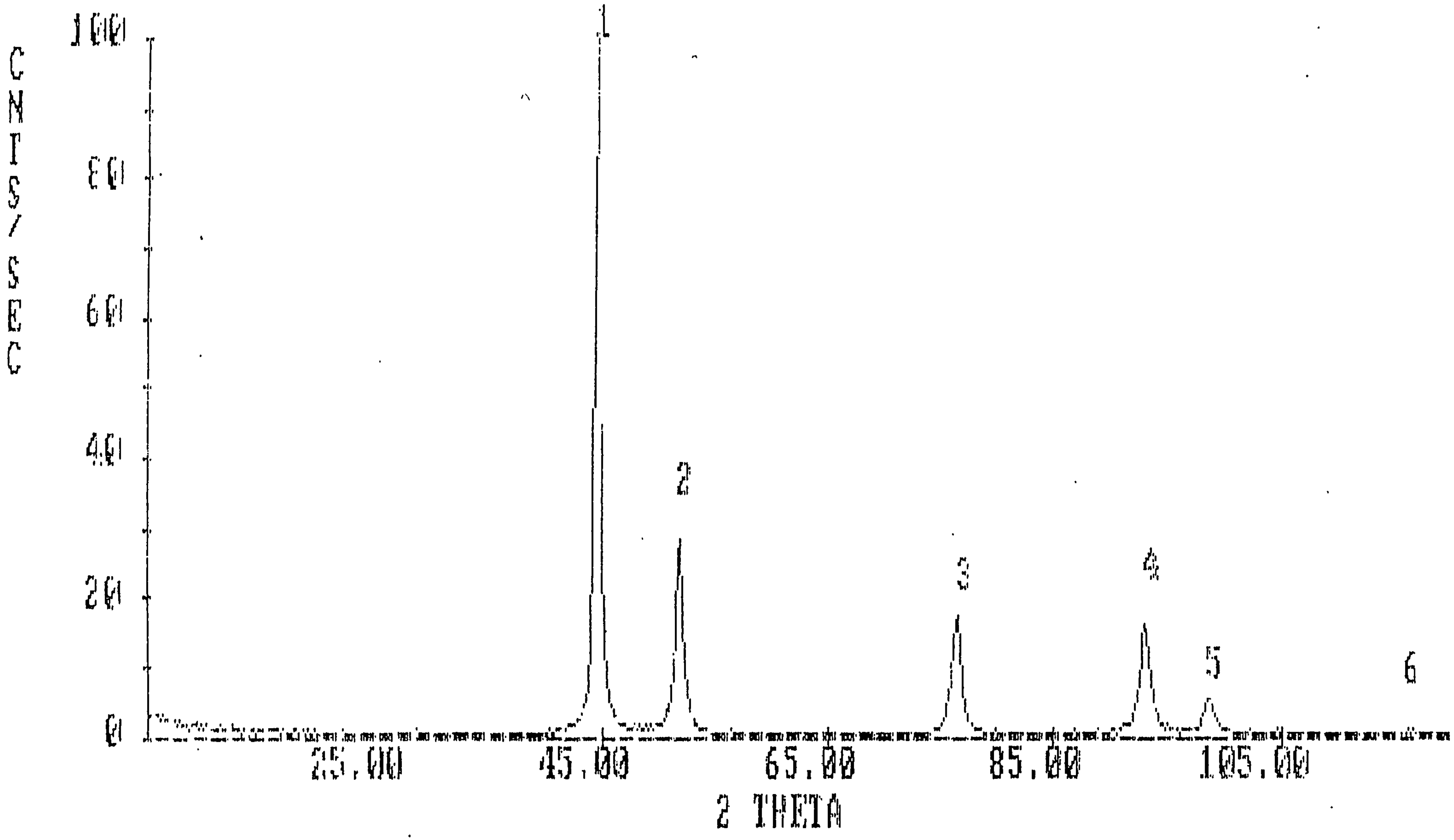
Standard tables for Nickel and Iron for comparison are given below:

Nickel, syn (Ni)4F 2.03_x 1.76_4 1.25_2 1.06_2 0.79_2 0.81_1 1.02_1 0.88_1

Fe, syn α-(Fe)2B 2.03_x 1.17_3 0.91_1 1.01_1 0.83_1

It seems that may be some iron co-deposited with nickel and imparted a brownish tinge to the deposited product. However, some other method of analyses is also required to confirm the findings. Copper, chromium and tungsten were absent.

Ni-Ti- 5/11/57 S: .20 T= 1.0   Ni-Ti-W



```
   0  6-11-57         Ni-Ti-W
START ANGLE=    5.000  DELTA 2THETA=   .200  TIME=  1.0 TUBE=CU LAMDA= 1.541
SM= 0  BKGR= 0 0 0  KA2=0  TUBE=CU SCH=15 SENS=3.00  CENTROID=15 NTP= 5

  N   2THETA    DSPACE  (1/D**2)  HEIGHT      AREA         P        W2        W4

  1   44.599   2.0301    .2427    3921.     3022.     2361.     2.114      .771
  2   51.917   1.7598    .3229    1128.     1116.      702.     1.879      .989
  3   76.481   1.2445    .6457     691.      798.      485.     1.879     1.154
  4   92.982   1.0621    .8865     632.      885.      512.     2.114     1.400
  5   98.510   1.0167    .9674     207.      280.      131.     1.879     1.355
  6  116.103    .9078   1.2134      55.       55.       20.     2.114      .999
```

NICKEL ELECTRO WINNING

One Anode and one Cathode System

Anode material - Platinum coated tungsten wire
Cathode material - Stainless Steel
Anode to cathode distance - 2.5 inches
Current Density applied - 1.31 A/dm^2 ≡ 20 A/ft^2

Voltage changes: At start - 7 V
At end - 7 V

Total Time allowed for Electro winning - 4 hours

Nickel- ion concentration as the function of time
Time interval for metal ion concentration determination - 1 hour

At the start of experiment -9.1 oz/gallon
a) After 1hour - not measured.
b) After 2 hours - 8.9 oz/gallon
c) After 3 hours - 9.1 oz/gallon
d) After 4 hours - 9.1 oz/gallon

Temperature Recording The thermometer was dipped in corner at a depth of ~2.5 inches.
Time interval for monitoring the temperature changes - 1 hour

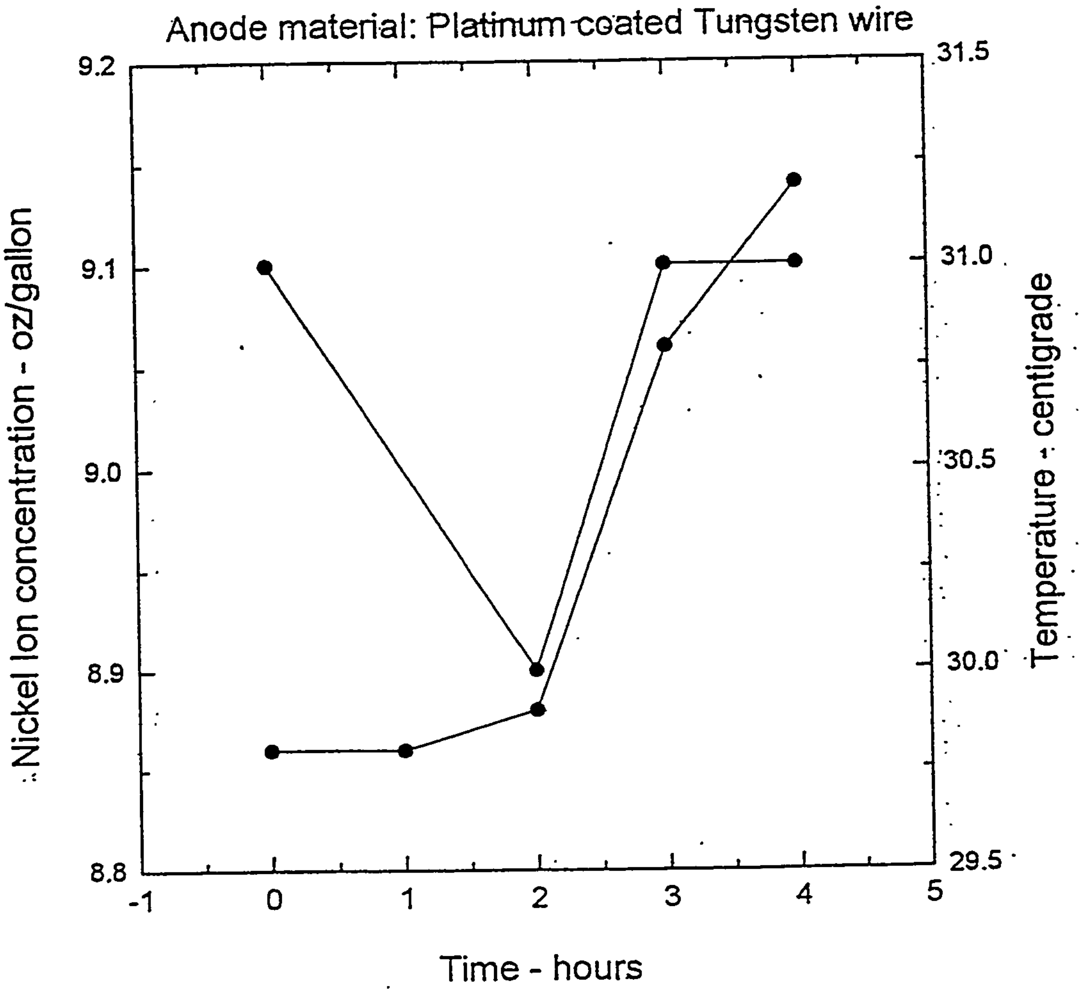
At the start of experiment -29.8 °C
a) After 1hour - 29.8 °C
b) After 2 hours - 29.9 °C
c) After 3 hours - 30.8 °C
d) After 4 hours - 31.2 °C
Experiment switched off.

Current Efficiency 65.1 %

Observations

- Vigorous gas bubbles evolved from the cathode and the anode surfaces
- The pungent smell of the anode gas identified it to be Cl_2 gas
- *Nickel deposited is brownish-metallic gray in color*
- *The nickel deposit is cracked at places and lifted up from the cathode surface*

Remarks This anode material is not suitable for the electro winning of nickel. The anode should be carefully selected and replaced by an inert material which will reduce or avoid Cl_2 gas evolution and the contamination of the solution through the anode dissolution, and consequently will require a lower cell driving voltage.



Change in the nickel ion concentration
and temperature with time.
Current density - 20 Amps/ft^2
Anode-cathode distance - 2.5 inches

NICKEL ELECTRO WINNING

One Anode and one Cathode System

Anode material - Titanium-vanadium Alloy
Cathode material - Stainless Steel
Anode to cathode distance - 2.5 inches
Current Density applied - 1.31 A/dm^2 $\equiv$ 20 A/ft^2

Results: The voltage could be increased to 25 V but the current could not increase more than 1A and it decreased continuously.
The experiment was switched off.

The same experiment was repeated with a SS Anode of the same composition as that of the cathode

Voltage changes: At start - 5 V
At end - 3 V

Total Time allowed for Electro winning - 4 hours

Nickel ion concentration as the function of time
Time interval for metal ion concentration determination - 1 hour

At the start of experiment -9.5 oz/gallon
a) After 1hour - 9.5 oz/gallon
b) After 2 hours - 9.4 oz/gallon
c) After 3 hours - 9.3 oz/gallon
d) After 4 hours - 9.3 oz/gallon

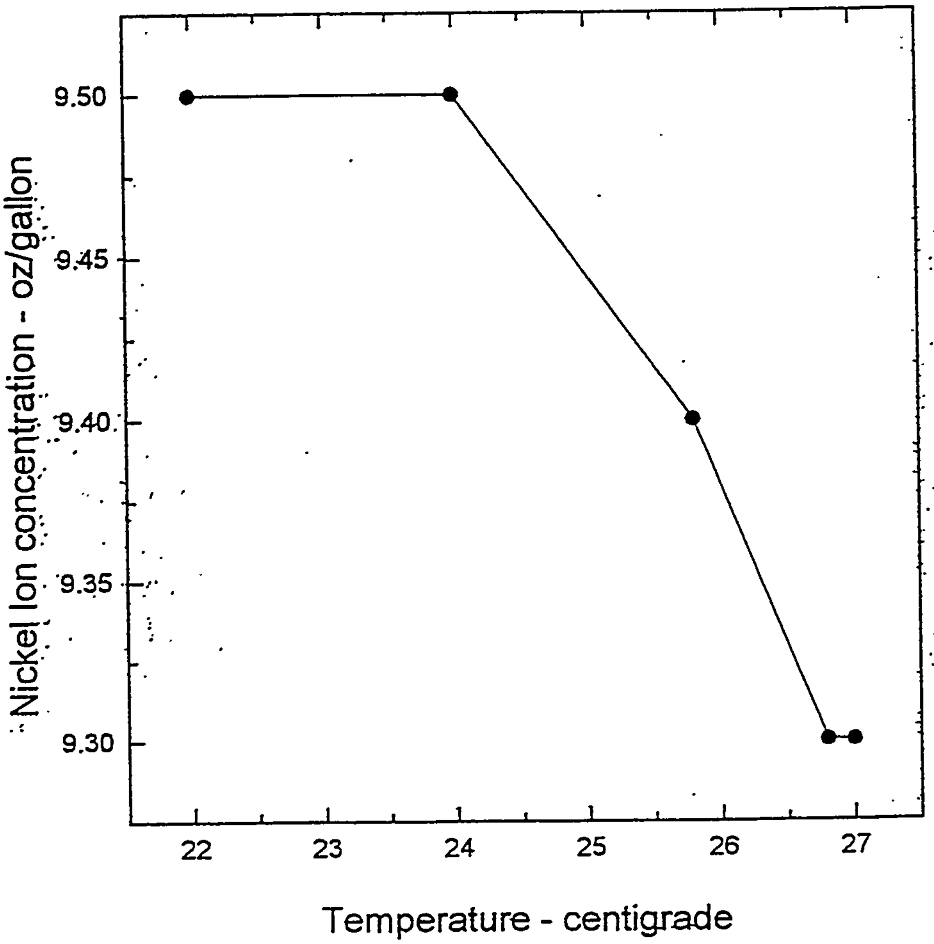
Temperature Recording The thermometer was dipped in corner at a depth of ~2.5 inches.
Time interval for monitoring the temperature changes - 1 hour

At the start of experiment -22 °C
a) After 1hour - 24 °C
b) After 2 hours - 25.8 °C
c) After 3 hours - 26.8 °C
d) After 4 hours - 27°C
Experiment switched off.

Current Efficiency 70.5 %

Observations
- Gas bubbles evolved from the cathode and anode surfaces
- Metallic nickel particles were observed floating in the solution
- Brown rust particles were observed at the anode/solution interface
- The anode pitted as elongated oval, cavities
- *Nickel deposited in the form of thin, ductile, dark flakes*

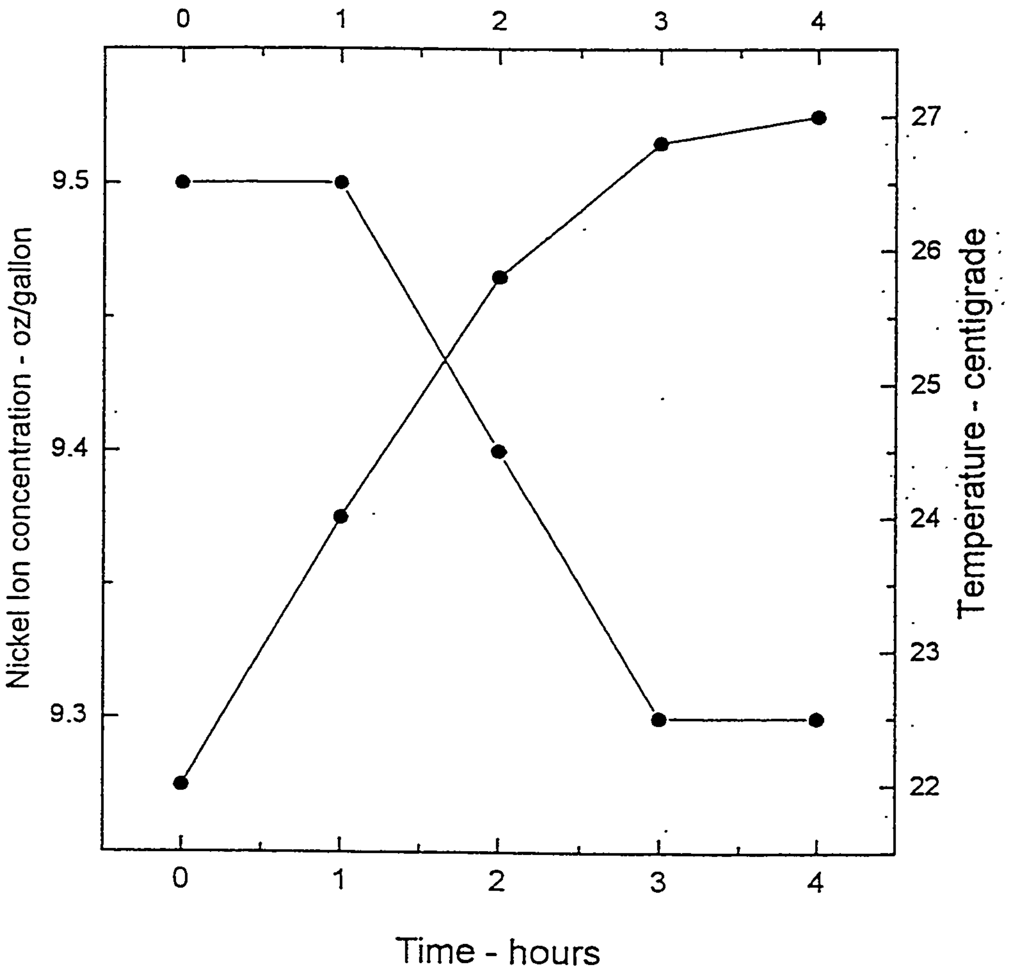
Remarks The anode material should be carefully selected to avoid gas evolution and the contamination of the solution through the anode dissolution.



Change in the nickel ion concentration with temperature.

Current density - 20 Amps/ft^2

Anode-cathode distance - 2.5 inches

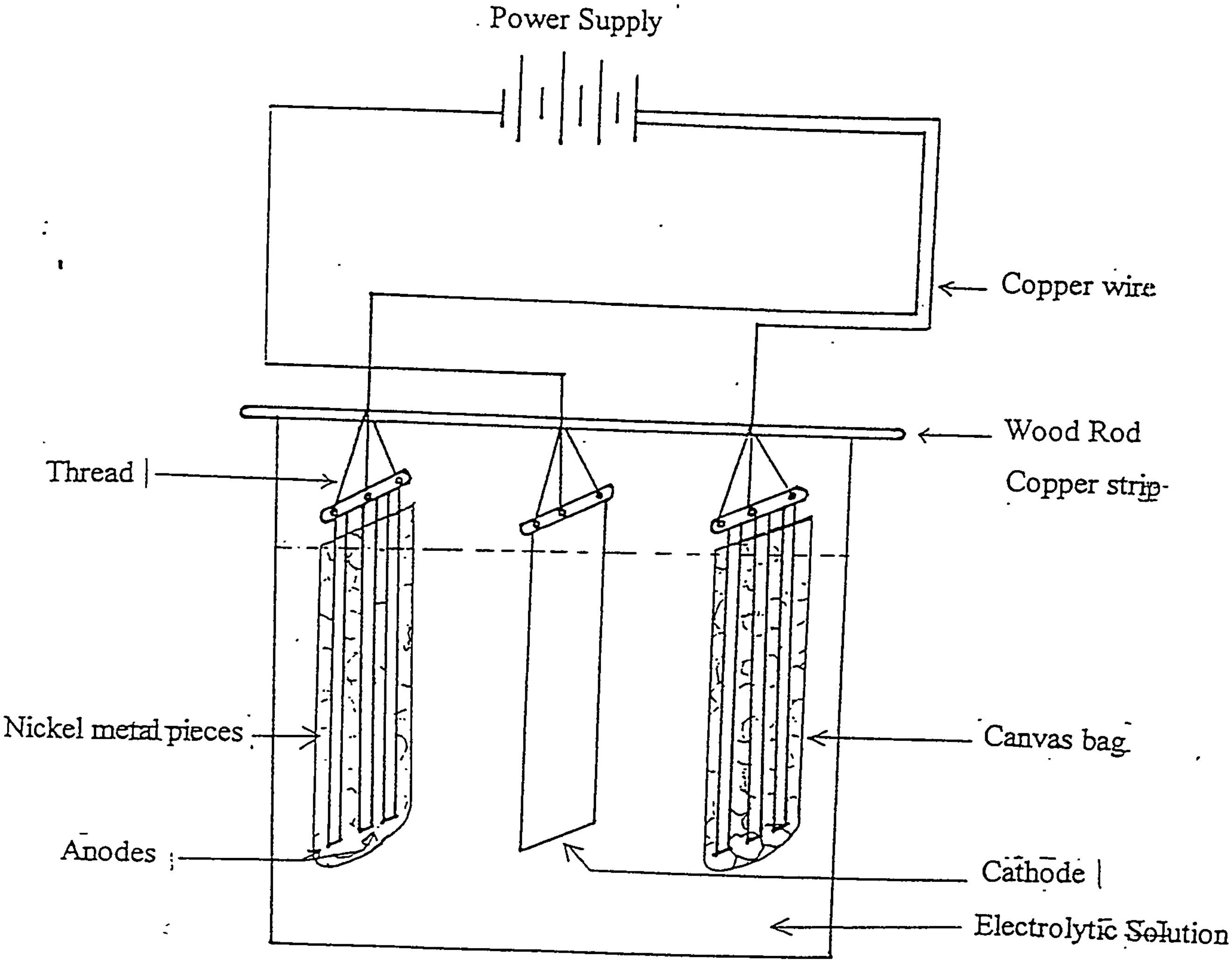


Change in the nickel ion concentration
and temperature with time.
Current density - 20 Amps/ft^2
Anode-cathode distance - 2.5 inches

ELECTROPLATING

OF

NICKEL



ELECTROLYTIC CELL FOR NICKEL ELECTROPLATING
Two Anodes and one Cathode System

NICKEL ELECTROPLATING

Two Anodes and one Cathode System

Current Density applied - 5.0 A/dm^2 46.4 A/ft^2
Voltage applied - 5V

Anode to cathode distance - 2.5 inches
Total Time allowed for Electroplating - 4 hours

Nickel ion concentration as the function of time
Time interval for metal ion concentration determination - 1 hour

a) At the start of experiment - 9.99 oz/gallon
b) After 1hour - 9.99 oz/gallon
c) After 2 hours - 9.97 oz/gallon
d) After 3 hours - 9.99 oz/gallon
e) After 4 hours - 9.90 oz/gallon

Experiment switched off.

Current Efficiency 95%

Observations Flakes of ductile whitish bright Nickel were deposited.

Remarks. A distance of 2.5 inches between the anodes and the central cathode should be increased for these experimental conditions for it may cause short circuiting.

NICKEL ELECTROPLATING

Two Anodes and one Cathode System

Current Density applied - 2.15 $A/dm^2 \equiv 20\ A/ft^2$
Voltage applied - 1.5 - 2.0V

Anode to cathode distance - 2.5 inches
Total Time allowed for Electroplating - 4 hours

Nickel ion concentration as the function of time
Time interval for metal ion concentration determination - 1 hour

At the start of experiment - 10.19 oz/gallon
a) After 1hour - 9.86 oz/gallon
b) After 2 hours - 10.08 oz/gallon
c) After 3 hours - 9.95 oz/gallon
d) After 4 hours - 10.06 oz/gallon

Experiment switched off.

Current Efficiency 88 %

Observations Flakes of ductile whitish bright Nickel as thin sheets were deposited.

Remarks. The Nickel deposited is in the form of thin sheets and hence the stirrer speed should be adjusted to avoid the breaking of the nickel sheet from the cathode surface.

NICKEL ELECTROPLATING

Two Anodes and one Cathode System

Current Density applied - 1.62 A/dm^2 ≡ 15A/ft^2
Voltage applied - 2.5$_{start}$ - 2.0V

Anode to cathode distance - 2.5 inches
Total Time allowed for Electroplating - 4 hours

Nickel ion concentration as the function of time
Time interval for metal ion concentration determination - 1 hour

At the start of experiment - 9.72 oz/gallon
a) After 1hour - 9.72 oz/gallon
b) After 2 hours - 9.80 oz/gallon
c) After 3 hours - 9.95 oz/gallon
d) After 4 hours - 9.83 oz/gallon

Temperature Recording The thermometer was dipped in corner at a depth of ~2.5 inches.
Time interval for monitoring the temperature changes - 1 hour

At the start of experiment -30 °C
a) After 1hour - 29.8 °C
b) After 2 hours - 29.8 °C
c) After 3 hours - 30 °C
d) After 4 hours - 30 °C

Experiment switched off.

Current Efficiency 68 %

Observations Flakes of ductile whitish bright Nickel as thin sheets were deposited.

Remarks. The nickel flakes grew outward in the middle towards the anodes. Short circuiting is highly expected under these conditions.

NICKEL ELECTROPLATING

Two Anodes and one Cathode System

Current Density applied - 1.24 A/dm^2 ≡ 11.5 A/ft^2
Voltage applied - 1.0V

Anode to cathode distance - 2.5 inches
Total Time allowed for Electroplating - 4 hours

Nickel ion concentration as the function of time
Time interval for metal ion concentration determination - 1 hour

At the start of experiment -6.98 oz/gallon
a) After 1hour - 7.01 oz/gallon
b) After 2 hours - 6.98 oz/gallon
c) After 3 hours - 7.01 oz/gallon
d) After 4 hours - 6.98 oz/gallon

Chloride ion concentration as nickel chloride
Time interval for metal ion concentration determination - 1 hour

At the start of experiment -9.54 oz/gallon
a) After 1hour - 9.54 oz/gallon
b) After 2 hours - 9.45 oz/gallon
c) After 3 hours - 9.50 oz/gallon
d) After 4 hours - 9.50 oz/gallon

Temperature Recording The thermometer was dipped in corner at a depth of ~2.5 inches.
Time interval for monitoring the temperature changes - 1 hour

At the start of experiment -25.5 °C
a) After 1hour - 25.5 °C
b) After 2 hours - 25.5 °C
c) After 3 hours - 25.5 °C
d) After 4 hours - 25°C

Experiment switched off.
Current Efficiency 70.5 %

Observations Flakes of ductile whitish bright Nickel as thin sheets were deposited.

Remarks. The Nickel deposited is in the form of thin sheets and hence the stirrer speed should be adjusted to avoid the breaking of the nickel sheet from the cathode surface. There is a danger of short circuiting under this set of experimental conditions.

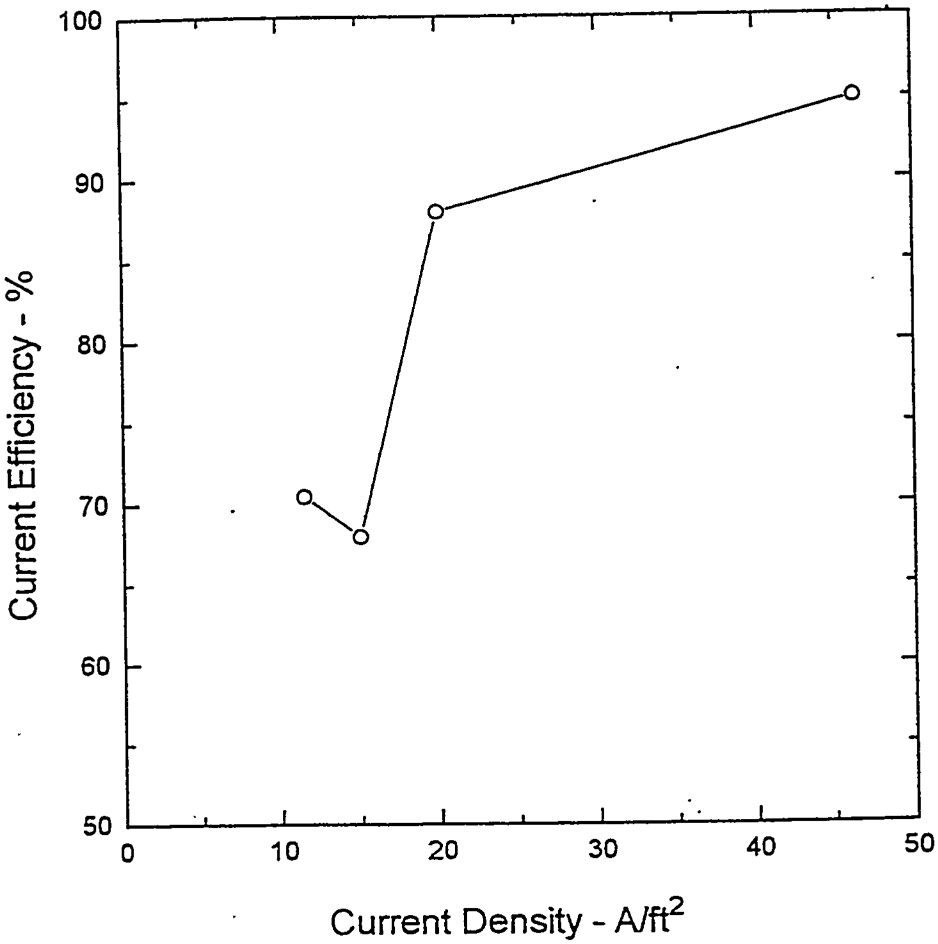


Figure showing the effect of the applied current density on the current efficiency at an anode to cathode distance of 2.5 inches

NICKEL ELECTROPLATING

Two Anodes and one Cathode System

Current Density applied - 2.15 A/dm^2 $\equiv$ 20 A/ft^2
Voltage applied - 1.5 - 2.0V

Anode to cathode distance - 3.0 inches
Total Time allowed for Electroplating - 4 hours

Nickel ion concentration as the function of time.
Time interval for metal ion concentration determination - 1 hour

At the start of experiment -7.29 oz/gallon
a) After 1hour - 7.26 oz/gallon
b) After 2 hours - 7.26oz/gallon
c) After 3 hours - 7.20 oz/gallon
d) After 4 hours - 7.23oz/gallon

Chloride ion concentration as nickel chloride
Time interval for metal ion concentration determination - 1 hour

At the start of experiment -9.54 oz/gallon
a) After 1hour - 9.41 oz/gallon
b) After 2 hours - 9.36oz/gallon
c) After 3 hours - 9.36 oz/gallon
d) After 4 hours - 9.36 oz/gallon

Temperature Recording The thermometer was dipped in corner at a depth of ~2.5 inches.
Time interval for monitoring the temperature changes - 1 hour

At the start of experiment -23 °C
a) After 15 minutes - 23 °C
b) After 40 minutes - 23.5 °C
c) After 1hour - 24.2 °C
b) After 2 hours - 25 °C
c) After 3 hours - 26 °C
d) After 4 hours - 26°C
Experiment switched off.

Current Efficiency 87.5 %
Observations Flakes of ductile whitish bright Nickel as thin sheets were deposited.
Remarks. The Nickel flakes deposited is in the form of thin sheets growing outwards from the sides. The stirrer speed was reduced to avoid the breaking of the nickel flakes from the cathode surface. It is unlike the previous experiments in which case the nickel flakes grew outward in the middle towards the anodes increasing the risk of short circuiting.

NICKEL ELECTROPLATING

Two Anodes and one Cathode System

Current Density applied - 2.15 A/dm^2 ≡ 20 A/ft^2
Voltage applied - 1.5 - 2.0V

Anode to cathode distance - 3.5 inches
Total Time allowed for Electroplating - 4 hours

Nickel ion concentration as the function of time
Time interval for metal ion concentration determination - 1 hour

At the start of experiment - 9.80 oz/gallon
a) After 1hour - 9.80 oz/gallon
b) After 2 hours - 9.80 oz/gallon
c) After 3 hours - 9.70 oz/gallon
d) After 4 hours - 9.80 oz/gallon

Temperature Recording The thermometer was dipped in corner at a depth of ~2.5 inches.
Time interval for monitoring the temperature changes - 1 hour

At the start of experiment -24 °C
a) After 1hour - 26 °C
b) After 2 hours - 26.2 °C
c) After 3 hours - 26.5 °C
d) After 4 hours - 26.7 °C

Experiment switched off.

Current Efficiency 82.6 %

Observations Flakes of ductile whitish bright Nickel as thin sheets were deposited.

Remarks. As the anode-cathode distance is increased he nickel flakes grew outward from the sides. These flakes were as large as 2 inches extending towards the sides into the solution.

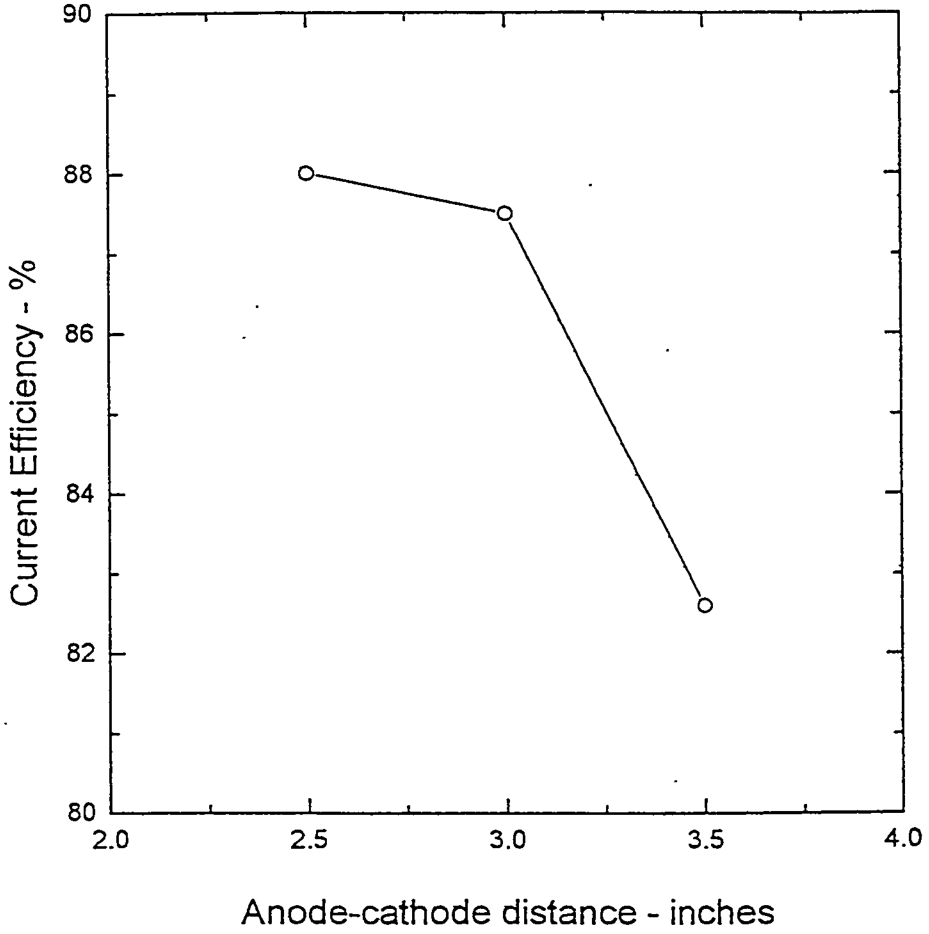
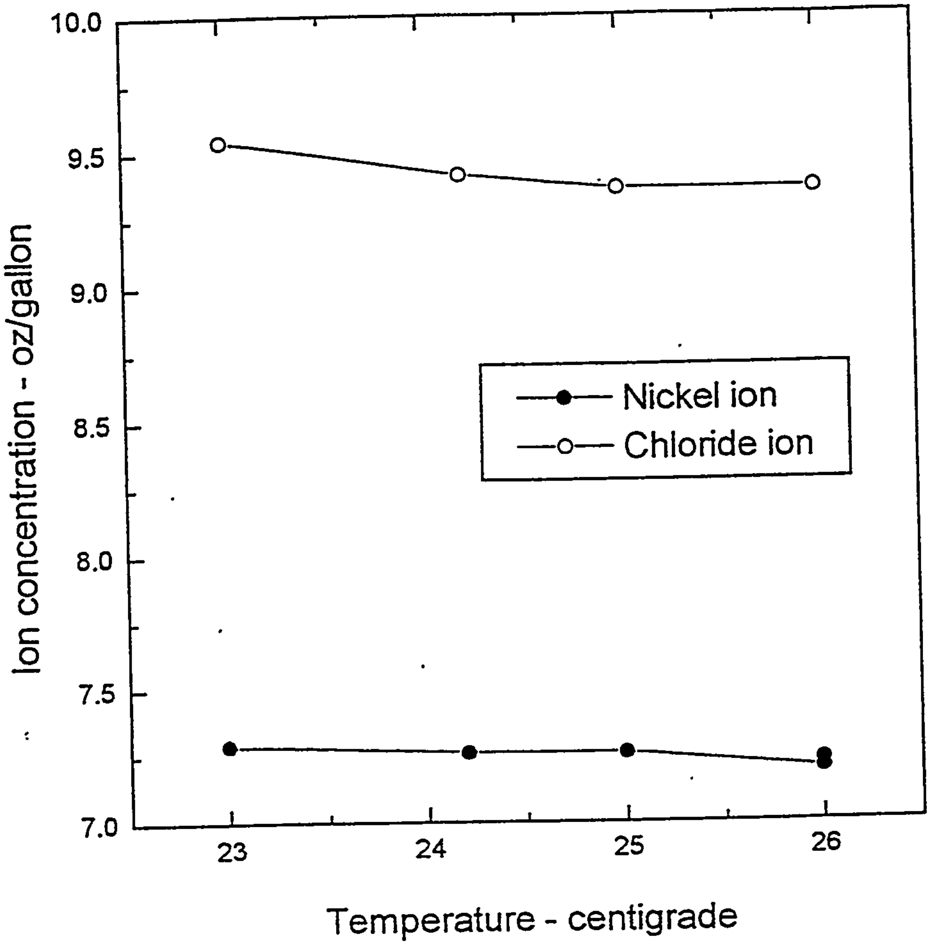


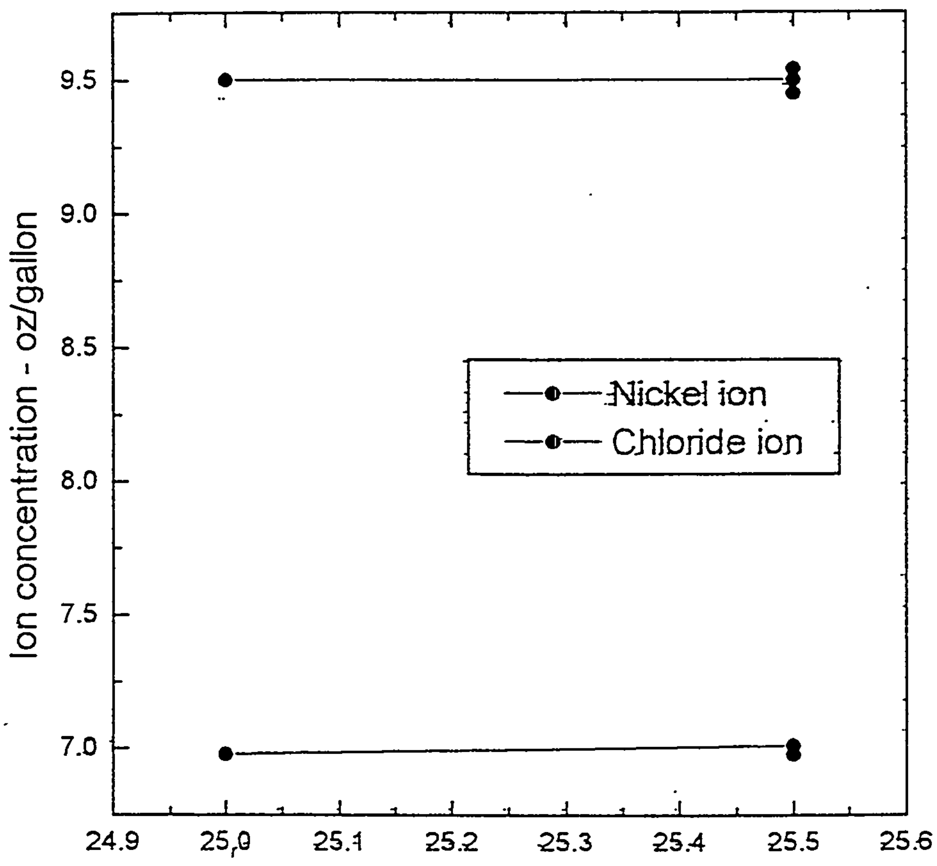
Figure showing the effect of the anode to cathode distance on the current efficiency at an applied current density of 20 A/ft^2



Change in the nickel ion and the chloride ion concentration with temperature.

Current density - 20 Amps/ft^2

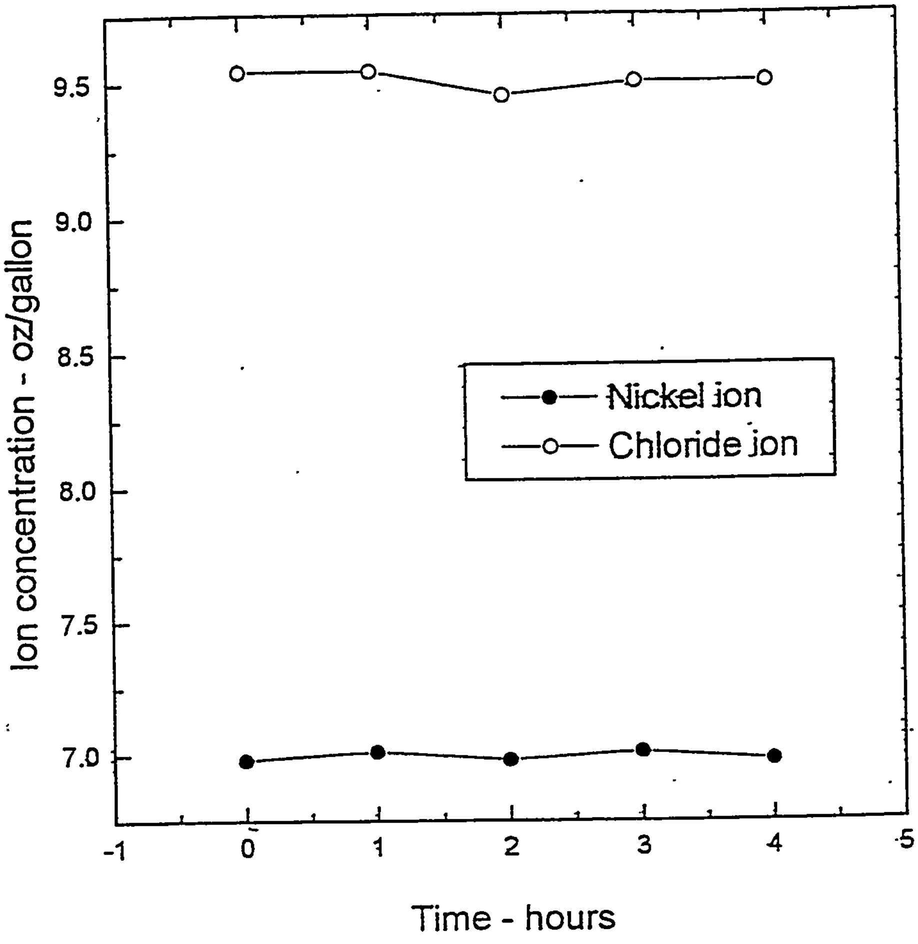
Anode-cathode distance - 3 inches



Change in the nickel ion and the chloride ion concentration with temperature.

Current density - 11.5 Amps/ft^2

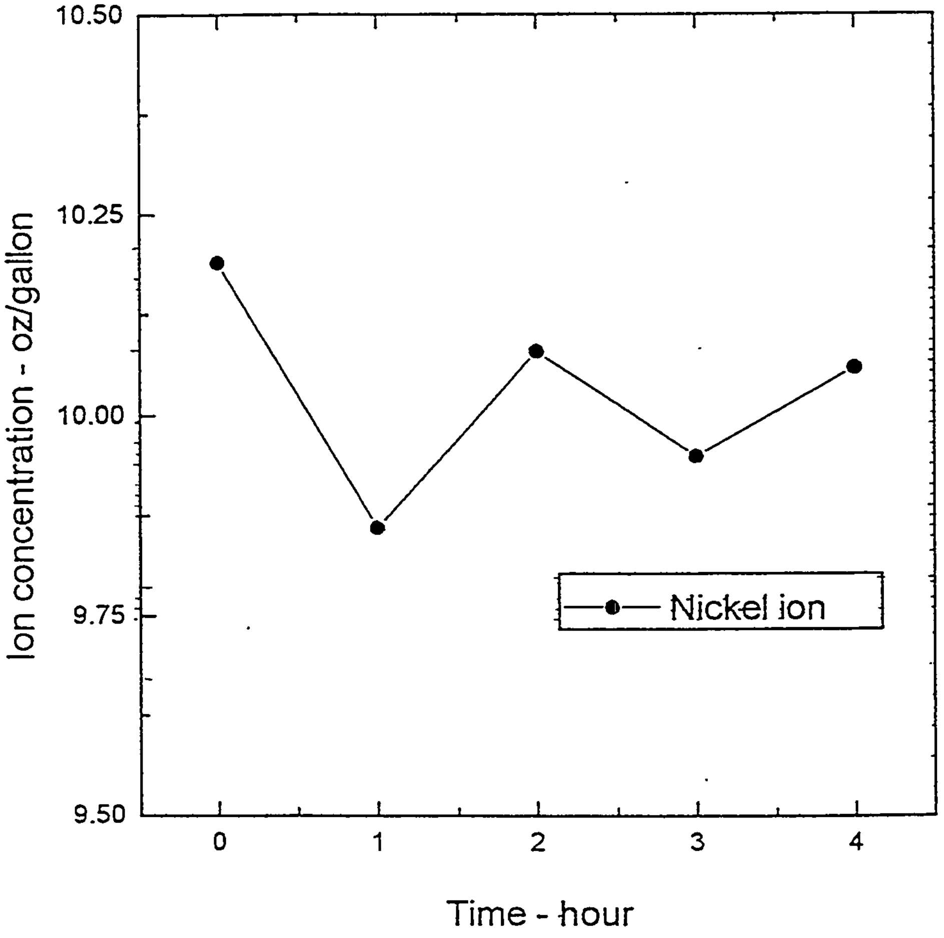
Anode-cathode distance - 2.5 inches



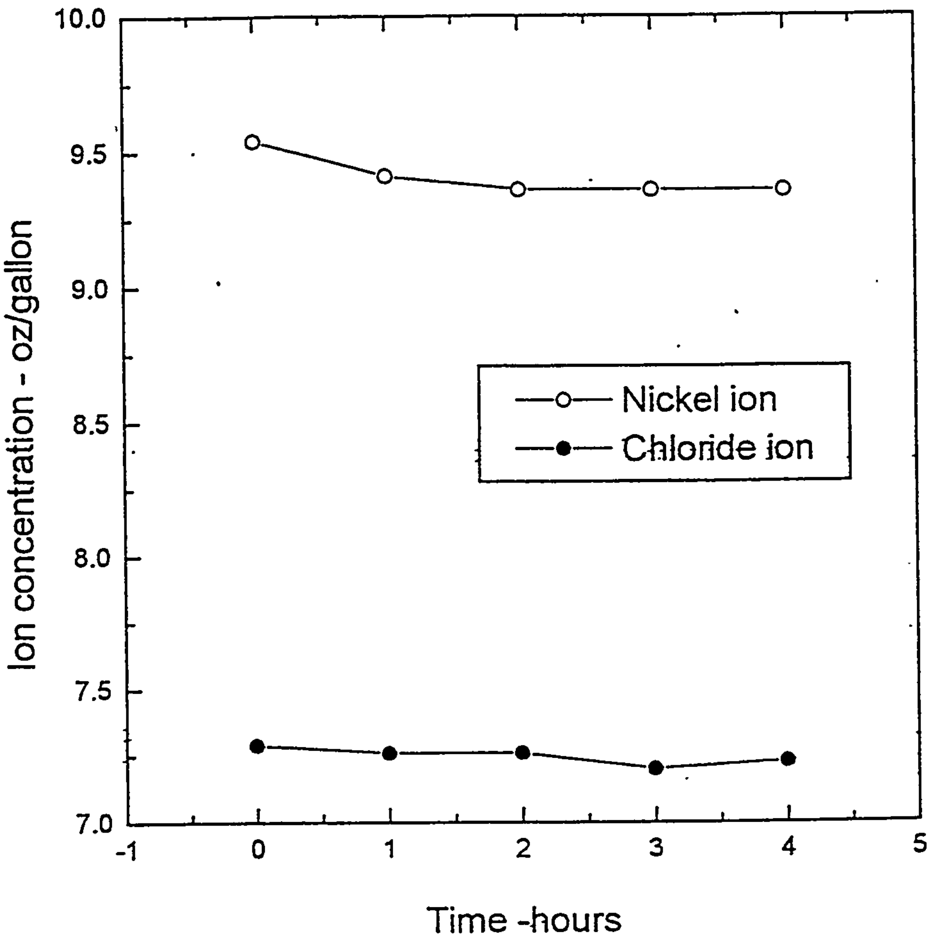
Change in the nickel ion and the chloride ion concentration with time.

Current density - 11.5 Amps/ft^2

Anode-cathode distance - 2.5 inches



Change in the nickel ion concentration with time.
Current density - 20 Amps/ft^2
Anode-cathode distance - 2.5 inches



Change in the nickel ion and the chloride ion concentration with time.

Current density - 20 Amps/ft^2

Anode-cathode distance - 3 inches

MANUFACTURING SCIENCES CORPORATION

U.S. DEPARTMENT OF ENERGY
METC/FETC

TASKS 311, 312, 313 COST SUMMARY

DATE	TASK NUMBERS	ENGINEERING LABOR+OH	PRODUCTION LABOR+OH	OTHER SUBCONTRACTORS COST	OTHER DIRECT COST	15.48% G & A AMOUNT	TOTAL COSTS
1997							
	311	$ 46,546.79	$ 189,559.79	$ 4,281.71	$ 151,219.66	$ 24,071.61	$ 415,679.56
	312	$ 8,596.24	$ -	$ 23,165.91	$ 248.74	$ 3,624.59	$ 35,635.47
	313	$ 228.05	$ 190.44	$ -	$ -	$ -	$ 418.49
TOTAL		$ 55,371.07	$ 189,750.23	$ 27,447.62	$ 151,468.40	$ 27,696.20	$ 451,733.52
1998							
	311	$ 46,609.10	$ 73,648.54	$ 8,843.12	$ 124,643.55	$ 20,663.74	$ 274,408.05
	312	$ 3,787.69	$ -	$ -	$ 94.00	$ 14.55	$ 3,896.25
	313	$ -	$ -	$ 39,459.26	$ 17,686.24	$ 8,846.12	$ 65,991.62
TOTAL		$ 50,396.80	$ 73,648.54	$ 48,302.38	$ 142,423.79	$ 29,524.41	$ 344,295.92
1999							
	311	$ -	$ 9,702.28	$ -	$ 22,033.48	$ 3,410.78	$ 35,146.55
	312	$ -	$ -	$ -	$ -	$ -	$ -
	313	$ -	$ -	$ -	$ -	$ -	$ -
TOTAL		$ -	$ 9,702.28	$ -	$ 22,033.48	$ 3,410.78	$ 35,146.55
GRAND TOTAL		$ 105,767.87	$ 273,101.05	$ 75,750.00	$ 315,925.67	$ 60,631.39	$ 831,175.99

OTHER DIRECT COSTS SUMMARY - TASKS 311, 312, 313 (ELECTROREFINING)

Numbers are approximate

Description	Includes	Price
Electrical supplies	Controllers, analyzers, meters, panel meters, instrumentation, power supplies, heaters	$27,858
Mold (anode melting)		$24,850
Main cell tank	Polyconcrete ~15' long	$5,300
Tanks, frames, piping	Sump, cementation, MRU, pipe, fittings, flex duct, spheres, gaskets, clamps, membrane frame	$33,967
External Support services	Design, safety analysis, programming, calculations, consultation, reporting	$63,234
Waste Tank	Tank for holding waste solutions	$1,365
Pumps		$2,842
External Installation	Install ducts, fans, scrubber, etc.	$12,350
Shipping		$1,802
Metal fab	Purchase or copper, steel, fabrication, stands, racks, buss, starter sheets, cementation starter, etc.	$36,784
Membrane frames	Cathode boxes	$17,585
Chemicals		$19,181
Anode bags		$354
Cationic membrane		$21,106
Miscellanous		$47,348
Total		$315,926

Appendix 3.2

Final Report

Project No. 40578CSP

on

Remote Laser Cutting Demonstration

to

Rockwell International
Canoga Park, CA

February 24, 1997

Lorne Weeter
EDISON WELDING INSTITUTE
1250 Arthur E. Adams Drive
Columbus, OH 43221

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Remote Laser Cutting Demonstration

1.0 Introduction

Rockwell is evaluating laser technology to size reduce radioactively contaminated power generation plants. The laser cutting process offers:

- Remote processing capability
- Low emission production
- Low consumable costs.

Because of these benefits, laser cutting is considered one of the prime processes for the size-reduction projects.

2.0 Objectives

The objective of this work was to use two different cutting end effectors to cut 0.375-in.-thick steel pipe. ETEC had prepared a matrix to establish a set of parameters for subsequent laser cutting demonstration tests. EWI used this matrix of parameters to vary power, travel speed, focal position, cutting gas, and cutting pressure to achieve "acceptable" cuts.

3.0 Testing

The chemical composition of the 0.375-in.-thick steel pipe that was used for the cutting trials is listed in Table 1. The pipe diameter was 12.75 in.. Rockwell agreed that this standard pipe would adequately simulate the piping that must be size reduced in the various applications.

All of the laser cutting trials were performed with a Lumonics/Hobart HLP3000 continuous wave Nd:YAG laser. The laser was operated at a maximum output of 2000 watts to simulate the achievable power of the MW2000 laser that Rockwell is leasing for additional trials. The power level was varied from 1000 to 2000 watts to determine the affect of output power on cutting speed and cut quality.

A 15-m-long fiberoptic cable was used to deliver the laser beam to the workpiece. The fiberoptic cable has a 0.6-mm-diameter fused silica core. Typical power losses for a 15-m-long fiberoptic cable are approximately 8% (3.5% at each end of the fiber and 1% in the fiber itself). Combined with the 2% loss in the beam injection optics inside of the laser and the 2% loss in the beam focusing optics at the end of the fiber, the total power loss is approximately 12%.

Two optical end effectors with different F numbers (the F number is the focal length divided by the beam diameter at the focusing optics) were used to perform the cutting. An F4 end effector that can produce a 1.2-mm spot size and an F8 end effector that can produce a 2.4-mm spot size were selected due to the thickness of the pipe and the long depth-of-field of these end effectors. Both end effectors use four optics to recollimate and focus the beam after the laser beam exits the fiberoptic cable. Both end effectors used 50-mm-diameter optics.

A standard co-axial cutting cone and nozzle were used to direct the cutting gas. A copper cutting nozzle with two different diameters (2.5 mm for the F4 and 4.4 mm for the F8) was used.

Both oxygen and air were used as the cutting gas. The gas pressures were varied from 10-60 psi for oxygen and 100-120 psi for air.

The range of parameters and values used for the cutting trials is listed in Table 2.

The experimental set-up and cutting end effector are shown in Figure 1.

A Motoman K60S articulated arm robot was used to locate the optical end effector above an Aronson rotary-tilt positioner. The speed of the rotary positioner was varied to provide the different cutting speeds. The rotary positioner had approximately 3 mm of run-out. This run-out affected the focal position of the beam on the cuts for the longer cuts or the 360-degree cuts, but did not dramatically affect the cut quality or the shorter cuts. The focal position was measured at various points to record the actual dimension. The robotic set-up, without the rotary positioner in place, is shown in Figure 2.

4.0 Results

4.1 F4 Optical End Effector Trials

The results of the better cuts for the F4 end effector trials are summarized in Table 3. Various tests were performed outside of the tabulated values to ensure that the best values were achieved. The total list of trials is included in Appendix A.

Results using the F4 (1.2-mm spot size) cutting end effector showed that the 0.375-in.-thick steel could be cut with minimal dross on the backside at laser powers between 1000 and 2000 watts (leaving the laser) and speeds of 26 to 32 ipm with a oxygen assist gas at 18 psi. The focal position could be varied ±3 mm (0.120 in.) with little affect on the cut quality or speed. Figure 3 shows the cut surface for the 2000 watt, 32 ipm with oxygen trial.

The main controlling parameters were gas type and gas pressure. Oxygen at too low or too high of gas pressure caused excessive dross on the backside. With the oxygen cutting gas set at 18 psi, the laser power could be varied from 1000 to 2000 watts with only a 6-ipm reduction in cutting speed (from 32 ipm at 2000 watts to 26 ipm at 1000 watts). The focal position also seemed to have minimal affect on the cut quality.

Parameters that cut through the material but left lots of dross could also be produced with air assist gas at 120 psi of gas pressure, but the cutting speed had to be reduced to approximately 10 ipm. The surface roughness of the air gas assist cuts also seemed rougher than the oxygen cuts.

The kerf width varied from 0.038 to 0.070 in. depending on the parameters. At slow speeds and high powers, the kerf width was wide. At faster speeds or lower powers, the kerf is narrower. The width of the cut is important in producing emissions or fumes and providing the path for the dross to bridge the gap. A wider kerf may be desirable to ensure that the two pieces fall apart after cutting.

4.2 F8 Optical End Effector Trials

The results of the F8 end effector trials are summarized in Table 4. Only the better cuts are tabulated in Table 1. The total list of trials are included in Appendix B.

The F8 end effector (2.4-mm spot size) trials were similar to the F4 trials except that a much higher oxygen flow level was required and the cutting speed was slightly slower. To produce acceptable cuts, the oxygen pressure had to be increased to 60 psi. The cutting speed decreased from 26 ipm at 1000 watts with an F4 to 20 ipm at 1000 watts with an F8 and from 32 ipm at 2000 watts with an F4 to 26 ipm at 2000 watts with an F8. Figure 4 shows the cut surface for the 2000 watt, 26 ipm with oxygen trial.

Again, acceptable cuts could be made from 1000 to 2000 watts. Similar to the F4 results, the gas type and gas pressure were the controlling parameters. Oxygen at too low or too high gas pressure caused excessive dross on the backside.

Air gas assist did not produce acceptable cuts. 2700 watts of laser power was used to determine if a higher laser power would help the cutting process. Even with the higher laser power, the cut was very heavy with dross.

The kerf width varied from 0.083 to 0.133 in. for the F8 end effector. The wider kerf was expected since the focused spot size is 1.2 mm (0.048 in.) wider than the F4. Although the F8 would produce more emissions than the F4, the F8s wider kerf width may be advantageous to ensure the pieces fall apart.

5.0 Discussion

The results indicate that the exothermic reaction of the oxygen cutting gas and the iron in the steel create the majority of the energy needed to melt the steel. The laser provides the initial heat source needed to reach this exothermic reaction level and some additional cutting power. However, doubling the laser power does not double the cutting speed since the oxygen is doing most of the cutting. The correct amount of oxygen per volume of steel is needed to produce acceptable cuts.

Too little oxygen for a set travel speed does not input enough oxygen to successfully cut through the steel. Increasing the laser power at low oxygen flows does help, but cannot overcome the loss of the oxygen.

Too much oxygen for the volume of metal in the kerf causes excessive melting of the steel. The oxygen's gas pressure can not force this excessive molten steel through the kerf and dross/burr is left on the backside of the cut. Increasing the cutting speed does not necessarily help since then not enough oxygen and laser power is delivered to melt the steel.

Since air is approximately 82% nitrogen, it does not produce the same exothermic reaction as oxygen. Thus, the cutting speed and cut quality are both reduced.

The F8 (2.4-mm spot size) optical end effector requires 3.3 times more oxygen pressure (60 psi compared to 18 psi) to achieve acceptable cuts. The F8 needed a nozzle opening of 4.4 mm compared to 2.5 mm to allow the laser beam to pass through the nozzle opening without clipping the nozzle. This larger nozzle diameter opening calculates to a 3-time increase in area of the nozzle opening. Because of this 3-time increase in area, the oxygen pressure had to be increased approximately 3 times to achieve the same volume of gas reaching the cutting surfaces.

The amount of additional oxygen used per cut is also compounded by the slower cutting speeds of the F8 end effector. The power density (laser power divided by the focused spot area) is 4 times less than the F4 end effector. As explained above, the oxygen cutting gas supplies the majority of the energy for cutting; thus, the lower power density does not have a major affect but it does decrease the cutting speed from 18 to 26%.

Because of the larger kerf width, the F8 end effector also generates more emissions than the F4 end effector. If we assume all of the material in the kerf produces emissions either as gas or as resolidified debris on the floor, the F8 end effector produce approximately 1.8-2.0 times as much emissions as the F4. These extra emissions will increase the costs of the fume filters and other associated emission devices that must be properly disposed.

The main reason for using the F8 end effector was to achieve a longer depth of field. A longer depth of field should permit more tolerance for focal position and possibly produce better cuts in thick materials. However, the results of this test did not demonstrate the benefits of a longer depth of field for this application. For these trials, it was assumed that the focal position would be fixed to the end of the nozzle. Thus, if the focus moved away from the part, the nozzle would also move the same distance away from the part. This was done to simulate the production application of laser cutting. Therefore, this study actually evaluated the compounded influence of focus and nozzle stand-off (the distance from the cutting nozzle to the workpiece) distance. Since this study has clearly demonstrated the major influence of the oxygen cutting gas on the cutting speed and quality, it is believed that the nozzle stand-off distance overshadowed the depth-of-field affect. This study did show that the nozzle stand-off and focus could change by as much as ±3 mm without detrimentally affecting the cutting speed or cut quality. For the production cutting cell, surface followers are available that can easily maintain the nozzle stand-off and focus to within ±0.2 mm.

Since only one material thickness was cut, it is impossible to quantify the affect of the F8's longer depth of field on cutting thicker material. Additional trials are required to assess this effect.

The F8 end effector did produce a wider kerf width than the F4 end effector. This wider kerf width helps keep the molten dross from bridging the gap and "rewelding" the cut closed. In production, this wider kerf width may be beneficial for remote cutting applications where it is difficult to maintain consistent conditions or the material thickness is changing.

6.0 Conclusions and Recommendations

The results from this study generate the following conclusions and recommendations:

1. The type of cutting gas and the cutting gas pressure are the major controlling factor for cutting thick steel. Oxygen generated the best cuts. The oxygen pressure depended greatly on the diameter of the cutting nozzle opening.

2. Consistent, acceptable cuts can be produced in 0.375-in.-thick steel material throughout a wide range of laser parameters. The laser power could change 50% with only a 20% change in speed required to maintain acceptable cuts.

3. The nozzle stand-off can vary ±3 mm without greatly affecting the cutting speed or cut quality. In production, a device that maintains a set stand-off should be incorporated into the cutting system.

4. The F4 end effector produced faster cuts and narrower kerf widths with 3 times less oxygen consumption than the F8 end effector.

5. Although not quantified, the F4 end effector should produce fewer emissions than the F8 end effector.

6. The wide kerf width of the F8 end effector may be beneficial for certain production applications where it is difficult to control the laser parameters.

7.0 Possible Follow-On Projects

The results of this study have shown several key factors for size reducing radioactively contaminated fabrications. Several important areas still exist to properly understand the laser cutting process for these applications.

1. Quantify the amount of emissions produced with both the F4 and F8 end effectors at the different laser parameters.
2. Evaluate cutting thicker (0.5-in.-thick) steel.
3. Optimize the cutting parameters for various thicknesses of steel.
4. Assist with the design of a production cutting system.
5. Assist in training operators and transferring the knowledge gained from this study to production.

EWI offers the capability, equipment and experience to assist Rockwell with additional portions of this project. We can provide assistance at EWI, at Rockwell or at Manufacturing Sciences Corporation. Please let us know how we can help you.

For more information, contact: Lorne Weeter at 614-688-5239.

Table 1. Chemical Composition of Steel Pipe

Element	Composition, weight %
C	0.072
Mn	0.098
P	0.009
S	0.007
Si	0.190
Cu	0.013
Sn	0.015
Ni	0.004
Cr	0.018
Mo	0.012
Al	0.028
V	0.000
Nb	0.035
Zr	0.001
Ti	0.001
Ca	0.001
Co	0.001
Fe	Balance

Table 2. Range of Parameters Used in Study

Variables	High Value	Low Value	Comments
Power (watts)	2000	1000	Measured at laser
Cutting speed (m/min)	32	10	
Gas pressure (psi)	60/120	10/80	Oxygen/air
Gas type	oxygen	air	
Focus (± mm)	+4	-2	
Nozzle distance (mm)	+4	0.2	3 mm is nominal
Constants			
Nozzle opening	2.5/4.4	2.5/4.4	F4/F8 optics
Fiber length (meters)	15	15	
Motion system	robot	robot	
Material	3/8-in. steel	3/8-in. steel	

Table 3. Cutting Results for F4 End Effector

Trial No.	Power (watts)	Speed (ipm)	Focus (mm)	Nozzle Diameter (mm)	Gas Type	Gas Pressure (psi)	Stand-off (mm)	Comments
2	2000	10	0	2.5	Air	100	3	Dross, almost through
4	2000	10	-2	2.5	Air	100	2	Still dross, but best cut so far
10	2000	10	0	2.5	Air	120	1.5	Still dross, but slightly better
14	2000	32	-2	2.5	O_2	18	1.5	No cut
17	2000	32	0	2.5	O_2	18	3.0	Cut all the way around. looks good.
19	1800	26	0	2.5	O_2	18	3.0	Fairly clean cut
27	1200	26	0	2.5	O_2	20	3.0	Nice cut. fairly clean
30	1000	26	0	2.5	O_2	20	3.0	Nice cut. fairly clean

Table 4. Cutting Results for F8 End Effector

Trial No.	Power (watts)	Speed (ipm)	Focus (mm)	Nozzle Diameter (mm)	Gas Type	Gas Pressure (psi)	Stand-off (mm)	Comments
3a	2000	20	0	4.4	O_2	35	3.0	Lots of dross, but would separate
5a	2000	26	0	4.4	O_2	60	2.0	Kerf is 0.133 in. good cut.
6a	1500	32	0	4.4	O_2	[illegible]	2.0	More dross, but would separate.
9a	1000	20	0	4.4	O_2	60	2.0	Full separation. some dross. Kerf = 0.094 in.
10a	2000	10	0	4.4	air	120	2.0	No cut
11a	2700	10	0	4.4	air	120	2.0	Looks like it would separate, but lots of dross. Kerf = 0.098 in.

Figure 1. Picture of the Experimental Set-up and Cutting end Effector

Figure 2. **Picture of the Robotic Set-Up, Without the Rotary Positioner in Place**

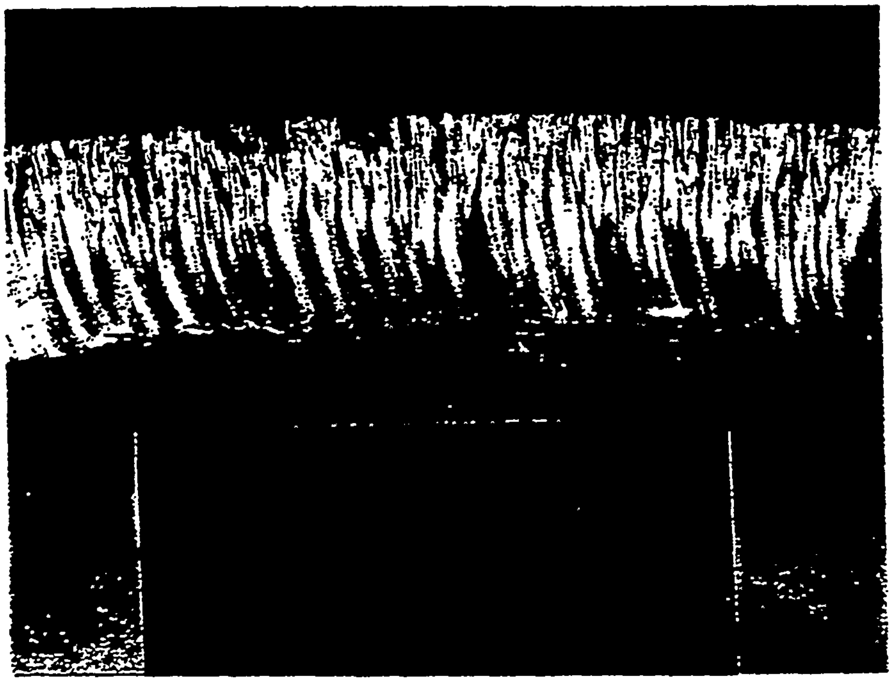

Figure 3. **Photomicrograph of Cut Surface Made with F4 End Effector** (2000 watts, 32 ipm and oxygen assist gas)

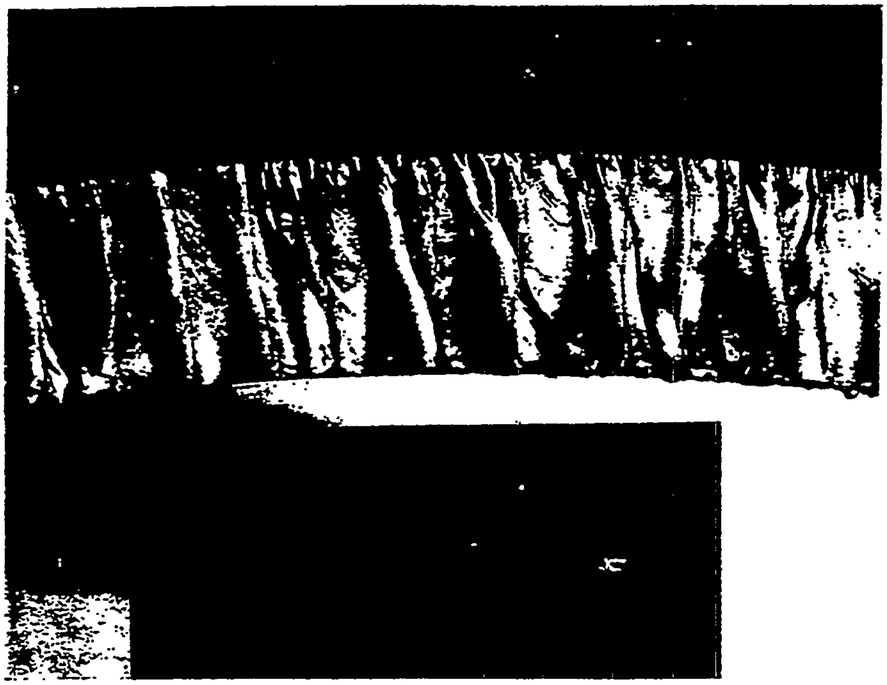

Figure 4. **Photomicrograph of Cut Surface Made with F8 End Effector** (2000 watts, 26 ipm and oxygen assist gas)

Appendix A

PROJECT 40578

SHEET NO. 1

MATERIAL NO.

OPERATOR S. Wirth

MACHINE HLP3000

MACHINE TOOLING Motoman K60 / Aronson Rotary track

FOCUSING SYSTEM F4 End Effector

ADDITIONAL INFORMATION

MATERIAL DETAILS .375" Steel

FOCUS SETT

+V
0 V
– V

Date	Specimen No.	Laser power, kW	Travel speed, m/min	Focus setting	Cutting data			Focal length	Standoff	General comments
					Nozzle #	Gas	Press.			
10/8/96	1	2000 * at Laser	10 In/min	0	2.5 mm	Air	100 psi	F4	3mm	.05" Kurph Width top. Almost Through cut
10/8/96	2	2000 * at laser	10 I/min	0mm	2.5 mm	Air	100psi	F4	3mm	retry of #1
10/8/96	3	2000 * at laser	10/5 in/min	+4mm	2.5 mm	Air	100 psi	F4	3mm	Not as clean a cut as 1 and 2 kurph – .075" top
10/8/96	4	2000 * at laser	10 In/min	-2mm	2.5 mm	Air	100psi	F4	2mm	Best cut so far Still Drosse Kurph – .05" top
10/8/96	5	2000 * at Laser	10 In/min	"	"	"	"	"	"	repeat of #4
10/8/96	6	2000 * at Laser	10 In/min	-2mm	2.5 mm	oxygen	18psi	F4	2mm	.05" Kurph width Through cut.
10/8/96	7	2000 * at Laser	10 In/min	0	2.5 mm	Oxygen	18 psi	F4	3mm	lots of Drosse Kerf= 0.070"
10/8/96	8	2000 * at Laser	10 In/min	0	2.5 mm	Oxygen	10 psi	F4	3mm	Varied oxygen pressure & speed nothing Better happened. Kerf= 0.0
10/8/96	9	2000 * at Laser	10 In/min	0	2.5 mm	OXY	18 PSI	F4	3mm	End effector turned for better nozzle alignment. (lots of Drosse)

* indicates power stabilization off, gives ≥ 1700 watts on part

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LASER & FORGE WELDING SECTIC

DATA SHEET

PROCESS: **LASER CUTTING**

PROJECT NO. 40578

CONTINUATION SHEET

SHEET NO. 2

Details as sheet No. 1

Date	Specimen No.	Laser power, kW	Travel speed, m/min	Focus setting	Cutting data Nozzle #	Cutting data Gas	Cutting data Press.	Focal length	Standoff	General comments
10/9/96	10	2000 W AT Laser	10 m/min	0	2.5 mm	Air	120 psi	F4	3 mm	Same as # 9 except using Air
10/9/96	11	"	"	~~0~~ -2mm	"	"	"	"	~~[illegible]~~ 1.5 mm	Lots of Dross
10/9/96	12	"	"	"	"	OXY	18 psi	F4	1.5 mm	Cleanest cut yet Kerf=0.063
10/9/96	13	2000 W	20 fpm ~~m/min~~	-2 mm	2.5 mm	OXY	18 psi	F4	1.5 mm	Looks Good. Table set 38 ~~Best~~ Better than 12 Kerf=0.048
10/9/96.	14	2000 W	32 fpm	-2mm	2.5 mm	OXY	18 psi	F4	1.5 mm	Not Through Table 45.9
10/9/96	15	2000 W	26 fpm	-2mm	2.5 mm	OXY	18 psi	F4	1.5 mm	~~[illegible]~~ Nice cut Table 38 Low pressure
10/9/96	16	2000 W	26 fpm	0	2.5 mm	OXY	18 psi	F4	3 mm	A fairly Clean cut. Kerf=0.038
10/9/96.	17	2000 W	32 fpm	0	2.5	OXY	18 psi	F4	3 mm	Cut All the Way Around Looks Good (45.9)
10/9/96	18.	1800 W AT LASER	32 fpm	0	2.5 mm	OXY	18 psi	F4	3 mm	No cut

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A pierce program would help eliminate the ball of molten metal at the start of each cut.

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LASER & FORGE WELDING SECT

DATA SHEET

PROCESS: **LASER CUTTING**

PROJECT NO. 40578

CONTINUATION SHEET

SHEET NO. 3

Details as sheet No. 2

Date	Specimen No.	Laser power, kW	Travel speed, m/min	Focus setting	Cutting data			Focal length	Standoff	General comments
					Nozzle #	Gas	Press.			
10/9/96	19	1800 * At Laser	26 Spm	0	2.5 mm	OXY	18 psi	F4	3 mm	Fairly clean cut (Tank set 3a b)
10/9/96	20	1800 * At Laser	26. Spm	0	2.5 mm	~~OXY~~ Air	120 psi	F4	3 mm	Did not cut.
10/9/96	21	1800 * At Laser	26 Spm	+3 mm	2.5 mm	OXY	18 psi	F4	6 mm	Did not cut
10/10/96	22	1800 * At Laser	26 Spm	0	2.5 mm	OXY	10 psi	F4	3 mm	No cut
10/10/96	23	2000 At Laser	26 Spm	0	2.5 mm	OXY	10 psi	F4.	3 mm	No. cut.
10/10/96	24	2000 * At Laser	26 Spm	0	2.5 mm	OXY	15 psi	F-4	3 mm	an O.K. cut but still some Drouse
10/10/96	25	2000 * At Laser	26 Spm	0	2.5 mm	OXY	20 psi	F-4	3 mm	Good cut Low Drouse
10/10/96	26	1500 At Laser	26 Spm	0	2.5 mm	OXY	20 psi	F4	3 mm	Nice cut fairly clean
10/10/96	27.	1200 At Laser	26 Spm	0	2.5 mm	OXY	20 psi	F4	3 mm	Nice cut fairly clean

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LASER & FORGE WELDING SECTI

DATA SHEET

PROCESS: **LASER CUTTING**

PROJECT NO. 40578

CONTINUATION SHEET

SHEET NO. 4

Details as sheet No. 3

Date	Specimen No.	Laser power, kW	Travel speed, m/min	Focus setting	Cutting data Nozzle ø	Cutting data Gas	Cutting data Press.	Focal length	Standoff	General comments
14/10/96	28	1000* AT Laser	26 Ipm	0	2.5 mm	OXY	20 psi	F4	3 mm	Nice cut
14/10/96	29	800* AT Laser	2.6 Ipm	0	2.5 mm	OXY	20 psi	F4	3 mm	Lots of Drouse.
10/1/96	30	1000* AT Laser	26 Ipm	0	2.5 mm	OXY	20 psi	F4	3 mm	Looks Good.

Appendix B

DATA SHEET **PROCESS: C LASER CUTTING** **LASER & FO WELDING SECTIO**

PROJECT No.	40578	MACHINE TOOLING	K-60 Motoman Robot
SHEET NO.	1	FOCUSING SYSTEM	F-8
MATERIAL NO.		ADDITIONAL INFORMATION	To Separate Material
OPERATOR	S Wirin		
MACHINE	HLP 3000	MATERIAL DETAILS	.575" Steel

FOCUS SETT

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- V

Date	Specimen No.	Laser power, kW	Travel speed, m/min	Focus setting	Cutting data			Focal length	Standoff	General comments
					Nozzle #	Gas	Press.			
12/13/96	1A	2000 watts	10 IPM	0	4.4 mm	OXY	25 psi	F-8	≈8mm	Almost through cut
12/13/96	2A	2000 watts	20 IPM	0	4.4 mm	OXY	25 psi	F-8	≈8mm	No cut
12/13/96	3A	2000 watts	20 IPM	0	4.4 mm	OXY	35 psi	F-8	≈3mm	Big Drouse But would Separate kerf width (.13"
12/13/96	4A	2000 watts	20 IPM	0	4.4 mm	OXY	45 psi	F-8	≈3mm	Still some Drouse but a obstruction free cut. links G
12/13/96	5A	2000 watts	26 IPM *	0	4.4 mm	OXY	45/60 psi	F-8 = 0.106 @ 45psi Kerf = 0.133 @ 60psi	≈3mm	Started at 45 psi upped to 60 psi (AT 60psi with 2mm standoff good cut)
12/13/96	6A	1500 watts	26 IPM	0	4.4 mm	OXY	60 psi	F-8 Kerf =	≤3mm 0.083"	little more drouse, but still a seperation.
12/13/96	7A	1300 watts	32 IPM	0	4.4 mm	OXY	60 psi	F-8 Kerf =	≈2mm 0.094	Still more Drouse but still Seperated.
12/13/96	8A	1000 watts	26 IPM	0	4.4 mm	OXY	60 psi	F-8	≈2mm	No cut!
12/13/96	9A	1000 watts	20 IPM	0	4.4 mm	OXY	60 psi	F-8	≈2mm	Full Separation some Drouse .094

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LASER & FORGE WELDING SECT

DATA SHEET

PROCESS: CO_2 LASER CUTTING

PROJECT NO. 40578

CONTINUATION SHEET

SHEET NO.

Details as sheet No. 1

Date	Specimen No.	Laser power, kW	Travel speed, m/min	Focus setting	Cutting data			Focal length	Standoff	General comments
					Nozzle ø	Gas	Press.			
12/13/96	10 A	2000 watts	10 ipm	0	4.4 mm	Air	120 psi	F-8	≈ 2mm	no cut
* 12/13/96	11A	2700 * watts	10 IPM	0	4.4 mm	Air	120. psi	F-8	≈ 2mm	Looks like it would Sperate, But lots of Drosse. (Kurf .098")

* = MAX output power attainable

REMOTE LASER SIZE-REDUCTION OF RADIOACTIVE COMPONENTS

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ABSTRACT

A technology development program was undertaken to evaluate the effectiveness of remote laser cutting in decontamination and decommissioning (D&D) activities. The program employed an off-the-shelf laser with fiberoptic beam delivery to size-reduce various components. It included four distinct steps: (1) an initial series of laboratory tests to establish the cutting capabilities of a commercially available Nd:YAG laser with a fiberoptic beam delivery system, and to develop laser system parameters for subsequent deployment demonstration programs; (2) a deployment demonstration at the Energy Technology Engineering Center (ETEC), where a large inventory of radiologically contaminated reactor fuel storage tubes was size-reduced for disposal using remote laser cutting; (3) a follow-on deployment demonstration at the Manufacturing Sciences Corporation (MSC), where the remote laser cutting operation was further automated and used to size-reduce thicker materials; and (4) a subsequent series of laboratory bench-scale tests to enhance the laser cutting data base in areas identified by the deployment demonstrations. The results of the program demonstrate that an off-the-shelf laser system with fiberoptic beam delivery can be used effectively in D&D applications.

I. INTRODUCTION

The DOE complex and the commercial nuclear industry encompass a large number of nuclear reactor and processing facilities that require near-term dismantlement. The dismantlement tasks are major challenges for which technology innovations in size reduction, decontamination, and characterization are required to decrease operating costs and personnel exposures over conventional techniques. The use of lasers has been investigated recently for nuclear facility decontamination and decommissioning (D&D) tasks. A preliminary Oak Ridge National Laboratory study showed that approximately $1 billion can be saved by using lasers for the size reduction of diffusion plant structural components at the K-25 Plant in Oak Ridge, Tennessee.[1] The Energy Technology Engineering Center (ETEC) investigated the application of laser technology to dismantlement tasks and waste management using fiberoptic beam delivery to remote cutting sites, and found it to offer substantial benefits as a new D&D tool.[2] The ETEC investigation identified two laser systems as promising candidates for remote laser cutting in D&D applications: Nd:YAG (neodymium-doped yttrium aluminum garnet) for current, off-the-shelf use, and the chemical oxygen iodine laser (COIL) for later-term use at much higher laser output powers. Both lasers have wavelengths that transmit through optical fibers with minimum power loss for remote beam delivery. Based on the study results, several bench-scale tests and two deployment tests were performed to investigate and demonstrate the effectiveness of available Nd:YAG lasers for D&D size-reduction activities.

The size-reduction test activities included four steps that lead toward the large-scale implementation of lasers in D&D applications: (1) an initial series of laboratory tests to establish Nd:YAG cutting capabilities and to develop laser system parameters for the subsequent deployment demonstration programs; (2) a deployment demonstration at ETEC to size-reduce a large inventory of radiologically contaminated fuel storage tubes for disposal using remote laser cutting; (3) a follow-on deployment demonstration at the Manufacturing Sciences Corporation (MSC) to further automate the remote laser cutting operation and size-reduce thicker materials; and (4) a subsequent series of laboratory bench-scale tests to enhance the laser cutting data base in areas identified by the deployment demonstrations. The results of the four sets of tests and demonstrations are summarized in this paper.

II. INITIAL LASER CUTTING TESTS

The initial series of laboratory tests was performed by the Edison Welding Institute under contract to ETEC.[3] A continuous-wave (non-pulsed) 3-kW Nd:YAG with a 15-m-long fiberoptic beam delivery system was used to cut 1-cm-thick steel pipe over a laser power range of 1000 to 2700 Watts. Variables examined included beam power, laser end-effector focal length, assist gas type and pressure, and cutting speed. The assist gas is a gas flow across the work area which blows debris created by the cutting process away from the cutting zone and supplies energy required to perform the cutting. An important consideration was the effect of these variables on kerf (cut) width, for which a smaller kerf minimizes secondary waste while a wider kerf helps keep molten dross from bridging the cut and rewelding it. A total of forty-one test cuts were made, with an estimated total power loss associated with the beam delivery system of about 12%. The assist gas type and pressure were found to be the primary parameters controlling the quality of the cuts. The results were used to select a laser focusing head, identify oxygen as the optimum assist gas, and establish initial cutting parameters to be used in the subsequent deployment demonstrations.

III. ETEC LASER DEPLOYMENT DEMONSTRATION

The ETEC laser deployment program used an off-the-shelf 2-kW Nd:YAG laser system to section approximately 300 radiologically contaminated nuclear fuel storage tubes for inspection and disposal.[4] Figure 1 provides a schematic diagram of the general configuration used for the deployment tests. The laser resonator, power supply, water chiller, gas assist supply, and operational controls were located in a nonhazardous environment for protection and maintenance accessibility. The laser focusing head and a tube-holding fixture were located within a radiologically controlled containment tent, which was fitted with a HEPA-filtered ventilation system. The laser beam was delivered to the focusing head through a 15-m-long fiberoptic cable. Each 3-m-long, 12.7-cm-diameter storage tube was positioned horizontally on the fixture, where two longitudinal cuts were made by moving the laser head lengthwise along the tube and one circumferential cut was made by rotating the tube. Each tube was thus sectioned into four approximately equal half-cylinder sections. A continuous-wave laser power level of 0.8 kW was used for these thin-wall (1.6-mm-thick) tubes, with an oxygen assist gas and a linear cutting speed of about 400 cm/min. Each tube was size-reduced in about 2½ minutes, including the time to load each tube and unload the tube sections manually. (Only the laser cutting was automated for this demonstration.) Figure 2 is a photograph of the laser cutting activity.

A number of additional test cuts were performed using a plasma-arc torch to provide comparative data using current-practice technologies. The plasma-arc tests used an identical geometry and cutting procedure. Comparisons showed that the laser and plasma-arc had comparable cutting speeds, but the plasma-arc generated a much larger quantity of secondary waste because of its much larger kerf. That difference, plus the higher plasma-arc maintenance requirements, result in higher cutting costs for the plasma-arc system. Cost comparisons for the ETEC demonstration,

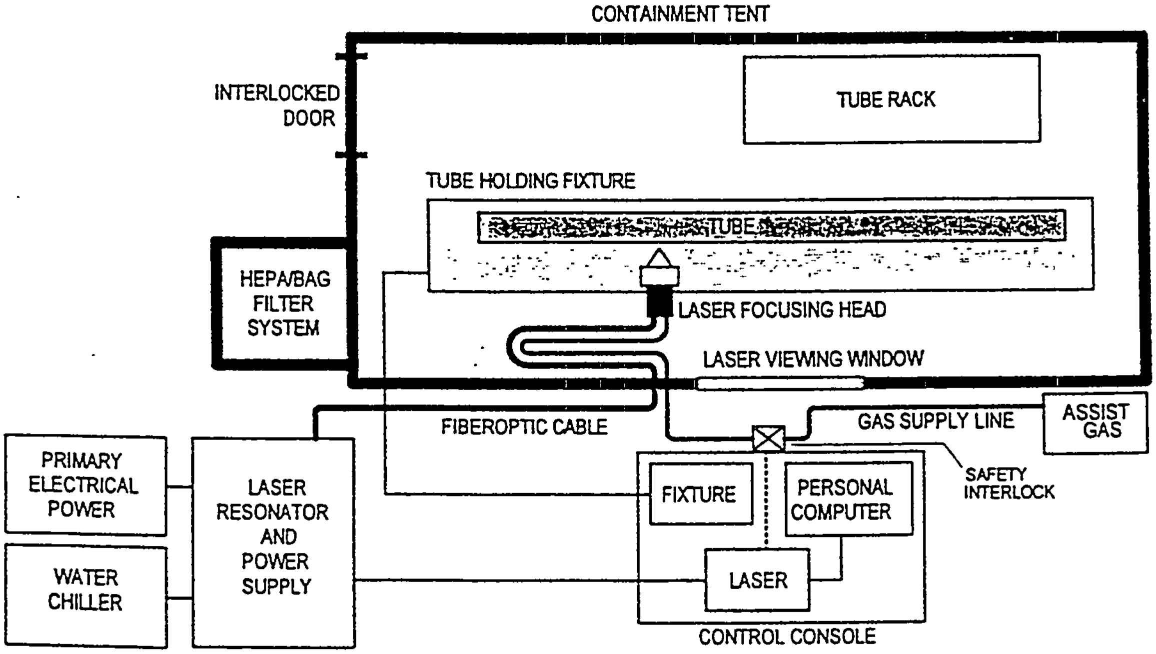

Figure 1. Schematic Diagram of the Laser Cutting Configuration for the ETEC Tests.

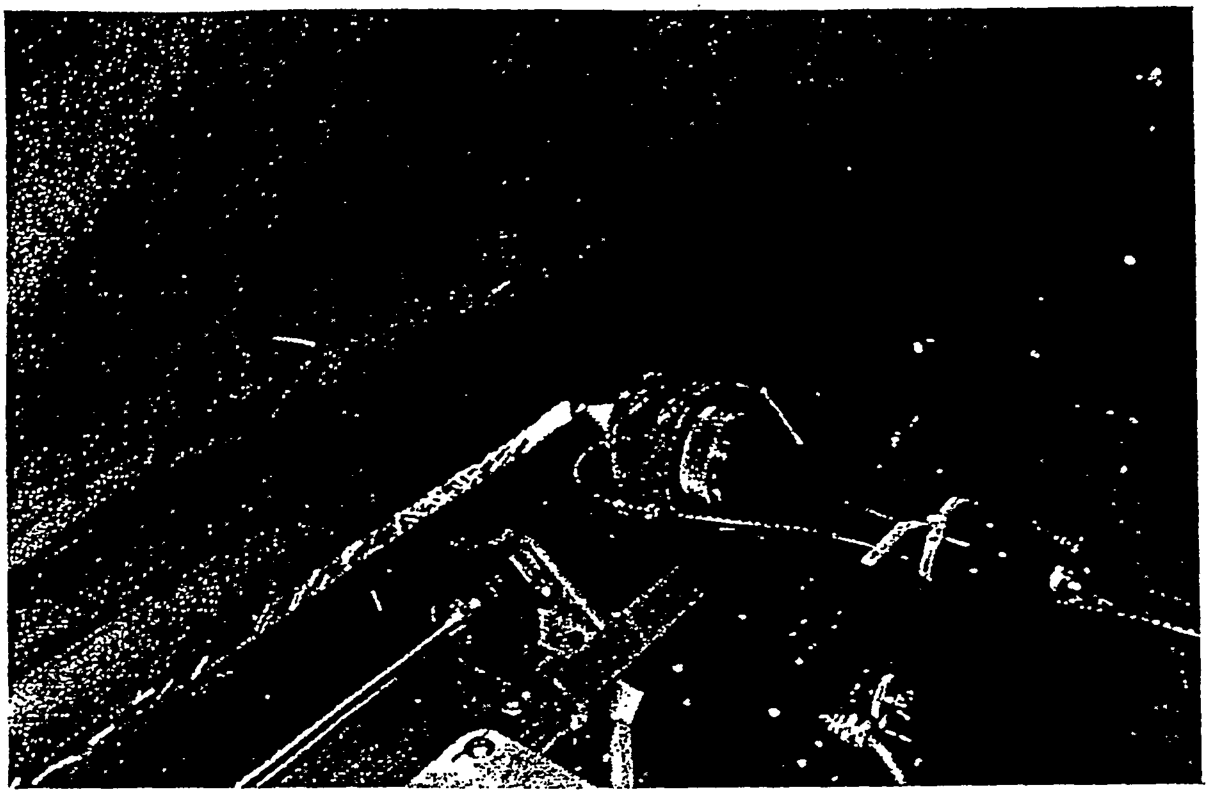

Figure 2. ETEC Laser Size Reduction of Radiologically Contaminated Storage Tubes Using Fiberoptic Beam Delivery to a Remote Cutting Head.

omitting set-up and hardware costs, are summarized in Table I. Significant cost savings were realized because of the reduced secondary waste and maintenance requirements. Maintenance requirements were still significant, however, because substantial airborne debris was generated that required frequent filter change-outs. Additional cost estimates were made for the size-reduction of significantly larger quantities of contaminated tubes. Those estimates included the capital equipment costs of the cutting hardware (significantly higher for the laser system), and assumed replacement of the filter system with self-cleaning filters, but otherwise adopted a manual procedure that does not take advantage of the added flexibility of a robotically controlled laser system. The results indicated that plasma-arc cutting is less expensive for smaller quantities of tubing (because of capital equipment costs), while laser cutting is less expensive for large quantities. The break-even in those comparisons was at about 230,000 tubes.

Table I
Summary Cost Comparison Between Remote Laser and Plasma-Arc
Size-Reduction of the ETEC Storage Tubes

Cost Element	Labor & Materials Cost ($)	
	Laser	Plasma-Arc
Tube Size-Reduction	5,600	5,600
Cutting Head Maintenance	5	423
Filter Maintenance	5,315	11,354
Transportation & Disposal	8,000	10,990
Totals:	*18,920*	*28,367*

The conclusion from the ETEC demonstration was that significant reductions in secondary waste generation, personnel exposure, and cost can be realized by the implementation of laser cutting technologies. The reduction in personnel exposure, not demonstrated in this task because of the manual tube handling, arises from the reduced routine maintenance requirements for the laser system and the placement of the key laser system components in a nonhazardous environment.

IV. MSC DEPLOYMENT DEMONSTRATION

The successful ETEC results provided the basis for technology transfer to commercial projects, and the Manufacturing Sciences Corporation performed a series of follow-on deployment tests that utilized the same laser system and capitalized on lessons learned from the ETEC demonstration. The MSC deployment further automated the laser cutting system and applied it to the size-reduction of a much larger component requiring the cutting of thicker materials. This component was a 1.8-m-diameter, 2.1-m-long cylindrical converter shell with 1.3-cm-thick carbon steel walls from the Oak Ridge K-25 site.

The MSC set-up is shown in Figure 3. Here the laser system was augmented with a Lumonics auto-focus cutting head that was mounted on the working arm of a Cincinnati Milacron robot, and the converter shell was placed on a track-mounted table with an automatic turning roll and part indexer. The function of the auto-focus cutting head was to maintain a constant stand-off distance between the cutting head and the cutting surface as the cuts were made. Axial cuts were made along the converter shell by using the robotic arm to move the cutting head axially along the converter shell, and circumferential cuts were made by rotating the converter shell on the remotely controlled table. Laser cutting head motions were directed by a programmable logic controller, which provided the capability to use the robotic arm to move the cutting head over the full converter shell surface. The adopted procedure limited the cutting head motion to linear travel with the laser head pointing downward, to restrict the laser orientation for increased safety and to allow the sectioned pieces to drop directly into a waste box. This waste box was attached to a track-mounted support structure that could be retracted from the work area remotely for material disposal. Similarly, the track-mounted table supporting the converter shell could be independently moved into and out of the work area under remote control. The work area was a pre-fabricated containment building with laser-protective film covering the observation ports. It utilized the high-efficiency facility ventilation system for airborne contaminant control.

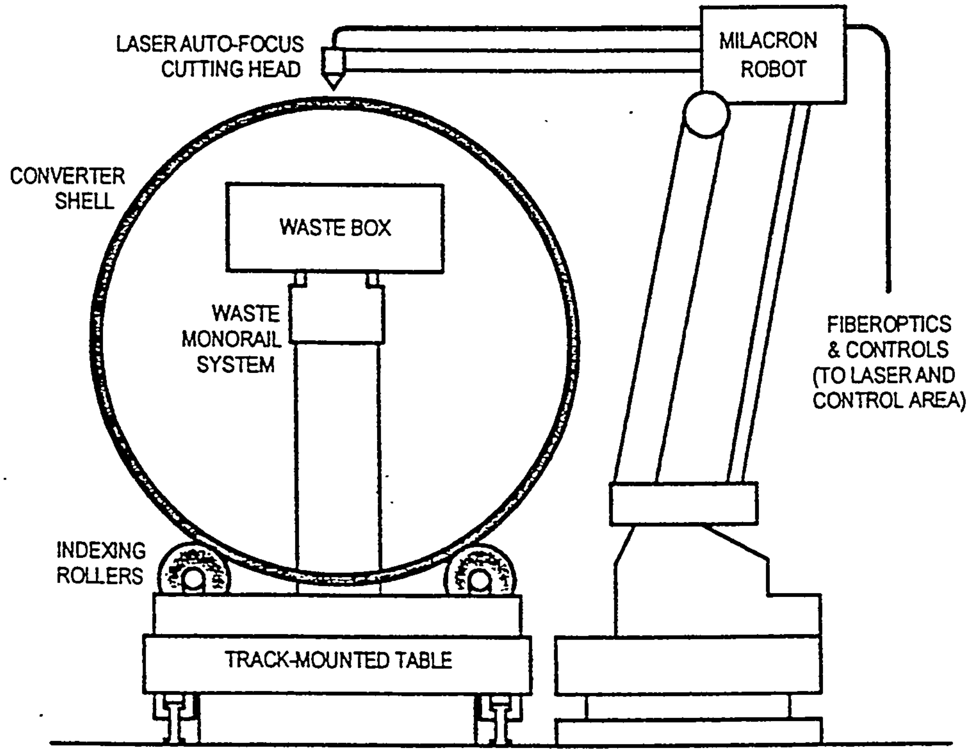

Figure 3. Schematic Diagram of the Laser Cutting Configuration for the MSC Tests.

This system was used to remove 30-cm by 30-cm sections of the converter shell by remote laser cutting. Cutting parameters included a laser power level of 1.8 kW, an oxygen assist gas, and a cutting speed of 76 cm/min. The auto-focus enhancement of the laser cutting head was found to provide improved cutting performance by controlling the cutting head stand-off distance. There were two notable differences in cutting performance between the MSC and ETEC deployment demonstrations. First, the airborne contaminant generation at MSC was much less and more easily manageable than at ETEC. This appears to be a function of the thickness of the material cut, with less airborne contaminant generation for thicker parts, and was also observed in the subsequent bench-scale tests. Second, the maintenance requirements for the focusing head were greater at MSC, with frequent cleaning necessary to remove deposited airborne debris. Focusing head cleaning was not required for any of the other cutting tests, and the difference was attributed to the location of the cutting head directly above the cutting site in conjunction with the greater amount of cutting performed. This problem was minimized by installing a protective glass plate in front of the focusing lens and directing a dry-nitrogen purge gas through the auto-focus head to blow airborne debris away from the lens assembly. Those changes reduced maintenance requirements significantly.

Additional laser cutting tests were performed at MSC to investigate the ability of the remote laser system to cut other materials. Results are summarized in Table II. Most of the laser cutting was performed using a continuous-wave (CW) laser beam. Aluminum was an exception, for which the laser beam was delivered in a square-wave pulsed mode. Aluminum cutting requires higher laser power than steel, and more instantaneous power can be delivered to the cutting site using a pulsed beam. Drawbacks are that the cutting may be uneven and the cutting speed is slower.

V. SUPPLEMENTAL BENCH-SCALE TESTS

The two deployment demonstrations identified a number of areas where additional laser cutting information would be beneficial for future applications. A supplemental series of laboratory bench-scale tests was subsequently performed at the Edison Welding Institute under contract to ETEC to investigate the following: (1) the quantity of airborne particulate that is generated during laser cutting; (2) maximum cutting speeds for cutting 1.3-cm-thick and 1.5-cm-thick steel pipe; and (3) the maximum thickness of steel plate that can be cut with a 3-kW off-the-shelf Nd:YAG laser using fiberoptic beam delivery.[5]

The airborne particulate studies used 0.2-cm-thick and 1-cm-thick flat plates, to simulate the material thicknesses in the ETEC and MSC tests, respectively, and a laser power level of 2 kW. Cutting was performed within a small enclosure using an exhaust system that drew the airborne particulate generated by the laser cuts through a HEPA filter system. The filters were weighed before and after each test to determine the weight of the collected debris. In addition, the material ejected from the bottom of the cuts and the dross removed from the bottom surface were collected and weighed, and kerf width measurements were made for the plate cuts. The data were used to determine the distribution of the secondary waste generated by the cutting process. Results are summarized in Table III.

The Table III results show that the airborne particulate was only a small fraction of the total debris generated by the laser cuts, and that the thin (0.2-cm-thick) material produced about thirteen times the airborne particulate as the thick (1-cm) material for an equivalent volume of cutting debris. This is consistent with the observations from the ETEC and MSC demonstrations. Reasons for the difference were not investigated, but may be associated with higher heat conduction in the thicker material, resulting in more melting and less vaporization.

Sixty test cuts were made to investigate maximum laser cutting speeds for steel pipe with 1.3-cm-thick and 1.5-cm-thick walls. For those tests, a pipe section was rotated beneath a fixed laser beam at various speeds, and the laser power level was stepped from 2 kW to 3 kW in 250-W increments. Some variations were also made in the focusing

Table II
Summary of the MSC Remote Laser Test Cuts Using Various Materials

Material	Thickness	Assist Gas	Laser Power	Cutting Speed
Carbon Steel	1.3 cm	10 psi oxygen	1800 W, CW	76 cm/min.
Carbon Steel	2.5 cm	10 psi oxygen	1900 W, CW	7.6 cm/min.
Stainless Steel	1.3 cm	10 psi oxygen	1800 W, CW	76 cm/min.
Aluminum	0.3 cm	10 psi oxygen	1500 W, pulsed	7.6 cm/min.

Table III
Results of the Cutting Tests to Quantify Airborne Particulate Generation

Material Thickness	Laser Power	Cutting Speed	Assist Gas	Airborne Particulate	
				Normalized Weight	Debris Fraction
0.2 cm	2 kW	152 cm/min.	20 psi O_2	0.45 g/cm^3 of cut	4.7%
1 cm	2 kW	51 cm/min.	30 psi O_2	0.034 g/cm^3 of cut	0.4%

head stand-off distance and in the oxygen assist gas pressure. Good cuts through the pipe wall were defined as those for which there was no weld-back on the interior surface of the pipe. Results of successful cuts are summarized in Table IV.

An additional fourteen test cuts were made to determine the maximum steel thickness that could be cut with a 3-kW, fiberoptic-delivered Nd:YAG beam. Those tests were performed using 1.9-cm-thick and 2.5-cm-thick steel plates, where the plates were moved remotely beneath a fixed laser beam. The tests showed that 1.9-cm-thick plate could be cut successfully at 51 cm/min. and 2.5-cm-thick plate could be cut successfully at 41 cm/min. The cut zones were cross-sectioned to examine the shapes of the cuts, and it was determined that thicker sections would be difficult to cut based on the amount of dross produced and the taper in the cuts for the 2.5-cm-thick plate. The depth profiles for the cuts showed a relatively narrow front surface width, a widening of the kerf below the surface (probably from divergence of the laser beam), and a very narrow kerf with significant slag at the back surface (Figure 4).

Table IV
Summary of Bench-Scale Laser Cutting Speed Tests

Pipe Wall Thickness	Laser Power	Successful Cutting Speed
1.3 cm	2000 W	43 cm/min.
	2250 W	43 cm/min.
	2500 W	56 cm/min.
	3000 W	89 cm/min.
1.5 cm	2000 W	48 cm/min.
	2250 W	58 cm/min.
	2500 W	58 cm/min.
	2750 W	58 cm/min.
	3000 W	76 cm/min.

VI. CONCLUSIONS

The series of tests described here demonstrated the laser cutting of both thin and thick components, various degrees of remote operation and automation, and the ability to cut 2.5-cm-thick steel at high linear cutting rates with a commercially available Nd:YAG laser. The ETEC demonstration showed the potential for cost savings using remote laser cutting, the MSC demonstration extended the application to thick materials using automated robotics, and the bench-scale tests provided further data on cutting capabilities using off-the-shelf laser systems. Although relatively high laser hardware costs make other technologies more cost effective for small-scale applications, reductions in personnel exposures by using remote laser cutting, with its minimal maintenance requirements, may override cost considerations in hazardous-environment applications. The overall conclusion from this successful series of laboratory and field-deployment tests is that current, off-the-shelf laser technology with remote fiberoptic beam delivery can be used effectively for component size-reduction in D&D applications.

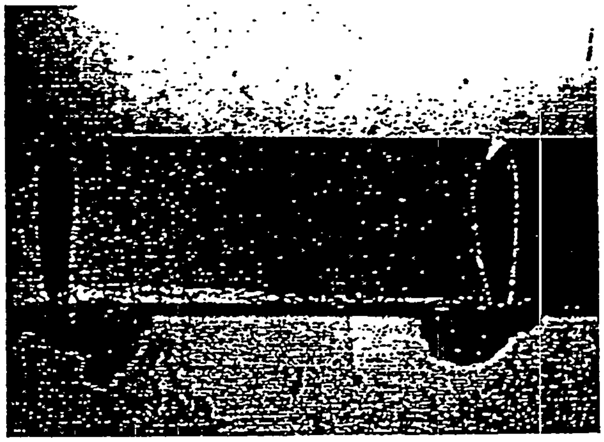

Figure 4. Photograph of Laser Cuts Made in 2.5-cm-Thick Steel with a 3-kW Nd:YAG Laser Beam, Incident from the Top.

ACKNOWLEDGMENTS

The ETEC activities were sponsored by the U.S. Department of Energy's Office of Environmental Management. The MSC activities were sponsored by the Department of Energy under the PRDA contract "Advanced Technologies for Decontamination and Conversion of Scrap Metals." The authors wish to thank T. Kugler and C. Ankerson of Lumonics, Ltd. for the loan of the Nd:YAG laser system for the demonstration activities. They also acknowledge the programmatic and funding support from J. M. Hyde (DOE-HQ), K. Abbott (DOE-OAK), J. Malholtra (FETC-Morgantown), and M. E. Lee (ETEC), and the technical support of M. A. Spenard, P. H. Waite, R. A. Marshall, R. I. Brownell, D. W. Hickman, R. G. Palmer, Jr., and K. Darcy (ETEC).

REFERENCES

1. *Technology Logic Diagram*, Oak Ridge National Laboratory Report ORNL/M-2751 (1993).
2. G. Subbaraman, L. B. Spiegel, J. Vetrovec, and R. N. Hindy, "Fiber Delivered Lasers for Dismantlement Applications," in *Decommissioning, Decontamination and Reutilization Worldwide Experience - D&D, What Does It Mean*, M. G. White, Ed., American Nuclear Society, Washington, DC, 10-14 November 1996, pp. 126-131.
3. L. Weeter, "Remote Laser Cutting Demonstration," Edison Welding Institute Final Report, Project 40578CSP, February 1997 (unpublished).
4. S. N. Shah, R. B. Hardy, and D. W. Kneff, "Remote Laser Processing of Radioactive Components at the Energy Technology Engineering Center (ETEC)," Proceedings from WM'98 Conference, Tucson, AZ, 1-4 March 1998 (to be published).
5. L. Weeter, "Thick Section Laser Cutting by Nd:YAG Lasers," Edison Welding Institute Final Report, Project 41931CSP, February 1998 (unpublished).

Appendix 3.3

MSC Recycled Contaminated Metals
Concept Paper
for Mr. Vince Adams, DOE Oak Ridge Operations

January 23, 1998

Currently their exists limited production capability within the US to produce products made from Recycled Contaminated Metals (RCM) in a cost competitive manner. Even more important, there is no obvious market for RCM that currently is competitive with existing candidate products. To be successful a product will have to be developed that will utilize the largest amount of metal possible using the most cost effective production method. In this way, reduction of DOE's liabilities will be maximized and RCM products will become competitive. Therefore, a product that substitutes for rather than mimics existing products would be the logical choice.

Fundamentally a product for recycled metals should include some or all of the following traits;

contain a large volume of RCM

not designed for efficient use of material

minimize processing steps

and compete as a substitute for, not as a replica of non-contaminated products.

It should be noted that product candidates requiring sophisticated machining and testing, with low material utilization, have much less chance of ever being successfully deployed.

Ideas for Recycled Contaminated Metals:

1. Pursue building an industrial base to manufacture biological shield plugs for spent nuclear fuel canisters. This would utilize large volumes of depleted uranium and the stainless steel cladding could be RCM as well. This has received significant attention from the DOE and it may be time now to implement the concept within the industry.

2. Look at additional options for the repository at Yucca Mountain (longer term). Potential candidates are rail ties and rails for the tunnel road system. Other options include the use of RCM in rock bolts and other structural members

used to stabilize the tunnels during mining and operation. This would take some development work to determine if the RCM could result in a product that could meet QA certification requirements imposed on Yucca Mountain by the NRC. Other considerations include using RCM as reinforcement rods for concrete in the repository and even using RCM as part of the aggregate in the concrete itself.

Both of the above options would require buy in by OCRWM in HQ and at the Yucca Mountain project.

3. A undetermined amount of material (carbon steel) could be taken from the K-25 site and cast into shield blocks for use in the Spallation Neutron Source. The cost would include the melting and casting into molds and a minimal amount of machining. To minimize cost you would want to cast at a continuous rate over a long period. Starting and stopping a casting operation adds considerable cost.

4. Lead can be recycled into liners at a reasonable cost and represents a significant liability reduction for the current owner. These products already appear to be competitive in today's market. Oak Ridge may want to be the center for the recovery and reuse of lead for DOE (i.e. require all of DOE to purchase or recycle their lead through a central location such as ORO).

5. Consider alternate designs for residue containers (i.e. maximum material, minimum processing).

6. Counter weights for cranes, fork lifts, and oil rigs. This would process a lot of metal however, it would be challenging to sell the concept to industry.

7. Use RCM as aggregate in concrete.

8. Use RCM for artificial reefs and break waters. This option would require the confidence of the public.

9. Develop specialty alloys that would not be in direct competition with a mass production vendor.

Currently with no clear market for RCM, the challenge is to find a low tech option that utilizes large quantities of metal. We would like to support the DOE in finding a successful end use of RCM. OCRWM has the potential to consume large quantities of metal within the repository given the right applications. Our success will only come from thinking out side the norm and doing this before design/selection is finalized.

#2

Appendix 3.4

Measurements reported by Thermo NUtech for Batch 2			
Sample Location	Results (pCi/L)*	Error (pCi/L)	Minimum detectable activity (pCi/L)
6	560.66 (28.03 pCi/g) [1.037 Bq/g]	31.26 (1.56 pCi/g) [0.058 Bq/g]	29.57 (1.48 pCi/g) [0.055 Bq/g]
6	465.09 (23.25 pCi/g) [0.860 Bq/g]	30.26 (1.51 pCi/g) [0.056 Bq/g]	30.99 (1.55 pCi/g) [0.057 Bq/g]
6	430.03 (21.50 pCi/g) [0.796 Bq/g]	19.63 (0.98 pCi/g) [0.036 Bq/g]	16.54 (0.83 pCi/g) [0.031 Bq/g]
6	120.70 (6.04 pCi/g) [0.223 Bq/g]	12.94 (0.65 pCi/g) [0.024 Bq/g]	16.15 (0.81 pCi/g) [0.030 Bq/g]

*The reported activity levels are for 20.0 g of sample in a 1.00-L solution.

Results from application of statistical tests to the measurements reported by Thermo NUtech for Batch 2	
Mean	19.705 pCi/g (0.730 Bq/g)
Standard deviation	9.519 pCi /g (0.355 Bq/g)
Standard error of the mean	4.759 pCi /g (0.177 Bq/g)
Probability that data is normally distributed	0.148

With a probability of 0.148, the data fails the Kolmogorov-Smirnov test for normality. The small probability suggests that there may be a systematic, rather than random, error in the measurements. Visual inspection of the Batch 2 data in Figure 1 reveals three measurement around 27 pCi (1 Bq/g) [the mean of the three measurements is 24.3 pCi/g (0.898 Bq/g)] and one much smaller value. The most likely systematic error that would produce the observed measurements would be the loss of ^{99}Tc during sample preparation. The validity of the measurements is also called into question by the change in minimum detectable activity: for two measurements the mean is 1.5 pCi/g (0.056 Bq/g) and for the other two the mean is 0.82 pCi/g (0.030 Bq/g). This suggests a substantial change in experimental conditions.

Set 1 measurements of beta particle emissions from cathode #3 with an alpha/beta scintillation probe		
Sample location	Beta particles count rate[a] (counts/min)	Alpha particles count rate (counts/min)
6	387	2
6	420	1
6	423	1
6	443	1
6	420	1
1A[b]	427	2
1A	447	1
1A	451	0
1A	443	3
1A	457	1
1B[c]	418	6
1B	427	5
1B	463	2
1B	486	3
1B	462	2
Background	408	3
Background	417	2
Background	472	3
Background	484	2
Background	385	1
Background	397	1

[a] Each count of the cathode or background was for one minute.

[b] Location 1A is on the same side of the cathode as location 6.

[c] Location 1B is on the opposite side of the cathode from locations 1A and 6.

Statistical analysis of Set 1 measurements of cathode #3 with an alpha/beta scintillation probe			
Sample location	Mean (counts/min)	Standard deviation (counts/min)	Standard error of the mean (counts/min)
6	418.6	20.11	8.99
1A[a]	445.0	11.31	5.06
1B[b]	451.2	28.08	12.56
Background	427.2	40.99	16.73

[a] Location 1A is on the same side of the cathode as location 6, the front.

[b] Location 1B is on the opposite side of the cathode from locations 1A and 6, the back.

The mean of the measurements taken at location 6 is smaller than the mean of the background measurements. Meyer , Meir and Zünd, and NCRP Report No. 58 state that such data should be used if possible rather than discarded. An applicable test to determine the potential usefulness of the data is an analysis of variance. If the measurements pass this test, they can be combined to produce an average count rate.

One-way analysis of variance of measurements from locations 6, 1A, and 1B

Source of variation	Degrees of freedom	Sum of squares	Mean squares	F statistic	Probability
Between groups	2	2996.9	1498.5	3.4	0.067
Residual	12	5284.0	440.8		
Total	14	8280.9			

Because the probability that the measurements are drawn from the same populations is greater than 0.05, the measurements pass the test. The measurements all arose from a single population. Therefore, they may be combined to obtain a single measure of the mean.

Statistical analysis for combined measurements from locations 6, 1A, and 1B		
Mean (counts/min)	Standard deviation (counts/min)	Standard error of the mean (counts/min)
438.3	24.32	6.28

The average surface activity of cathode #3 may be calculated from the Set 1 measurements by subtracting the mean combined count rate from the mean background count rate and converting to Becquerels.

$$A = \left((438.3 \pm 24.3) - (427.2 \pm 41.0) \frac{\text{counts}}{\text{minute}} \right) \left(\frac{\text{disintegrations}}{0.094 \text{ counts}} \right) \left(\frac{\text{min}}{60 \text{ sec}} \right) \left(\frac{\text{Bq}}{\frac{\text{disintegrations}}{\text{sec}}} \right)$$

$$A = 1.97 \pm 0.30 \text{ Bq} \cong 2.0 \pm 0.3 \text{ Bq}$$

The average bulk activity of cathode #3 is found by dividing the measured surface activity by the mass of the emitting layer, 2.23g.

$$A_{bulk,cathode\#3} = \frac{(1.97 \pm 0.30\,\text{Bq})}{2.23\,\text{g}} = 0.883 \pm 0.13 \frac{\text{Bq}}{\text{g}} \cong 0.88 \pm 0.13 \frac{\text{Bq}}{\text{g}}$$

Set 2 measurements of beta particle emissions from cathode #3 with an alpha/beta scintillation probe

Location on the cathode								
5A[a]	1A	2A	3A	4A	1B[b]	2B	3B	4B
Beta particles count rate[c] (counts/min)								
156	179	179	203	168	189	168	158	169
197	189	213	190	191	191	165	169	199
166	176	209	186	174	192	182	181	146
173	214	186	195	167	182	185	161	172
186	195	158	198	204	185	183	140	170

[a] Locations designated A are on the front of cathode #3.
[b] Locations designated B are on the back of cathode #3.
[c] All counts were one minute long.

Set 2 background measurements with an alpha/beta scintillation probe

Beta particle count rate[a] (counts/min)				
183	209	169	168	153
157	170	147	168	176

[a] All counts were one minute long.

Statistical analysis of Set 2 measurements of cathode #3 with an alpha/beta scintillation probe

Sample location	Mean (counts/min)	Standard deviation (counts/min)	Standard error of the mean (counts/min)
5A	175.6	16.20	7.24
1A	190.6	15.14	6.77
2A	189.0	22.62	10.11
3A	194.4	6.66	2.98
4A	180.8	16.15	7.22
1B	187.8	4.21	1.88
2B	176.6	9.34	4.18
3B	161.8	15.09	6.75
4B	171.2	18.81	8.41
Background	170.0	17.39	5.50

The mean of the measurements taken at location 3B is smaller than the mean of the background measurements and the mean of measurements taken at locations 2B, 4B, and 5A are only slightly greater than the background measurement average. As stated by Meyer, Meir and Zünd, and NCRP Report No. 58, such data should be used if possible rather than discarded. An analysis of variance was used to determine if the measurements from the nine locations are sufficiently similar to be averaged together.

One-way analysis of variance of measurements from locations 1A, 2A, 3A, 4A, 5A, 1B, 2B, 3B, and 4B

Source of variation	Degrees of freedom	Sum of squares	Mean squares	F statistic	Probability
Between groups	8	4475.2	559.4	2.524	0.027
Residual	36	7978.0	221.611		
Total	44	12453.2			

The probability of similarity among the measurements is 0.027. This is a statistically significant difference, the differences in the mean values among the measurements are greater than would be expected by chance. Therefore, the measurements from the nine locations can not be averaged together to obtain an average surface activity.

The measurements were tested to determine if exclusion of a single group of measurements (Group 3B, the measurements from location 3B, with a mean surface activity of 161.8 counts/min) would allow the measurements to be averaged. A one-way analysis of variance test was conducted on the eight groups of measurements remaining after Group 3B was excluded.

One-way analysis of variance of measurements from locations 1A, 2A, 3A, 4A, 5A, 1B, 2B, and 4B

Source of variation	Degrees of freedom	Sum of squares	Mean squares	F statistic	Probability
Between groups	7	2430.3	347.186	1.572	0.18
Residual	32	7067.2	220.85		
Total	39	9497.5			

Without the Group 3B data, the probability that the data are sufficiently similar to be combined is 0.180. This probability is much greater than the cutoff value of 0.05. Hence, the measurements may be combined to compute an average surface activity.

The following analysis of beta particle emissions measurements excludes data collected from location 3B (Group 3B). The Group 3B data are inconsistent with the normally distributed measurements of beta emissions from other locations on cathode #3. The inconsistency may arise from two possible causes: (1) the number of beta particles emitted from location 3B is different from the emissions elsewhere on cathode #3 or (2) a machine or operator error occurred. The first possible cause is inconsistent with the assumption that the ^{99}Tc is uniformly distributed throughout the cathode. Without confirmatory measurements, such as liquid scintillation counting, from location 3B, potential cause (1) will be discarded. It is assumed that a machine or operator error occurred and that no significant information will be lost by excluding the data collected from location 3B from further analysis.

Statistical analysis for combined measurements from cathode #3 locations 1A, 2A, 3A, 4A, 5A, 1B, 2B, and 4B		
Mean (counts/min)	Standard deviation (counts/min)	Standard error of the mean (counts/min)
183.25	15.61	2.47

The average surface activity of cathode #3 may be calculated from the Set 2 measurements by subtracting the mean combined count rate from the mean background count rate and converting to Becquerels.

$$A = \left((183.25 \pm 15.61) - (170.0 \pm 17.4) \frac{\text{counts}}{\text{minute}} \right) \left(\frac{\text{disintegrations}}{0.094\ \text{counts}} \right) \left(\frac{\text{min}}{60\ \text{sec}} \right) \left(\frac{\text{Bq}}{\frac{\text{disintegrations}}{\text{sec}}} \right)$$

$$A = 2.35 \pm 0.44\ \text{Bq} \cong 2.4 \pm 0.4\ \text{Bq}$$

The average bulk activity of cathode #3 resulting from Set 2 measurements is computed by dividing the average surface activity by the mass of the emitting layer, 2.23g.

$$A_{bulk,cathode\#3} = \frac{(2.35 \pm 0.44\ \text{Bq})}{2.23\ \text{g}} = 1.054 \pm 0.197 \frac{\text{Bq}}{\text{g}} \cong 1.05 \pm 0.20 \frac{\text{Bq}}{\text{g}}$$

Set 3 measurements of particle emissions from cathode #3 with an alpha/beta scintillation probe		
Sample location	Beta particles count rate[a] (counts/min)	Alpha particles count rate (counts/min)
2A[b]	236	2
2A	263	0
2A	264	1
2A	230	0
2A	235	2
2A	268	0
2A	235	1
2A	248	0
2A	237	1
2A	270	0
2B[c]	250	2
2B	222	1
2B	229	1
2B	238	0
2B	225	0
2B	251	3
2B	234	3
2B	217	2
2B	245	1
2B	221	1
Background	281	3
Background	289	0
Background	298	3
Background	299	2
Background	286	2
Background	244	2

[a] All counts were one minute long.

[b] Location 2A is on the front of cathode #3.

[c] Location 2B is on the back of cathode #3.

Statistical analysis of Set 3 measurements of beta particle emissions from cathode #3 with an alpha/beta scintillation probe

Sample location	Mean (counts/min)	Standard deviation (counts/min)	Standard error of the mean (counts/min)
2A[a]	248.6	15.95	5.04
2B[b]	233.2	12.40	3.92
Background	282.8	20.25	8.27

[a] Location 2A is on the front of cathode #3.
[b] Location 2B is on the back of cathode #3.

The means of the Set 3 measurements of beta particle emissions from cathode #3 are both more than two standard deviations below the mean background. Although the data appear to be internally consistent, they are unusable. The alpha/beta scintillation probe and scaler appear to have been malfunctioning.

Cathode #3 was moved to a new location, the receiving area of the manufacturing facility, prior to collecting the Set 4 measurements. The move was an attempt to find a low-background location. According to Tsoulfanidis and NCRP Report No. 58, an increase in the integration time will increase the total number of counts received and lower the minimum detectable activity. Hence, in an attempt to lower the minimum detectable activity, the counting or integration time for the Set 4 measurements was set to two minutes. This is a factor of two greater than the integration time used while measuring Sets 1-3.

Set 4 measurements of particle emissions from cathode #3 with an alpha/beta scintillation probe

Sample location	Beta particles Total counts [a]	Alpha particles Total counts [a]
2B[b]	509	1
2B	498	6
2B	538	2
2B	539	1
2B	504	5
2B	533	2
2B	513	1
2B	530	1
2B	524	2
2B	527	4
2B	547	0
2B	534	2
2B	534	1
2B	541	1
2B	534	0
2B	544	1
2B	485	2
2B	567	4
2B	560	2
2B	515	2
Background	632	1
Background	601	2
Background	636	1
Background	627	3
Background	601	1
Background	627	2
Background	616	1
Background	668	0
Background	623	3
Background	552	2

[a] Each count was for two minutes.

[b] Location 2B is on the back of cathode #3.

Statistical analysis of Set 4 measurements of beta particle emissions from cathode #3 with an alpha/beta scintillation probe

Sample location	Mean (counts/2 min)	Standard deviation (counts/2 min)	Standard error of the mean (counts/2 min)
2B[a]	521.5	14.55	4.60
2B	536.1	23.10	7.30
Background	618.3	30.08	9.51

[a] Location 2B is on the back of cathode #3.

As with the Set 3 measurements, the average Set 4 beta particle emissions from cathode #3 were more than two standard deviations from the mean of the background measurements. Clearly, these data are unusable. The alpha/beta scintillation probe and scaler appear to have been malfunctioning.

Following the unsuccessful attempts to acquire useful information from sets 3 and 4, it was decided to increase the counting interval from 1-2 min to 30 min. This required modifications to the scalar for the alpha/beta scintillation counting system. After the modifications, the system efficiency was 18.7% for alpha particles and 11.0% for beta particles.

Set 5 measurements of particle emissions from cathode #3 with an alpha/beta scintillation probe

Sample Location[a]	Counting start time	Beta particles Total counts[b]	Alpha particles Total counts
Background	9:40 AM	5985	62
2B[c]	10:15 AM	6025	81
2B	10:45 AM	5948	63
2B	11:15 AM	5895	59
2B	11:45 AM	5970	54
2B	12:15 PM	5843	56
2B	12:45PM[d]	5707	57
Background	2:15 PM	8501	54
Background	2:45 PM	8400	57
Background[e]	3:20 PM	7581	41

[a] Except as noted, all measurements were made in the Manufacturing Sciences Corporation laboratory.

[b] Each count was for 30 minutes.

[c] Location 2B is on the back of cathode #3.

[d] The scintillation counter and scaler were not turned off between 1:10 and 2:15 PM. The instrument remained on from 9:40 AM until 3:50 PM.

[e] This background measurement was made in the engineering offices located in the manufacturing facility. The move from the laboratory was to determine if a lower background area was available.

Statistical analysis of Set 5 measurements of beta particle emissions from cathode #3 with an alpha/beta scintillation probe

Sample location	Mean Total counts	Standard deviation Total counts	Standard error of the mean
2B[a]	5898.0	112.54	45.95
Background	7628.7	1424.35	822.35
Background[b]	7581	N/A[c]	N/A

[a] Location 2B is on the back of cathode #3.

[b] This background measurement was made in a separate building.

[c] N/A = not applicable

Once again the average beta particle emissions are more than two standard deviations away from the mean background. The most likely explanation is that the alpha/beta scintillation probe and scaler were malfunctioning.

Measurements of particle emissions from barrier nickel and cathode #3 with an alpha/beta scintillation probe

Sample location	Beta particles count rate[a] (counts/min)
Barrier nickel	2032
Barrier nickel	1990
Barrier nickel	1965
Barrier nickel	1979
Barrier nickel	1953
Barrier nickel	2016
Barrier nickel	1987
Barrier nickel	1950
Barrier nickel	1896
Barrier nickel	1935
Cathode #3 (removed piece)	294
Cathode #3 (removed piece)	301
Cathode #3 (removed piece)	333
Cathode #3 (removed piece)	331
Cathode #3 (removed piece)	317
Cathode #3 (removed piece)	323
Cathode #3 (removed piece)	330
Cathode #3 (removed piece)	317
Cathode #3 (removed piece)	328
Cathode #3 (removed piece)	355
Background	315
Background	283
Background	316
Background	316
Background	327
Background	304
Background	290

Background	278
Background	253
Background	305

[a] Each count was for 30 minutes.

Measurements of particle emissions from barrier nickel and cathode #3 with a shielded Geiger-Müller tube

Sample location	Beta particles Total counts[a]
Barrier nickel	34,670
Barrier nickel	34,138
Barrier nickel	34,511
Barrier nickel	34,767
Cathode #3 (removed piece)	2128
Cathode #3 (removed piece)	2147
Cathode #3 (removed piece)	2225
Cathode #3 (removed piece)	2094
Cathode #3 (location 1B)	2213
Cathode #3 (location 1B)	2013
Cathode #3 (location 1B)	2121

[a] Each count was for 60 minutes.

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FACSIMILE COVER SHEET

TO: Valerie MacNair

COMPANY: Manufacturing Sciences

LOCATION:

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TIME: 12:02 pm.

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FROM: Cheryl Conner

COMPANY: *Thermo NUtech*
Oak Ridge, Tennessee
PHONE NO: 423/481-0683
FAX NO: 423/481-0121 (Admin)
423/483-4621 (Lab)

MESSAGE:

Lab #97-02022 - TC-99 Results

Valerie MacNair
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PO#: 971555
SDG: 9702022
Matrix: Solid

Final Report of Analysis
Date of Report: 2/25/97
Page 1 of 1

Lab ID		Client ID	Sample Date	Receipt Date	Analysis Date	Batch ID	Analyte	Method	Result	Error	MDA	Units
97-02022-01	K	KNOWN	02/11/97	02/11/97	02/20/97	9702022	Technetium-99	EICHrom TCS01 Modified	538.51	11.80		pCi/g
97-02022-01	S	SPIKE	02/11/97	02/11/97	02/20/97	9702022	Technetium-99	EICHrom TCS01 Modified	563.96	6.41	1.72	pCi/g
97-02022-02	B	BLANK	02/11/97	02/11/97	02/20/97	9702022	Technetium-99	EICHrom TCS01 Modified	-0.55	0.93	1.65	pCi/g
97-02022-03	D	#1	10/01/96	02/11/97	02/20/97	9702022	Technetium-99	EICHrom TCS01 Modified	3.20	1.22	1.92	pCi/g
97-02022-04		#1	10/01/96	02/11/97	02/20/97	9702022	Technetium-99	EICHrom TCS01 Modified	2.14	1.20	1.95	pCi/g
97-02022-05		#2	10/01/96	02/11/97	02/20/97	9702022	Technetium-99	EICHrom TCS01 Modified	26.50	1.67	1.69	pCi/g
97-02022-06		#3	10/01/96	02/11/97	02/20/97	9702022	Technetium-99	EICHrom TCS01 Modified	4.90	1.10	1.82	pCi/g
97-02022-07		#4	10/01/96	02/11/97	02/20/97	9702022	Technetium-99	EICHrom TCS01 Modified	2.23	1.05	1.89	pCi/g

Approved by: C. E. Shoup For 2/25/97
M.R. McDougall, Laboratory Manager

K=Known,S=Spike,B=Blank,D=Duplicate,MS=Matrix Spike

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PO#: 972443
SDG: 9705089
Matrix: Liquid

Final Report of Analysis
Date of Report: 5/28/97
Page 1 of 1

Lab ID		Client ID	Sample Date	Receipt Date	Analysis Date	Batch ID	Analyte	Method	Result	Error	MDA	Units
97-05089-01	K	KNOWN	05/20/97	05/20/97	05/23/97	9705089	Technetium-99	EiCHrom TCW01 Modified	546.26	12.00		PCI/L
97-05089-01	S	SPIKE	05/20/97	05/20/97	05/23/97	9705089	Technetium-99	EiCHrom TCW01 Modified	575.20	6.62	1.80	PCI/L
97-05089-02	B	BLANK	05/20/97	05/20/97	05/23/97	9705089	Technetium-99	EiCHrom TCW01 Modified	-0.42	0.98	1.72	PCI/L
97-05089-03	D	#1		05/20/97	05/23/97	9705089	Technetium-99	EiCHrom TCW01 Modified	560.66	31.26	29.57	PCI/L
97-05089-04		#1		05/20/97	05/23/97	9705089	Technetium-99	EiCHrom TCW01 Modified	465.09	30.26	30.99	PCI/L
97-05089-05		#2		05/20/97	05/23/97	9705089	Technetium-99	EiCHrom TCW01 Modified	430.03	19.63	16.54	PCI/L
97-05089-06		#3		05/20/97	05/23/97	9705089	Technetium-99	EiCHrom TCW01 Modified	120.70	12.94	16.15	PCI/L

Approved by: 5/28/97
M.R. McDougall, Laboratory Manager

K=Known,S=Spike,B=Blank,D=Duplicate,MS=Matrix Spike

113C Union Valley Road
Oak Ridge, Tn 37830-8244

Analytical Services Organization

November 17, 1997

Manufacturing Science Corp.

Dear Libby Terry:

Enclosed please find a Level 4 "like" package for the analysis performed on A972120260. All the information that you need to validate the numbers is included. If you have any questions, please feel free to contact me.

Sincerely,

Darin K. Mann, Ph.D.
Radiochemistry Applied R&D

CASE NARRATIVE

Lab Project ID: KMSMSC
Sample Numbers: A972120260 (QA File # PE-1141)

II. Method Summary

The methods requested were defined in the ASO Project Summary, which was given to the lab.

Parameter	Separation Procedure ID	Counting Procedure ID
Technetium-99	ACD-160074.R2	ACD-160063.R1

A duplicate and spike were analyzed with the sample.

The samples received were in the form of Nickel drill turnings. The turnings were reduced in size with metal cutters to facilitate digestion. An appropriate amount of sample was placed in a reaction vessel and digestion in a CEM STAR Open Vessel Microwave system using both Nitric and Hydrogen Peroxide to solubilize the sample. The digestion temperature was controlled to 80 +/- 0.1 degrees Celsius. The digested sample was diluted to 1M HNO3 and placed in a Gilson ASPEC XL Automated Solid Phase Extraction (SPE) Unit. An audit log of the run is attached. The sample is extracted from the column and counted by Liquid Scintillation on a Packard Tri-Carb 2550 LSC unit. Data reduction is performed by a VMS spreadsheet.

III. Quality Control Summary

A. Holding Times

All holding times were met, where applicable.

B. Calibration

1. Initial Calibration

Guidelines for initial instrument calibration are in each counting procedure, which are listed under the Method Summary. Initial Calibration/Verification is conducted using NIST traceable standards. The values must be within 10% of the known value to be acceptable.

2. Continuing Calibration

Quality control standards are counted on each instrument or detector daily and must fall within established limits before samples are counted. Daily instrument checks are also discussed in the counting procedures listed in the Method Summary.

3. Tuning

Not Applicable

C. Quality Control Samples

1. Blanks

All blanks were acceptable.

2. Laboratory Control Samples

All laboratory control samples recovered within specified parameters.

3. **Duplicates**
   The duplicate was within acceptable limits, being –8.17%

4. **Matrix Spikes**
   The spike was within acceptable limits, with a recovery of 97.96%

5. **Post-Digestion Spikes (Surrogates/Tracers)**
   Not applicable.

6. **Matrix Interferences**
   None experienced.

7. **Standards**
   All standards are traceable to a certified agency, and certificates of analysis are on file in the laboratory. Alternate sources are used if protocol dictates.

Date Printed: Friday, August 01, 1997

ASO Sample Worksheet

Laboratory Site: K-25 Radiochemistry

Deadline: 8/24/97 11:59:59 PM

Due In Nuclear Measurements: Immediately

Sample ID A972120260

Project:	MSC
Subproject:	ERD94YA212
CUSTOMER:	FM FAULCON
Charge Number:	SN949400
Project Manage	FM FAULCON (007949)
Logged In By:	A MCGEE
Batched By:	A. LIKENS (034896)
Date Sampled:	10/31/96
Sampled From:	MAN. SCIENCES CORP.
Date Received:	7/28/97 11:45:47 AM
Matrix	SOLID
Material Descr.:	NICKEL SHAVINGS
Comments:	RETURN UN-USED SAMPLE TO MSC.
Special Handling:	
Sample Activity Screen Results:	

QC-File Number: PE-1141

Test Title	Lab Test ID	Test Components	Units
Tc-99 Act. using LSC by 160063	TC99-WP	Tc-99	pCi/g

A-File Number: PE-1141

QA Logbook/Page: 11719 / p.14

RADIOCHEMISTRY LAB

SAMPLE BATCH LOG

Project Number: KMSMSC

Report Deadline: 8/24/97 11:59:59 PM

☑ Requires Data Pkg.

SDG Case #: MSC

Batch Type/Customer: Faulcon (MSC)

Project Manager: FM FAULCON (007949)

Sample Matrix: SOLID

☐ Homogenization Required Method: ☐ Sonification ☐ High-Speed Mixing ☐ Shaking ☐ Other: ____

☑ Sample Prep. Method: ☐ HPD-OPEN ☐ MAD-CLOSE ☑ MAD-OPEN ☐ MAD-ASH ☐ TAF-ASH ☐ Other: ____

Analysis	QC Batch	Reprep (X)	Entry Date	Peer Review	Analysis	QC Batch	Reprep (X)	Entry Date	Peer Review
1 TC99-WP	QC97213044		10/27/97 SPW	10/27/97 AHL					

pecified Location of Samples: 215-3

QA Batch contains 1 samples.

A972120260			

dditional Comments: MSC Ni sample

pecial Handling: NICKEL SHAVINGS

10/27/97
JK

```
                       TC-99/TEVA/UG (STD #R1-128-W) Internal Control Chart
                            Program - 160600          Batch - 4

                                           Points
                                Number    Omitting                                           Coefficient of
                                Points    Outliers        XBAR           Std Dev              Variation

Current                           20         19          972.228         138.205                14.22%

Units: dpm/ml              Lower = 783.70              Upper = 1073.70
                                          Std = 928.70
Pt     Date      Badge QA File Number    Result--------------------------------------------------
71 24-OCT-1997  34120 PE-1141             989.64|        |              |     *        |         |
70 23-OCT-1997  33626 WP-5202/5222       1050.75|        |              |            * |         |
69 23-OCT-1997  33626 WP-5222            1507.93|        |              |              |         *
68 22-OCT-1997  33626 4                   963.94|        |              |   *          |         |
67 22-OCT-1997  34120 WP-5224             972.93|        |              |    *         |         |
66 19-OCT-1997  34120 ENV-2006            998.80|        |              |      *       |         |
65 14-OCT-1997  33626 WP-5217             995.27|        |              |      *       |         |
64 14-OCT-1997  34120 WP-5210             999.26|        |              |      *       |         |
63  9-OCT-1997  34896 WP-5208             794.00|        |*             |              |         |
62  8-OCT-1997  34120 ENV-2005            933.90|        |              |*             |         |
61  8-OCT-1997  34120 WP-5207             919.58|        |             *|              |         |
60  8-OCT-1997  34120 WP-5203             869.65|        |        *     |              |         |
59  7-OCT-1997  33626 WP-5213             915.69|        |             *|              |         |
58  6-OCT-1997  34120 ENV-2002            939.64|        |              |*             |         |
57  5-OCT-1997  33626 WP-5213             963.75|        |              |   *          |         |
56  5-OCT-1997  33626 WP-5213             907.91|        |            * |              |         |
55  5-OCT-1997  34120 WP-5195             922.03|        |             *|              |         |
54  3-OCT-1997  34120 WP-5196             987.05|        |              |     *        |         |
53  1-OCT-1997  33626 ENV-2003            929.14|        |              *              |         |
52 12-SEP-1997  33626 WP-5174             883.69|        |         *    |              |         |
```

```
1#66k2S
                                                              DATE COUNTED:24-OCT-1997
                                                           DATE CALCULATED:27-OCT-1997
                                                                  QA FILE-PE-1141
                                                                  ANALYST:DKM/KAL


                                   QA DATA SHEET
                                   =============


SAMPLE COUNTING TIME IN UNITS OF MINUTES:                      240.0

PACKARD 2550TR LIQ. SCINTILLATION COUNTER #s 1, 2, OR 3:          2

DESIRED ACTIVITY UNITS (1=pCi, 2=uCi, 3=dpm):                      1

DESIRED WEIGHT FRACTION UNITS (1=g, 2=filter):                     1

INTERNAL CONTROL CHART PROGRAM NUMBER:                             0
   ENTER:  0 FOR 160600          3 FOR 160601 (LL mode)
           1 FOR 162010
           2 FOR 162011


INTERNAL CONTROL CHART BATCH NUMBER:                               4



MEASURED CONTROL VALUE, CORRECTED FOR DECAY:                 989.64 DPM/ML

UPPER CONTROL LIMIT IN UNITS OF DPM/ML:                     1073.70

LOWER CONTROL LIMIT IN UNITS OF DPM/ML:                      783.70

ACTIVITY OF THE SAMPLE THAT WAS DUPLICATED:                 2.51e+01   (pCi/g)

ACTIVITY OF THE SAMPLE THAT WAS SPIKED:                     2.51e+01   (pCi/g)

D971020001   WAS DUPLICATED, PERCENT DIFFERENCE:              -8.17 %

D971020001   WAS SPIKED, PERCENT RECOVERY:                    97.96 %

KNOWN ACTIVITY OF CONTROL, DECAY CORRECTED:                 1023.56 DPM/ML
   STANDARD:# R1-128-W
           REFERENCE DATE:        01-JAN-1987




             AnaLIS report:
                                   ACTUAL    AMOUNT      SPIKE     PERCENT
                           UNITS   RESULT    SPIKED      RESULT    RECOVERY
                           =====   ======    ======      ======    ========
                            DPM       4.30    511.78     505.66       97.96

 20/20 License #4828, Program validation #:SOP160063.R4-T2LG, 09/28/94 DFH
0
10H1#66k05s08
```

1#66k2S

DATE COUNTED:24-OCT-1997
DATE CALCULATED:27-OCT-1997
QA FILE-PE-1141
ANALYST:DKM/KAL

Tc-99 (TevaSpec)

SAMPLE NUMBER	MD or MS	EFFIC. FACT. CPM/DPM	tSIE VALUE	GROSS ACTIVITY (CPM)	DILUTION FACTOR (ml)	WEIGHT FRACTION (g)	NET ACTIVITY (pCi/L)	NET ACTIVITY (pCi/g)	COUNTING CERTAINTY (+/-)	
BLANK		0.922	255.03	23.32						
CONTROL		0.921	245.89	479.06	0.500	0.000	4.47e+05	NA	2.83e+03	
DUPLICATE		0.926	262.69	26.92	0.000	0.077	NA	2.73e+01	5.73e+00	1.01 +/- 0.21 dps/g
SPIKE		0.925	260.51	490.21	0.000	0.077	NA	2.95e+03	1.85e+01	
D971020001MD		0.929	267.45	26.71	0.000	0.077	NA	2.51e+01	5.71e+00	0.93 +/- 0.21 dps/g
D971020001MS		0.929	267.45	26.71	0.000	0.077	NA	2.51e+01	5.71e+00	0.93 +/- 0.21 dps/g
D971010002A		0.921	253.15	26.06	0.000	0.076	NA	2.39e+01	5.81e+00	0.88 +/- 0.22 dps/g
D971020002B		0.925	260.53	26.10	0.000	0.076	NA	2.29e+01	5.80e+00	0.85 +/- 0.22 "
D971010002C		0.925	260.62	25.70	0.000	0.076	NA	2.03e+01	5.78e+00	0.75 +/- 0.21 "

8.80e^{-1} +/- 4.9e^{-1}

□
10H1#66k0Ss0B

LMES, inc.
Lockheed Martin Energy Systems, Inc.
Analytical Services Organization

Nuclear Measurements
K-25 Plant M.S.-7458 Oak Ridge, TN 37830
(615) 574-7384

RADIOCHEMICAL ANALYSIS

LABORATORY PREPARATION SHEET FOR LIQUID SCINTILLATION COUNTING

Prep.# *(Circle)*: 1 2 3 Count Time: 240 min.

QA File #: PE-1141

Project/Analysis: DEV / Tc

Ct.Room Rec'd: 10 / 24 / 97 by DKM

Count Date: 10 / 24 / 97 by DKM

Instr.#/S.#/Protocol#: 2 / 103999 / 5

Package Deadline: ASAP /

Prep.Date/Analyst: 10 / 24 / 97 by DKM

Control Number: 160600

Pipette ID: Daily Check *by*:

Microwave Filename: NA

Procedure Number: ACD-160074

**Scales ID#:* Sample ID:	**PREP (Circle): Leach Ash Direct HDV Prolabo* Complete Prep. Dilution (*Indicate units: g, fil, ml*)	Cassette# (PID)	Sample Position#
Blank Tc 99 in W;	40 ml 1M HNO_3		
Control (Tc 99 in W;)	40 ml 1M HNO_3 + 0.5 ml Tc-99 Std.		
Duplicate D971020 -001A	0.3084g / 40 ml x 10 ml		
Spike D971020 -001B	0.3084g / 40 ml x 10 ml + 0.5 ml Tc-99 Std.		
Sam D971020-001C	0.3084g / 40 ml x 10 ml		
Sam D971020-002A	0.3022g / 40 ml x 10 ml		
Sam D971020-002B	0.3022g / 40 ml x 10 ml		
Sam D971020-002C	0.3022g / 40 ml x 10 ml		

Comments: Samples run on Gilson System. - Counted Packard 2, Protocol #5

SUPERVISOR APPROVAL:

```
24 Oct 97  21:18
Protocol #: 5                 Tc-99/Teva UG              User : K25 Radiochemistry

Time: 240.00
Data Mode: DPM                  Nuclide: TC_TEVA_UG     Quench Set: TC_TEVA
Background Subtract: Manual

             LL       UL    LCR    2S%     BKG
Region A:    0.0 -   293     0     0.0     0.00
Region B:    6.0 -   293     0     0.0     0.00
Region C:    293 -  2000     0     0.0     0.00

Quench Indicator: tSIE/AEC
  Ext Std Terminator: Count

Packard Liquid Scintillation 2550TR-#2  SN.103999
Luminescence Correction On
Heterogeneity Monitor On
Color Quench Correction On

 P# PID  S#    TIME      CPMA     DPM1     CPMB     SIS    tSIE      EFF.  FLAG  LUM
  5  75   1  240.00     23.32    25.30    21.27  38.190  255.03  0.921590         51
  5  75   2  240.00    479.06   520.12   466.00  69.516  245.89  0.921055          3
  5  75   3  240.00     26.92    29.06    25.24  58.609  262.69  0.926301         29
  5  75   4  240.00    490.21   530.03   478.64  77.933  260.51  0.924872          2
  5  75   5  240.00     26.71    28.74    24.99  68.436  267.45  0.929270         17
  5  75   6  240.00     26.06    28.29    24.64  67.968  253.15  0.921389         17
  5  75   7  240.00     26.10    28.22    24.63  67.558  260.53  0.924882         16
  5  75   8  240.00     25.70    27.78    24.58  69.725  260.62  0.924941         16

SYSTEM NORMALIZED


C14 IPA DATA PROCESSED


C14 CHI SQUARE IPA DATA PROCESSED


H3 IPA DATA PROCESSED


H3 CHI SQUARE IPA DATA PROCESSED


BKG IPA DATA PROCESSED
```

DPM Tc

10/27/97
DKM

Analysis Tc-99
QA File # PE-1141

Date Printed 10/27/97 3:21 PM

MDA GENERAL CALCULATION

Equation Used: [(4.65 x Sqrt(Blk. Cts.)) + 2.71/Time x Eff. x Vol x Convertion]

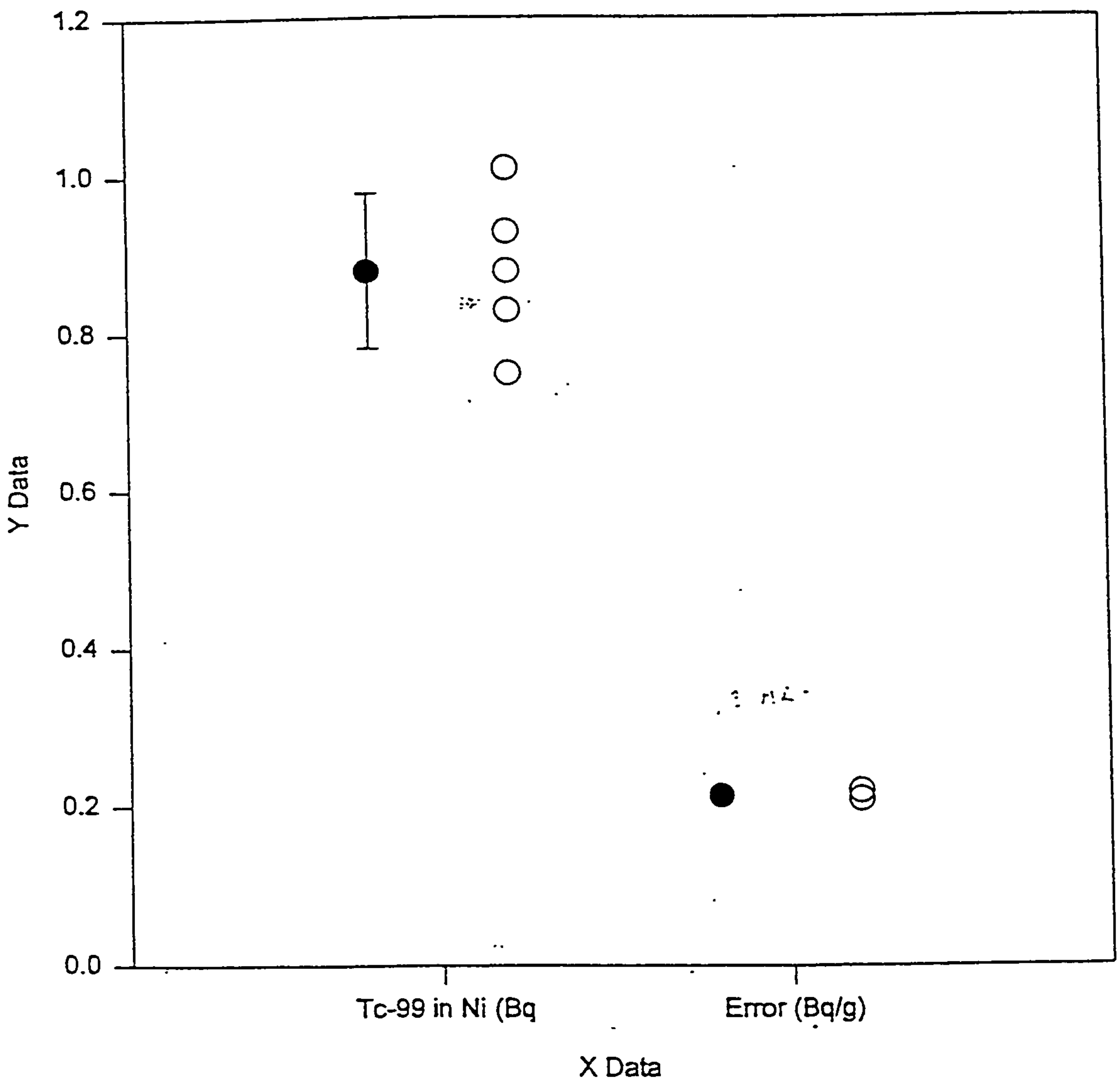
Count Time 240 Minutes

Blank Counts 5596.8

Sample I.D.	Type	Det. Eff.	Weight (g)	MDA (dpm/g)	MDA (pCi/g)	MDA (Bq/g)
D971020001AMD	A	0.926	7.71E-02	2.05E+01	9.22E+00	3.41E-01
D971020001BMS	A	0.925	7.71E-02	2.05E+01	9.23E+00	3.41E-01
D971020001C	A	0.929	7.71E-02	2.04E+01	9.19E+00	3.40E-01
D971020002A	A	0.921	7.56E-02	2.10E+01	9.46E+00	3.50E-01
D971020002B	A	0.925	7.56E-02	2.09E+01	9.42E+00	3.48E-01
D971020002C	A	0.925	7.56E-02	2.09E+01	9.42E+00	3.48E-01

```
                                                                    Monday, October 27, 1997, 16:02:55
Descriptive Statistics:

Column              Size  Missing      Mean        Std Dev        Std. Error    Range       Max
Tc-99 in Ni (Bq      5      0          0.880        0.0985         0.0440       0.260       1.010
Error (Bq/g)         5      0          0.214        0.00548        0.00245      0.01000     0.220

Column              Min    Median      25%          75%             Sum     Sum of Squares Confidence
Tc-99 in Ni (Bq     0.750  0.880       0.810        0.950           4.400       3.911       0.122
Error (Bq/g)        0.210  0.210       0.210        0.220           1.070       0.229       0.00680

Column              SkewnessKurtosisK-S Distance  P Value
Tc-99 in Ni (Bq     -5.551E-015        -0.245      0.107            0.693
Error (Bq/g)        0.609   -3.333     0.367       0.026
```

Point and Column Means



11/27/97

```
1997-10-22
14:41:12  **** Open file Logl.Log ****
          Gilson Sampler Controller Software
          721  V2.00
*********************************************
NAME : FILE1

          RINSE ASPEC NEEDLE
14:41:39  rinsing aspec needle...
          CONDITION
14:42:13  Dispensing 5.000ml in DEC
14:44:03  Dispensing 5.000ml in DEC
14:45:27  Dispensing 5.000ml in DEC
14:47:17  Dispensing 5.000ml in DEC
14:48:41  Dispensing 5.000ml in DEC
14:50:31  Dispensing 5.000ml in DEC
14:51:56  Dispensing 5.000ml in DEC
14:53:46  Dispensing 5.000ml in DEC
          CONDITION
14:55:11  Aspirating 9.500ml from SOLVENT_A
14:56:26  Dispensing 9.500ml in DEC
14:58:57  Aspirating 9.500ml from SOLVENT_A
15:00:11  Dispensing 9.500ml in DEC
15:02:44  Aspirating 9.500ml from SOLVENT_A
15:03:58  Dispensing 9.500ml in DEC
15:06:31  Aspirating 9.500ml from SOLVENT_A
15:07:55  Dispensing 9.500ml in DEC
15:10:27  Aspirating 9.500ml from SOLVENT_A
15:11:42  Dispensing 9.500ml in DEC
15:14:14  Aspirating 9.500ml from SOLVENT_A
15:15:29  Dispensing 9.500ml in DEC
15:18:01  Aspirating 9.500ml from SOLVENT_A
15:19:17  Dispensing 9.500ml in DEC
15:21:49  Aspirating 9.500ml from SOLVENT_A
15:23:03  Dispensing 9.500ml in DEC
          WAIT
15:25:37  Waiting for : 2.00 min

15:27:40  Current vial: 1 / 8
          RINSE ASPEC NEEDLE
15:27:42  rinsing aspec needle...
          LOAD
15:28:14  Aspirating 9.970ml from SAMPLE_A
15:29:28  Dispensing 9.970ml in DEC
          WAIT
15:32:13  Waiting for : 0.30 min
          LOAD
15:32:35  Aspirating 9.970ml from SAMPLE_A
15:33:49  Dispensing 9.970ml in DEC
          WAIT
15:36:35  Waiting for : 0.30 min
          LOAD
15:36:56  Aspirating 9.970ml from SAMPLE_A
15:38:10  Dispensing 9.970ml in DEC
          WAIT
15:40:55  Waiting for : 0.30 min
```

```
          LOAD
15:41:16  Aspirating 9.970ml from SAMPLE_A
15:42:30  Dispensing 9.970ml in DEC
          RINSE ASPEC NEEDLE
15:45:14  rinsing aspec needle...
          DISPENSE
15:45:46  Aspirating 8000ul from SOLVENT_A
15:46:52  Dispensing 8000ul into SAMPLE_A
          MIX
15:47:32  Mixing SAMPLE_A with 7000ul of liquid
          LOAD
15:48:33  Aspirating 8.100ml from SAMPLE_A
15:49:37  Dispensing 8.100ml in DEC
          RINSE ASPEC NEEDLE
15:51:53  rinsing aspec needle...
          DISPENSE
15:52:25  Aspirating 8000ul from SOLVENT_A
15:53:29  Dispensing 8000ul into SAMPLE_A
          MIX
15:54:07  Mixing SAMPLE_A with 7000ul of liquid
          LOAD
15:55:08  Aspirating 8.100ml from SAMPLE_A
15:56:13  Dispensing 8.100ml in DEC

15:58:30  Current vial: 2 / 8
          RINSE ASPEC NEEDLE
15:58:33  rinsing aspec needle...
          LOAD
15:59:05  Aspirating 9.970ml from SAMPLE_A
16:00:19  Dispensing 9.970ml in DEC
          WAIT
16:03:04  Waiting for : 0.30 min
          LOAD
16:03:25  Aspirating 9.970ml from SAMPLE_A
16:04:39  Dispensing 9.970ml in DEC
          WAIT
16:07:23  Waiting for : 0.30 min
          LOAD
16:07:44  Aspirating 9.970ml from SAMPLE_A
16:08:58  Dispensing 9.970ml in DEC
          WAIT
16:11:44  Waiting for : 0.30 min
          LOAD
16:12:06  Aspirating 9.970ml from SAMPLE_A
16:13:20  Dispensing 9.970ml in DEC
          RINSE ASPEC NEEDLE
16:16:05  rinsing aspec needle...
          DISPENSE
16:16:36  Aspirating 8000ul from SOLVENT_A
16:17:41  Dispensing 8000ul into SAMPLE_A
          MIX
16:18:19  Mixing SAMPLE_A with 7000ul of liquid
          LOAD
16:19:20  Aspirating 8.100ml from SAMPLE_A
16:20:23  Dispensing 8.100ml in DEC
          RINSE ASPEC NEEDLE
16:22:41  rinsing aspec needle...
```

```
          DISPENSE
16:23:13  Aspirating 8000ul from SOLVENT_A
16:24:19  Dispensing 8000ul into SAMPLE_A
          MIX
16:24:57  Mixing SAMPLE_A with 7000ul of liquid
          LOAD
16:25:58  Aspirating 8.100ml from SAMPLE_A
16:27:02  Dispensing 8.100ml in DEC

16:29:18  Current vial: 3 / 8
          RINSE ASPEC NEEDLE
16:29:20  rinsing aspec needle...
          LOAD
16:29:52  Aspirating 9.970ml from SAMPLE_A
16:31:06  Dispensing 9.970ml in DEC
          WAIT
16:33:52  Waiting for : 0.30 min
          LOAD
16:34:14  Aspirating 9.970ml from SAMPLE_A
16:35:28  Dispensing 9.970ml in DEC
          WAIT
16:38:13  Waiting for : 0.30 min
          LOAD
16:38:34  Aspirating 9.970ml from SAMPLE_A
16:39:48  Dispensing 9.970ml in DEC
          WAIT
16:42:33  Waiting for : 0.30 min
          LOAD
16:42:54  Aspirating 9.970ml from SAMPLE_A
16:44:08  Dispensing 9.970ml in DEC
          RINSE ASPEC NEEDLE
16:46:53  rinsing aspec needle...
          DISPENSE
16:47:26  Aspirating 8000ul from SOLVENT_A
16:48:31  Dispensing 8000ul into SAMPLE_A
          MIX
16:49:10  Mixing SAMPLE_A with 7000ul of liquid
          LOAD
16:50:11  Aspirating 8.100ml from SAMPLE_A
16:51:14  Dispensing 8.100ml in DEC
          RINSE ASPEC NEEDLE
16:53:31  rinsing aspec needle...
          DISPENSE
16:54:03  Aspirating 8000ul from SOLVENT_A
16:55:07  Dispensing 8000ul into SAMPLE_A
          MIX
16:55:45  Mixing SAMPLE_A with 7000ul of liquid
          LOAD
16:56:47  Aspirating 8.100ml from SAMPLE_A
16:57:51  Dispensing 8.100ml in DEC

17:00:08  Current vial: 4 / 8
          RINSE ASPEC NEEDLE
17:00:12  rinsing aspec needle...
          LOAD
17:00:44  Aspirating 9.970ml from SAMPLE_A
17:01:57  Dispensing 9.970ml in DEC
```

```
         WAIT
17:04:42 Waiting for : 0.30 min
         LOAD
17:05:04 Aspirating 9.970ml from SAMPLE_A
17:06:17 Dispensing 9.970ml in DEC
         WAIT
17:09:02 Waiting for : 0.30 min
         LOAD
17:09:24 Aspirating 9.970ml from SAMPLE_A
17:10:39 Dispensing 9.970ml in DEC
         WAIT
17:13:24 Waiting for : 0.30 min
         LOAD
17:13:46 Aspirating 9.970ml from SAMPLE_A
17:15:00 Dispensing 9.970ml in DEC
         RINSE ASPEC NEEDLE
17:17:45 rinsing aspec needle...
         DISPENSE
17:18:17 Aspirating 8000ul from SOLVENT_A
17:19:21 Dispensing 8000ul into SAMPLE_A
         MIX
17:20:00 Mixing SAMPLE_A with 7000ul of liquid
         LOAD
17:21:01 Aspirating 8.100ml from SAMPLE_A
17:22:05 Dispensing 8.100ml in DEC
         RINSE ASPEC NEEDLE
17:24:23 rinsing aspec needle...
         DISPENSE
17:24:55 Aspirating 8000ul from SOLVENT_A
17:26:01 Dispensing 8000ul into SAMPLE_A
         MIX
17:26:39 Mixing SAMPLE_A with 7000ul of liquid
         LOAD
17:27:40 Aspirating 8.100ml from SAMPLE_A
17:28:44 Dispensing 8.100ml in DEC

17:31:00 Current vial: 5 / 8
         RINSE ASPEC NEEDLE
17:31:03 rinsing aspec needle...
         LOAD
17:31:36 Aspirating 9.970ml from SAMPLE_A
17:32:50 Dispensing 9.970ml in DEC
         WAIT
17:35:36 Waiting for : 0.30 min
         LOAD
17:35:58 Aspirating 9.970ml from SAMPLE_A
17:37:11 Dispensing 9.970ml in DEC
         WAIT
17:39:56 Waiting for : 0.30 min
         LOAD
17:40:18 Aspirating 9.970ml from SAMPLE_A
17:41:32 Dispensing 9.970ml in DEC
         WAIT
17:44:17 Waiting for : 0.30 min
         LOAD
17:44:38 Aspirating 9.970ml from SAMPLE_A
17:45:52 Dispensing 9.970ml in DEC
```

```
          RINSE ASPEC NEEDLE
17:48:38  rinsing aspec needle...
          DISPENSE
17:49:11  Aspirating 8000ul from SOLVENT_A
17:50:16  Dispensing 8000ul into SAMPLE_A
          MIX
17:50:54  Mixing SAMPLE_A with 7000ul of liquid
          LOAD
17:51:55  Aspirating 8.100ml from SAMPLE_A
17:52:59  Dispensing 8.100ml in DEC
          RINSE ASPEC NEEDLE
17:55:16  rinsing aspec needle...
          DISPENSE
17:55:48  Aspirating 8000ul from SOLVENT_A
17:56:53  Dispensing 8000ul into SAMPLE_A
          MIX
17:57:33  Mixing SAMPLE_A with 7000ul of liquid
          LOAD
17:58:34  Aspirating 8.100ml from SAMPLE_A
17:59:38  Dispensing 8.100ml in DEC

18:01:55  Current vial: 6 / 8
          RINSE ASPEC NEEDLE
18:01:58  rinsing aspec needle...
          LOAD
18:02:30  Aspirating 9.970ml from SAMPLE_A
18:03:44  Dispensing 9.970ml in DEC
          WAIT
18:06:29  Waiting for : 0.30 min
          LOAD
18:06:50  Aspirating 9.970ml from SAMPLE_A
18:08:04  Dispensing 9.970ml in DEC
          WAIT
18:10:50  Waiting for : 0.30 min
          LOAD
18:11:12  Aspirating 9.970ml from SAMPLE_A
18:12:27  Dispensing 9.970ml in DEC
          WAIT
18:15:12  Waiting for : 0.30 min
          LOAD
18:15:34  Aspirating 9.970ml from SAMPLE_A
18:16:48  Dispensing 9.970ml in DEC
          RINSE ASPEC NEEDLE
18:19:33  rinsing aspec needle...
          DISPENSE
18:20:05  Aspirating 8000ul from SOLVENT_A
18:21:10  Dispensing 8000ul into SAMPLE_A
          MIX
18:21:49  Mixing SAMPLE_A with 7000ul of liquid
          LOAD
18:22:51  Aspirating 8.100ml from SAMPLE_A
18:23:55  Dispensing 8.100ml in DEC
          RINSE ASPEC NEEDLE
18:26:12  rinsing aspec needle...
          DISPENSE
18:26:45  Aspirating 8000ul from SOLVENT_A
18:27:49  Dispensing 8000ul into SAMPLE_A
```

```
          MIX
18:28:28  Mixing SAMPLE_A with 7000ul of liquid
          LOAD
18:29:29  Aspirating 8.100ml from SAMPLE_A
18:30:33  Dispensing 8.100ml in DEC

18:32:49  Current vial: 7 / 8
          RINSE ASPEC NEEDLE
18:32:53  rinsing aspec needle...
          LOAD
18:33:26  Aspirating 9.970ml from SAMPLE_A
18:34:40  Dispensing 9.970ml in DEC
          WAIT
18:37:26  Waiting for : 0.30 min
          LOAD
18:37:48  Aspirating 9.970ml from SAMPLE_A
18:39:02  Dispensing 9.970ml in DEC
          WAIT
18:41:47  Waiting for : 0.30 min
          LOAD
18:42:08  Aspirating 9.970ml from SAMPLE_A
18:43:22  Dispensing 9.970ml in DEC
          WAIT
18:46:08  Waiting for : 0.30 min
          LOAD
18:46:30  Aspirating 9.970ml from SAMPLE_A
18:47:44  Dispensing 9.970ml in DEC
          RINSE ASPEC NEEDLE
18:50:30  rinsing aspec needle...
          DISPENSE
18:51:02  Aspirating 8000ul from SOLVENT_A
18:52:07  Dispensing 8000ul into SAMPLE_A
          MIX
18:52:45  Mixing SAMPLE_A with 7000ul of liquid
          LOAD
18:53:45  Aspirating 8.100ml from SAMPLE_A
18:54:49  Dispensing 8.100ml in DEC
          RINSE ASPEC NEEDLE
18:57:05  rinsing aspec needle...
          DISPENSE
18:57:37  Aspirating 8000ul from SOLVENT_A
18:58:43  Dispensing 8000ul into SAMPLE_A
          MIX
18:59:22  Mixing SAMPLE_A with 7000ul of liquid
          LOAD
19:00:23  Aspirating 8.100ml from SAMPLE_A
19:01:27  Dispensing 8.100ml in DEC

19:03:43  Current vial: 8 / 8
          RINSE ASPEC NEEDLE
19:03:46  rinsing aspec needle...
          LOAD
19:04:18  Aspirating 9.970ml from SAMPLE_A
19:05:32  Dispensing 9.970ml in DEC
          WAIT
19:08:16  Waiting for : 0.30 min
          LOAD
```

```
19:08:37  Aspirating 9.970ml from SAMPLE_A
19:09:52  Dispensing 9.970ml in DEC
          WAIT
19:12:38  Waiting for : 0.30 min
          LOAD
19:13:00  Aspirating 9.970ml from SAMPLE_A
19:14:13  Dispensing 9.970ml in DEC
          WAIT
19:16:58  Waiting for : 0.30 min
          LOAD
19:17:19  Aspirating 9.970ml from SAMPLE_A
19:18:33  Dispensing 9.970ml in DEC
          RINSE ASPEC NEEDLE
19:21:17  rinsing aspec needle...
          DISPENSE
19:21:49  Aspirating 8000ul from SOLVENT_A
19:22:55  Dispensing 8000ul into SAMPLE_A
          MIX
19:23:35  Mixing SAMPLE_A with 7000ul of liquid
          LOAD
19:24:37  Aspirating 8.100ml from SAMPLE_A
19:25:41  Dispensing 8.100ml in DEC
          RINSE ASPEC NEEDLE
19:27:58  rinsing aspec needle...
          DISPENSE
19:28:31  Aspirating 8000ul from SOLVENT_A
19:29:35  Dispensing 8000ul into SAMPLE_A
          MIX
19:30:14  Mixing SAMPLE_A with 7000ul of liquid
          LOAD
19:31:15  Aspirating 8.100ml from SAMPLE_A
19:32:19  Dispensing 8.100ml in DEC

1997-10-23
08:34:22  End of execution
```

DKM 11/19/97

LSC Instrument Daily checks.

3H BACKGROUND:

Value	Date	Time
16.15	01 Aug 97	10:06
16.37	02 Aug 97	20:45
16.40	03 Aug 97	00:11
16.20	04 Aug 97	09:50
16.35	05 Aug 97	09:55
16.22	05 Aug 97	18:32
16.12	06 Aug 97	00:32
16.23	06 Aug 97	11:05
15.22	07 Aug 97	09:57
16.70	08 Aug 97	10:13
15.98	11 Aug 97	10:12
15.65	12 Aug 97	10:28
16.77	13 Aug 97	10:18
16.33	13 Aug 97	17:22
16.88	14 Aug 97	09:59
17.10	18 Aug 97	10:12
15.97	19 Aug 97	10:10
16.85	20 Aug 97	10:15
16.27	21 Aug 97	10:14
16.85	21 Aug 97	18:45
16.17	22 Aug 97	10:10
16.45	22 Aug 97	16:20
16.22	25 Aug 97	10:25
17.25	26 Aug 97	09:45
15.62	26 Aug 97	14:46
16.87	27 Aug 97	10:10
16.42	28 Aug 97	10:16
16.32	28 Aug 97	18:45
17.65	29 Aug 97	10:12
16.45	29 Aug 97	11:58
15.65	30 Aug 97	17:41
16.62	02 Sep 97	10:21
16.63	03 Sep 97	10:07
16.45	04 Sep 97	13:31
16.67	05 Sep 97	08:48
16.52	05 Sep 97	18:15
15.42	07 Sep 97	10:52
17.58	08 Sep 97	10:26
16.08	09 Sep 97	10:20
17.17	10 Sep 97	10:34
16.65	10 Sep 97	17:07
16.12	11 Sep 97	10:29
16.92	12 Sep 97	10:36
19.00	30 Sep 97	12:32
14.27	30 Sep 97	14:02
15.37	30 Sep 97	15:47
14.75	01 Oct 97	10:57
14.38	02 Oct 97	11:59
14.53	03 Oct 97	09:05
15.22	05 Oct 97	12:09
14.75	06 Oct 97	12:30
13.72	07 Oct 97	08:59
13.22	07 Oct 97	12:40
15.48	07 Oct 97	21:29
14.95	08 Oct 97	08:01

15.28	08 Oct 97	09:45
13.60	09 Oct 97	08:08
14.95	09 Oct 97	11:44
14.13	10 Oct 97	07:58
15.65	13 Oct 97	08:50
15.30	13 Oct 97	18:03
14.75	14 Oct 97	09:53
14.47	15 Oct 97	09:46
15.35	16 Oct 97	09:39
15.97	17 Oct 97	09:39
14.95	18 Oct 97	13:46
14.97	19 Oct 97	17:09
15.60	20 Oct 97	10:14
15.52	21 Oct 97	09:43
14.77	22 Oct 97	09:59
15.30	22 Oct 97	17:58
15.23	23 Oct 97	09:57
15.20	23 Oct 97	21:24
15.60	24 Oct 97	10:18
15.52	24 Oct 97	17:09
14.70	26 Oct 97	03:27
14.80	26 Oct 97	15:30
16.37	27 Oct 97	00:24
15.23	27 Oct 97	10:43
14.70	28 Oct 97	09:19
15.58	29 Oct 97	09:47
15.87	30 Oct 97	10:50
14.62	31 Oct 97	10:39
15.47	02 Nov 97	18:31
15.17	03 Nov 97	09:43
16.17	03 Nov 97	13:41
15.08	04 Nov 97	09:43
14.48	06 Nov 97	15:41
15.25	07 Nov 97	09:32
15.65	10 Nov 97	09:26
15.00	11 Nov 97	09:35
14.82	11 Nov 97	18:50
14.77	12 Nov 97	09:42
15.30	13 Nov 97	09:28
15.78	13 Nov 97	14:13
15.67	14 Nov 97	09:25
13.98	14 Nov 97	19:19
15.48	15 Nov 97	13:13
14.98	15 Nov 97	16:03
14.88	16 Nov 97	22:45

15.68	Mean
0.953	SD
100	# of points

3H EFFICIENCY:

Value	Date	Time
66.02	02 Aug 97	19:34
66.35	02 Aug 97	23:00
66.61	04 Aug 97	08:39

66.72	05	Aug	97	08:44
66.51	05	Aug	97	17:21
66.64	05	Aug	97	23:21
66.66	06	Aug	97	09:54
66.55	07	Aug	97	08:46
67.02	08	Aug	97	09:02
66.38	11	Aug	97	09:01
66.55	12	Aug	97	09:17
66.75	13	Aug	97	09:07
66.72	13	Aug	97	16:11
66.95	14	Aug	97	08:48
66.46	18	Aug	97	09:01
66.71	19	Aug	97	08:59
66.63	20	Aug	97	09:04
66.50	21	Aug	97	09:03
66.94	21	Aug	97	17:34
66.49	22	Aug	97	08:59
66.84	22	Aug	97	15:09
66.52	25	Aug	97	09:14
66.38	26	Aug	97	08:34
66.49	26	Aug	97	13:35
66.81	27	Aug	97	08:59
66.59	28	Aug	97	09:05
66.82	28	Aug	97	17:34
66.57	29	Aug	97	09:01
66.41	29	Aug	97	10:47
66.61	30	Aug	97	16:30
66.19	02	Sep	97	09:10
66.61	03	Sep	97	08:56
66.61	04	Sep	97	12:20
66.26	05	Sep	97	07:37
66.44	05	Sep	97	17:05
66.55	07	Sep	97	09:41
66.51	08	Sep	97	09:15
66.71	09	Sep	97	09:09
66.72	10	Sep	97	09:23
66.48	10	Sep	97	15:56
66.61	11	Sep	97	09:18
66.46	12	Sep	97	09:25
67.09	30	Sep	97	11:21
67.24	30	Sep	97	12:51
66.78	30	Sep	97	14:36
67.00	01	Oct	97	09:46
66.52	02	Oct	97	10:48
66.90	03	Oct	97	07:54
66.75	05	Oct	97	10:58
67.00	06	Oct	97	11:19
66.76	07	Oct	97	07:48
67.02	07	Oct	97	11:29
66.09	07	Oct	97	20:18
65.94	08	Oct	97	06:50
65.94	08	Oct	97	08:34
65.83	09	Oct	97	06:57
66.04	09	Oct	97	10:33
65.82	10	Oct	97	06:47
65.85	13	Oct	97	07:38
65.67	13	Oct	97	16:52
66.03	14	Oct	97	08:42

66.00	15 Oct 97	08:35
65.92	16 Oct 97	08:28
66.00	17 Oct 97	08:28
66.08	18 Oct 97	12:35
65.99	19 Oct 97	15:58
65.91	20 Oct 97	09:03
65.77	21 Oct 97	08:32
65.73	22 Oct 97	08:48
66.04	22 Oct 97	16:47
65.68	23 Oct 97	08:46
65.67	23 Oct 97	20:13
65.82	24 Oct 97	09:07
65.92	24 Oct 97	15:58
65.86	26 Oct 97	02:16
65.86	26 Oct 97	14:19
65.93	26 Oct 97	23:13
65.72	27 Oct 97	09:32
65.92	28 Oct 97	08:08
65.69	29 Oct 97	08:36
65.97	30 Oct 97	09:39
65.79	31 Oct 97	09:28
66.26	02 Nov 97	17:19
65.73	03 Nov 97	08:32
66.06	03 Nov 97	12:30
66.02	04 Nov 97	08:32
65.86	06 Nov 97	09:05
65.92	06 Nov 97	14:30
65.90	07 Nov 97	08:21
65.87	10 Nov 97	08:15
66.05	11 Nov 97	08:24
65.74	11 Nov 97	17:39
65.69	12 Nov 97	08:31
65.97	13 Nov 97	08:17
65.63	13 Nov 97	13:02
65.78	14 Nov 97	08:14
65.84	14 Nov 97	18:08
65.62	15 Nov 97	12:02
65.58	15 Nov 97	14:52
65.82	16 Nov 97	21:34
66.27	Mean	
0.434	SD	
100	# of points	

3H FIGURE OF MERIT (E^2/B):

Value	Date	Time
272.52	01 Aug 97	10:06
263.48	02 Aug 97	20:45
266.11	03 Aug 97	00:11
273.27	04 Aug 97	09:50
271.98	05 Aug 97	09:55
275.63	05 Aug 97	18:32
272.81	06 Aug 97	00:32
274.65	06 Aug 97	11:05
286.20	07 Aug 97	09:57

265.71	08 Aug 97	10:13
275.39	11 Aug 97	10:12
282.65	12 Aug 97	10:28
263.96	13 Aug 97	10:18
272.92	13 Aug 97	17:22
263.53	14 Aug 97	09:59
256.37	18 Aug 97	10:12
279.81	19 Aug 97	10:10
260.03	20 Aug 97	10:15
269.26	21 Aug 97	10:14
266.36	21 Aug 97	18:45
271.84	22 Aug 97	10:10
271.43	22 Aug 97	16:20
270.20	25 Aug 97	10:25
254.78	26 Aug 97	09:45
280.86	26 Aug 97	14:46
263.26	27 Aug 97	10:10
269.84	28 Aug 97	10:16
272.55	28 Aug 97	18:45
248.74	29 Aug 97	10:12
266.83	29 Aug 97	11:58
286.70	30 Aug 97	17:41
263.12	02 Sep 97	10:21
265.41	03 Sep 97	10:07
269.45	04 Sep 97	13:31
262.26	05 Sep 97	08:48
267.97	05 Sep 97	18:15
285.02	07 Sep 97	10:52
251.64	08 Sep 97	10:26
274.59	09 Sep 97	10:20
257.96	10 Sep 97	10:34
261.79	10 Sep 97	17:07
273.51	11 Sep 97	10:29
261.46	12 Sep 97	10:36
238.03	30 Sep 97	12:32
311.01	30 Sep 97	14:02
287.33	30 Sep 97	15:47
300.75	01 Oct 97	10:57
301.01	02 Oct 97	11:59
307.13	03 Oct 97	09:05
288.23	05 Oct 97	12:09
301.24	06 Oct 97	12:30
322.75	07 Oct 97	08:59
340.03	07 Oct 97	12:40
276.88	07 Oct 97	21:29
290.43	08 Oct 97	08:01
279.44	08 Oct 97	09:45
313.41	09 Oct 97	08:08
287.61	09 Oct 97	11:44
304.08	10 Oct 97	07:58
272.69	13 Oct 97	08:50
278.11	13 Oct 97	18:03
289.75	14 Oct 97	09:53
296.57	15 Oct 97	09:46
279.36	16 Oct 97	09:39
268.27	17 Oct 97	09:39
288.38	18 Oct 97	13:46
287.12	19 Oct 97	17:09

273.34	20 Oct 97	10:14
274.69	21 Oct 97	09:43
289.52	22 Oct 97	09:59
279.20	22 Oct 97	17:58
278.43	23 Oct 97	09:57
277.34	23 Oct 97	21:24
272.67	24 Oct 97	10:18
278.71	24 Oct 97	17:09
290.85	26 Oct 97	03:27
288.78	26 Oct 97	15:30
261.18	27 Oct 97	00:24
279.01	27 Oct 97	10:43
291.69	28 Oct 97	09:19
275.45	29 Oct 97	09:47
271.77	30 Oct 97	10:50
291.72	31 Oct 97	10:39
277.03	02 Nov 97	18:31
278.40	03 Nov 97	09:43
263.18	03 Nov 97	13:41
284.51	04 Nov 97	09:43
293.85	06 Nov 97	15:41
279.58	07 Nov 97	09:32
269.48	10 Nov 97	09:26
286.06	11 Nov 97	09:35
287.16	11 Nov 97	18:50
286.59	12 Nov 97	09:42
277.76	13 Nov 97	09:28
268.55	13 Nov 97	14:13
272.15	14 Nov 97	09:25
306.09	14 Nov 97	19:19
271.19	15 Nov 97	13:13
284.71	15 Nov 97	16:03
286.67	16 Nov 97	22:45

278.21	Mean
15.557	SD
100	# of points

3H CHI-SQUARE:

Value	Date	Time
13.48	02 Aug 97	19:45
13.70	02 Aug 97	23:10
13.00	04 Aug 97	08:49
12.91	05 Aug 97	08:55
14.82	05 Aug 97	17:32
23.71	05 Aug 97	23:31
15.27	06 Aug 97	10:05
15.36	07 Aug 97	08:57
18.14	08 Aug 97	09:13
15.52	11 Aug 97	09:12
31.42	12 Aug 97	09:28
19.55	13 Aug 97	09:17
7.43	13 Aug 97	16:21
16.68	14 Aug 97	08:59
12.64	18 Aug 97	09:12

26.22	19 Aug 97	09:09
29.01	20 Aug 97	09:14
21.29	21 Aug 97	09:14
19.10	21 Aug 97	17:44
25.45	22 Aug 97	09:09
22.59	22 Aug 97	15:20
18.84	25 Aug 97	09:24
22.23	26 Aug 97	08:45
17.07	26 Aug 97	13:46
16.65	27 Aug 97	09:10
9.84	28 Aug 97	09:16
20.98	28 Aug 97	17:44
23.49	29 Aug 97	09:12
9.40	29 Aug 97	10:57
19.53	30 Aug 97	16:40
24.43	02 Sep 97	09:21
17.02	03 Sep 97	09:07
21.48	04 Sep 97	12:31
14.87	05 Sep 97	07:48
22.09	05 Sep 97	17:15
12.05	07 Sep 97	09:51
15.91	08 Sep 97	09:26
22.54	09 Sep 97	09:19
22.35	10 Sep 97	09:33
17.14	10 Sep 97	16:06
15.63	11 Sep 97	09:28
12.66	12 Sep 97	09:35
25.78	30 Sep 97	11:32
23.51	30 Sep 97	13:02
22.84	30 Sep 97	14:47
15.35	01 Oct 97	09:57
31.43	02 Oct 97	10:59
19.03	03 Oct 97	08:04
16.94	05 Oct 97	11:09
14.92	06 Oct 97	11:29
22.56	07 Oct 97	07:59
18.18	07 Oct 97	11:40
16.25	07 Oct 97	20:28
15.41	08 Oct 97	07:00
10.04	08 Oct 97	08:45
23.07	09 Oct 97	07:08
30.52	09 Oct 97	10:44
19.42	10 Oct 97	06:58
20.42	13 Oct 97	07:49
11.91	13 Oct 97	17:03
14.99	14 Oct 97	08:52
18.82	15 Oct 97	08:45
31.64	16 Oct 97	08:39
14.21	17 Oct 97	08:39
12.89	18 Oct 97	12:46
14.61	19 Oct 97	16:08
27.56	20 Oct 97	09:14
20.14	21 Oct 97	08:43
26.31	22 Oct 97	08:59
25.65	22 Oct 97	16:58
25.41	23 Oct 97	08:56
18.81	23 Oct 97	20:24
17.87	24 Oct 97	09:17

26.22	19 Aug 97	09:09
29.01	20 Aug 97	09:14
21.29	21 Aug 97	09:14
19.10	21 Aug 97	17:44
25.45	22 Aug 97	09:09
22.59	22 Aug 97	15:20
18.84	25 Aug 97	09:24
22.23	26 Aug 97	08:45
17.07	26 Aug 97	13:46
16.65	27 Aug 97	09:10
9.84	28 Aug 97	09:16
20.98	28 Aug 97	17:44
23.49	29 Aug 97	09:12
9.40	29 Aug 97	10:57
19.53	30 Aug 97	16:40
24.43	02 Sep 97	09:21
17.02	03 Sep 97	09:07
21.48	04 Sep 97	12:31
14.87	05 Sep 97	07:48
22.09	05 Sep 97	17:15
12.05	07 Sep 97	09:51
15.91	08 Sep 97	09:26
22.54	09 Sep 97	09:19
22.35	10 Sep 97	09:33
17.14	10 Sep 97	16:06
15.63	11 Sep 97	09:28
12.66	12 Sep 97	09:35
25.78	30 Sep 97	11:32
23.51	30 Sep 97	13:02
22.84	30 Sep 97	14:47
15.35	01 Oct 97	09:57
31.43	02 Oct 97	10:59
19.03	03 Oct 97	08:04
16.94	05 Oct 97	11:09
14.92	06 Oct 97	11:29
22.56	07 Oct 97	07:59
18.18	07 Oct 97	11:40
16.25	07 Oct 97	20:28
15.41	08 Oct 97	07:00
10.04	08 Oct 97	08:45
23.07	09 Oct 97	07:08
30.52	09 Oct 97	10:44
19.42	10 Oct 97	06:58
20.42	13 Oct 97	07:49
11.91	13 Oct 97	17:03
14.99	14 Oct 97	08:52
18.82	15 Oct 97	08:45
31.64	16 Oct 97	08:39
14.21	17 Oct 97	08:39
12.89	18 Oct 97	12:46
14.61	19 Oct 97	16:08
27.56	20 Oct 97	09:14
20.14	21 Oct 97	08:43
26.31	22 Oct 97	08:59
25.65	22 Oct 97	16:58
25.41	23 Oct 97	08:56
18.81	23 Oct 97	20:24
17.87	24 Oct 97	09:17

21.59	24 Oct 97	16:09
25.74	26 Oct 97	02:27
21.57	26 Oct 97	14:30
15.07	26 Oct 97	23:23
9.70	27 Oct 97	09:43
10.75	28 Oct 97	08:19
10.65	29 Oct 97	08:46
13.26	30 Oct 97	09:49
32.43	31 Oct 97	09:38
20.38	02 Nov 97	17:30
16.60	03 Nov 97	08:43
17.50	03 Nov 97	12:40
15.67	04 Nov 97	08:43
17.98	06 Nov 97	09:16
5.99	06 Nov 97	14:40
29.41	07 Nov 97	08:32
23.16	10 Nov 97	08:26
21.62	11 Nov 97	08:34
20.26	11 Nov 97	17:49
13.59	12 Nov 97	08:42
30.71	13 Nov 97	08:28
13.09	13 Nov 97	13:13
17.07	14 Nov 97	08:24
11.14	14 Nov 97	18:18
11.62	15 Nov 97	12:12
20.91	15 Nov 97	15:02
9.85	16 Nov 97	21:45

18.67 Mean
5.921 SD
100 # of points

14C BACKGROUND:

Value	Date	Time
22.25	01 Aug 97	10:06
22.62	02 Aug 97	20:45
23.15	03 Aug 97	00:11
23.20	04 Aug 97	09:50
23.27	05 Aug 97	09:55
22.92	05 Aug 97	18:32
22.50	06 Aug 97	00:32
22.80	06 Aug 97	11:05
21.47	07 Aug 97	09:57
23.78	08 Aug 97	10:13
22.47	11 Aug 97	10:12
22.67	12 Aug 97	10:28
23.50	13 Aug 97	10:18
22.95	13 Aug 97	17:22
23.10	14 Aug 97	09:59
23.80	18 Aug 97	10:12
22.28	19 Aug 97	10:10
23.43	20 Aug 97	10:15
22.75	21 Aug 97	10:14
23.37	21 Aug 97	18:45
22.88	22 Aug 97	10:10

23.05	22 Aug 97	16:20
22.80	25 Aug 97	10:25
24.07	26 Aug 97	09:45
22.48	26 Aug 97	14:46
24.15	27 Aug 97	10:10
23.43	28 Aug 97	10:16
23.37	28 Aug 97	18:45
24.22	29 Aug 97	10:12
23.57	29 Aug 97	11:58
22.40	30 Aug 97	17:41
23.23	02 Sep 97	10:21
22.88	03 Sep 97	10:07
23.20	04 Sep 97	13:31
23.85	05 Sep 97	08:48
23.00	05 Sep 97	18:15
21.87	07 Sep 97	10:52
24.53	08 Sep 97	10:26
22.95	09 Sep 97	10:20
24.20	10 Sep 97	10:34
23.52	10 Sep 97	17:07
22.82	11 Sep 97	10:29
23.88	12 Sep 97	10:36
26.35	30 Sep 97	12:32
21.85	30 Sep 97	14:02
23.48	30 Sep 97	15:47
22.43	01 Oct 97	10:57
22.12	02 Oct 97	11:59
22.30	03 Oct 97	09:05
22.12	05 Oct 97	12:09
22.45	06 Oct 97	12:30
21.58	07 Oct 97	08:59
20.92	07 Oct 97	12:40
23.32	07 Oct 97	21:29
23.27	08 Oct 97	08:01
23.58	08 Oct 97	09:45
22.27	09 Oct 97	08:08
22.52	09 Oct 97	11:44
22.28	10 Oct 97	07:58
24.03	13 Oct 97	08:50
23.18	13 Oct 97	18:03
22.98	14 Oct 97	09:53
23.10	15 Oct 97	09:46
23.53	16 Oct 97	09:39
24.28	17 Oct 97	09:39
23.00	18 Oct 97	13:46
22.87	19 Oct 97	17:09
23.97	20 Oct 97	10:14
23.35	21 Oct 97	09:43
22.98	22 Oct 97	09:59
24.12	22 Oct 97	17:58
23.87	23 Oct 97	09:57
22.48	23 Oct 97	21:24
24.15	24 Oct 97	10:18
23.67	24 Oct 97	17:09
22.70	26 Oct 97	03:27
22.85	26 Oct 97	15:30
24.87	27 Oct 97	00:24
23.87	27 Oct 97	10:43

```
   23.63   28 Oct 97   09:19
   23.40   29 Oct 97   09:47
   23.50   30 Oct 97   10:50
   22.62   31 Oct 97   10:39
   23.33   02 Nov 97   18:31
   23.52   03 Nov 97   09:43
   24.80   03 Nov 97   13:41
   22.78   04 Nov 97   09:43
   22.83   06 Nov 97   15:41
   22.98   07 Nov 97   09:32
   23.52   10 Nov 97   09:26
   23.10   11 Nov 97   09:35
   23.35   11 Nov 97   18:50
   22.95   12 Nov 97   09:42
   24.20   13 Nov 97   09:28
   24.48   13 Nov 97   14:13
   24.53   14 Nov 97   09:25
   21.75   14 Nov 97   19:19
   23.65   15 Nov 97   13:13
   22.82   15 Nov 97   16:03
   23.72   16 Nov 97   22:45
________
   23.19   Mean
   0.808   SD
     100   # of points



14C EFFICIENCY:
   Value   Date        Time
   96.46   02 Aug 97   19:22
   96.39   02 Aug 97   22:48
   96.33   04 Aug 97   08:27
   96.76   05 Aug 97   08:33
   96.61   05 Aug 97   17:09
   96.62   05 Aug 97   23:09
   96.14   06 Aug 97   09:43
   96.27   07 Aug 97   08:34
   96.56   08 Aug 97   08:50
   96.32   11 Aug 97   08:49
   96.26   12 Aug 97   09:05
   96.08   13 Aug 97   08:55
   96.48   13 Aug 97   15:59
   96.09   14 Aug 97   08:36
   96.34   18 Aug 97   08:50
   96.15   19 Aug 97   08:47
   96.39   20 Aug 97   08:52
   95.94   21 Aug 97   08:52
   96.69   21 Aug 97   17:22
   96.67   22 Aug 97   08:47
   96.48   22 Aug 97   14:57
   96.56   25 Aug 97   09:02
   96.60   26 Aug 97   08:22
   96.65   26 Aug 97   13:23
   96.81   27 Aug 97   08:47
   96.38   28 Aug 97   08:53
   96.40   28 Aug 97   17:22
```

96.51	29 Aug 97	08:49
96.73	29 Aug 97	10:35
96.61	30 Aug 97	16:18
96.24	02 Sep 97	08:59
96.08	03 Sep 97	08:45
96.22	04 Sep 97	12:08
96.66	05 Sep 97	07:25
96.76	05 Sep 97	16:53
96.19	07 Sep 97	09:29
96.30	08 Sep 97	09:04
96.23	09 Sep 97	08:57
96.34	10 Sep 97	09:11
96.90	10 Sep 97	15:44
95.94	11 Sep 97	09:06
96.14	12 Sep 97	09:13
96.49	30 Sep 97	11:10
96.27	30 Sep 97	12:39
96.53	30 Sep 97	14:24
96.13	01 Oct 97	09:34
96.80	02 Oct 97	10:37
96.79	03 Oct 97	07:42
96.29	05 Oct 97	10:46
96.81	06 Oct 97	11:07
96.90	07 Oct 97	07:36
97.10	07 Oct 97	11:17
96.53	07 Oct 97	20:06
97.36	08 Oct 97	06:38
97.06	08 Oct 97	08:22
96.68	09 Oct 97	06:45
96.75	09 Oct 97	10:21
96.87	10 Oct 97	06:35
97.00	13 Oct 97	07:27
96.93	13 Oct 97	16:41
96.95	14 Oct 97	08:30
96.96	15 Oct 97	08:23
96.52	16 Oct 97	08:16
96.38	17 Oct 97	08:17
96.67	18 Oct 97	12:23
96.77	19 Oct 97	15:46
96.73	20 Oct 97	08:51
97.02	21 Oct 97	08:20
96.80	22 Oct 97	08:36
96.95	22 Oct 97	16:36
96.40	23 Oct 97	08:34
96.63	23 Oct 97	20:02
96.45	24 Oct 97	08:55
96.41	24 Oct 97	15:47
97.07	26 Oct 97	02:05
96.78	26 Oct 97	14:08
96.14	26 Oct 97	23:01
96.65	27 Oct 97	09:21
96.52	28 Oct 97	07:56
96.85	29 Oct 97	08:24
95.90	30 Oct 97	09:27
96.85	31 Oct 97	09:16
96.27	02 Nov 97	17:08
96.91	03 Nov 97	08:20
97.04	03 Nov 97	12:18

97.48	04 Nov 97	08:20
96.72	06 Nov 97	08:54
96.93	06 Nov 97	14:18
96.95	07 Nov 97	08:09
96.18	10 Nov 97	08:04
96.83	11 Nov 97	08:12
96.87	11 Nov 97	17:27
97.08	12 Nov 97	08:20
96.48	13 Nov 97	08:05
96.65	13 Nov 97	12:50
96.93	14 Nov 97	08:02
97.00	14 Nov 97	17:56
96.68	15 Nov 97	11:50
96.92	15 Nov 97	14:40
96.43	16 Nov 97	21:23

96.59	Mean
0.324	SD
100	# of points

14C FIGURE OF MERIT (E^2/B):

Value	Date	Time
550.52	01 Aug 97	10:06
544.48	02 Aug 97	20:45
522.82	03 Aug 97	00:11
516.81	04 Aug 97	09:50
528.68	05 Aug 97	09:55
539.71	05 Aug 97	18:32
553.04	06 Aug 97	00:32
524.63	06 Aug 97	11:05
578.00	07 Aug 97	09:57
518.01	08 Aug 97	10:13
547.07	11 Aug 97	10:12
537.74	12 Aug 97	10:28
512.51	13 Aug 97	10:18
539.02	13 Aug 97	17:22
531.96	14 Aug 97	09:59
517.24	18 Aug 97	10:12
548.58	19 Aug 97	10:10
534.90	20 Aug 97	10:15
543.17	21 Aug 97	10:14
532.07	21 Aug 97	18:45
531.30	22 Aug 97	10:10
533.98	22 Aug 97	16:20
527.30	25 Aug 97	10:25
520.55	26 Aug 97	09:45
532.76	26 Aug 97	14:46
496.87	27 Aug 97	10:10
526.44	28 Aug 97	10:16
521.41	28 Aug 97	18:45
515.72	29 Aug 97	10:12
516.72	29 Aug 97	11:58
528.75	30 Aug 97	17:41
531.84	02 Sep 97	10:21
538.28	03 Sep 97	10:07

528.73	04 Sep 97	13:31
528.11	05 Sep 97	08:48
535.13	05 Sep 97	18:15
567.17	07 Sep 97	10:52
505.09	08 Sep 97	10:26
528.30	09 Sep 97	10:20
515.95	10 Sep 97	10:34
522.59	10 Sep 97	17:07
543.99	11 Sep 97	10:29
510.37	12 Sep 97	10:36
475.15	30 Sep 97	12:32
524.08	30 Sep 97	14:02
477.42	30 Sep 97	15:47
504.89	01 Oct 97	10:57
512.77	02 Oct 97	11:59
518.16	03 Oct 97	09:05
520.07	05 Oct 97	12:09
512.41	06 Oct 97	12:30
520.97	07 Oct 97	08:59
555.46	07 Oct 97	12:40
494.93	07 Oct 97	21:29
500.81	08 Oct 97	08:01
487.67	08 Oct 97	09:45
502.34	09 Oct 97	08:08
512.34	09 Oct 97	11:44
515.44	10 Oct 97	07:58
462.62	13 Oct 97	08:50
496.70	13 Oct 97	18:03
486.99	14 Oct 97	09:53
490.93	15 Oct 97	09:46
494.96	16 Oct 97	09:39
472.87	17 Oct 97	09:39
507.41	18 Oct 97	13:46
512.68	19 Oct 97	17:09
479.03	20 Oct 97	10:14
494.85	21 Oct 97	09:43
492.50	22 Oct 97	09:59
463.53	22 Oct 97	17:58
479.39	23 Oct 97	09:57
503.93	23 Oct 97	21:24
472.55	24 Oct 97	10:18
485.02	24 Oct 97	17:09
499.01	26 Oct 97	03:27
501.91	26 Oct 97	15:30
460.84	27 Oct 97	00:24
488.31	27 Oct 97	10:43
482.08	28 Oct 97	09:19
486.44	29 Oct 97	09:47
486.21	30 Oct 97	10:50
502.91	31 Oct 97	10:39
477.56	02 Nov 97	18:31
497.95	03 Nov 97	09:43
461.14	03 Nov 97	13:41
517.65	04 Nov 97	09:43
496.94	06 Nov 97	15:41
509.42	07 Nov 97	09:32
477.07	10 Nov 97	09:26
501.41	11 Nov 97	09:35

486.96	11 Nov 97	18:50
507.87	12 Nov 97	09:42
475.57	13 Nov 97	09:28
465.71	13 Nov 97	14:13
474.13	14 Nov 97	09:25
534.67	14 Nov 97	19:19
486.17	15 Nov 97	13:13
519.26	15 Nov 97	16:03
478.27	16 Nov 97	22:45
510.35	Mean	
25.294	SD	
100	# of points	

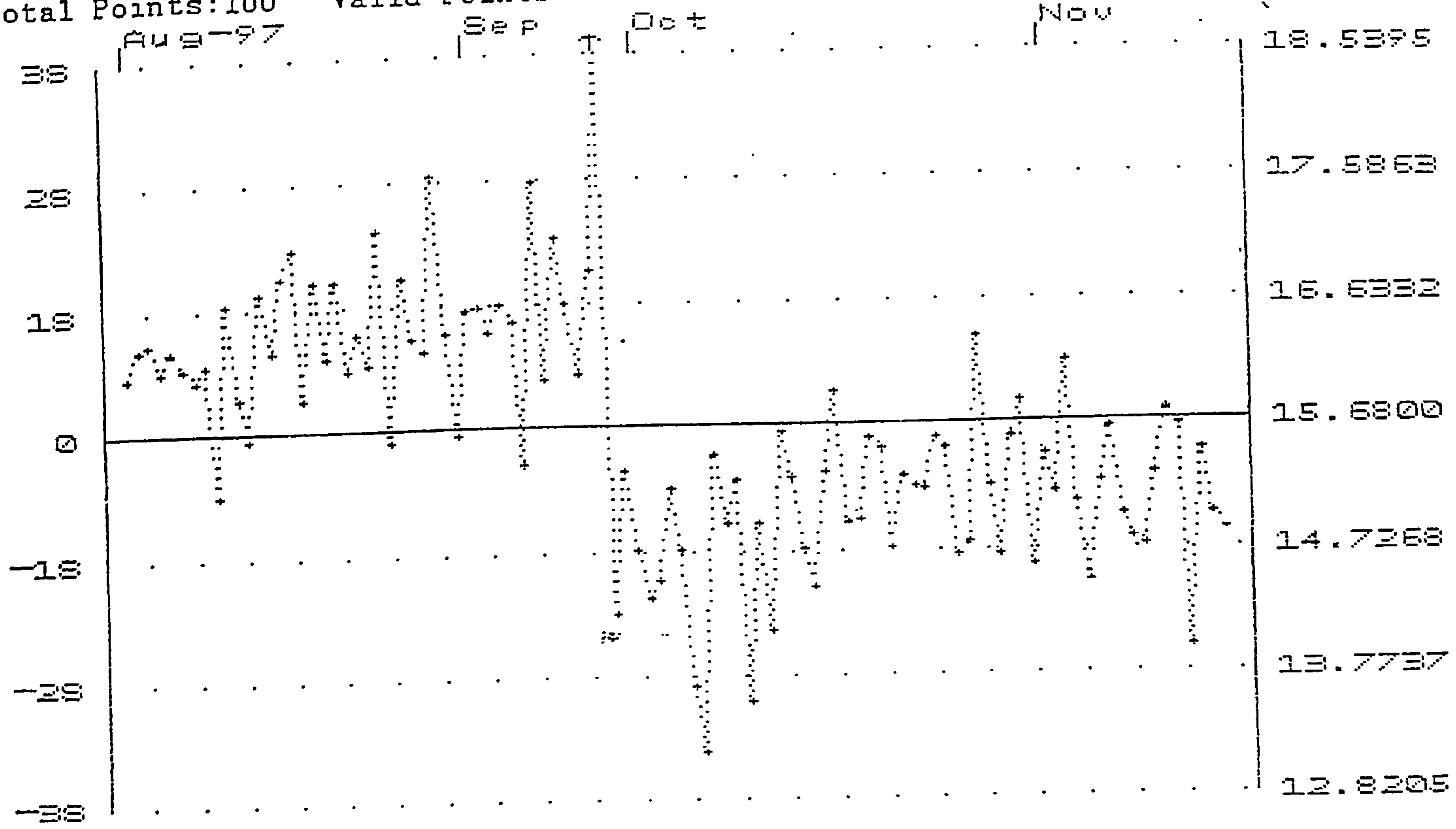
14C CHI-SQUARE:

Value	Date	Time
19.29	02 Aug 97	19:33
23.03	02 Aug 97	22:58
21.16	04 Aug 97	08:38
12.48	05 Aug 97	08:43
34.61	05 Aug 97	17:20
33.21	05 Aug 97	23:19
21.74	06 Aug 97	09:53
26.50	07 Aug 97	08:45
27.82	08 Aug 97	09:01
24.02	11 Aug 97	09:00
17.65	12 Aug 97	09:16
19.59	13 Aug 97	09:06
16.62	13 Aug 97	16:09
16.05	14 Aug 97	08:47
21.04	18 Aug 97	09:00
15.96	19 Aug 97	08:58
27.06	20 Aug 97	09:03
16.96	21 Aug 97	09:02
20.95	21 Aug 97	17:33
18.44	22 Aug 97	08:58
22.59	22 Aug 97	15:08
14.91	25 Aug 97	09:13
15.72	26 Aug 97	08:33
15.28	26 Aug 97	13:34
14.22	27 Aug 97	08:58
18.52	28 Aug 97	09:04
11.62	28 Aug 97	17:32
22.41	29 Aug 97	09:00
23.18	29 Aug 97	10:46
16.96	30 Aug 97	16:28
12.54	02 Sep 97	09:09
18.13	03 Sep 97	08:55
15.90	04 Sep 97	12:19
25.46	05 Sep 97	07:36
19.40	05 Sep 97	17:03
23.59	07 Sep 97	09:39
18.97	08 Sep 97	09:14
13.14	09 Sep 97	09:08
23.10	10 Sep 97	09:22

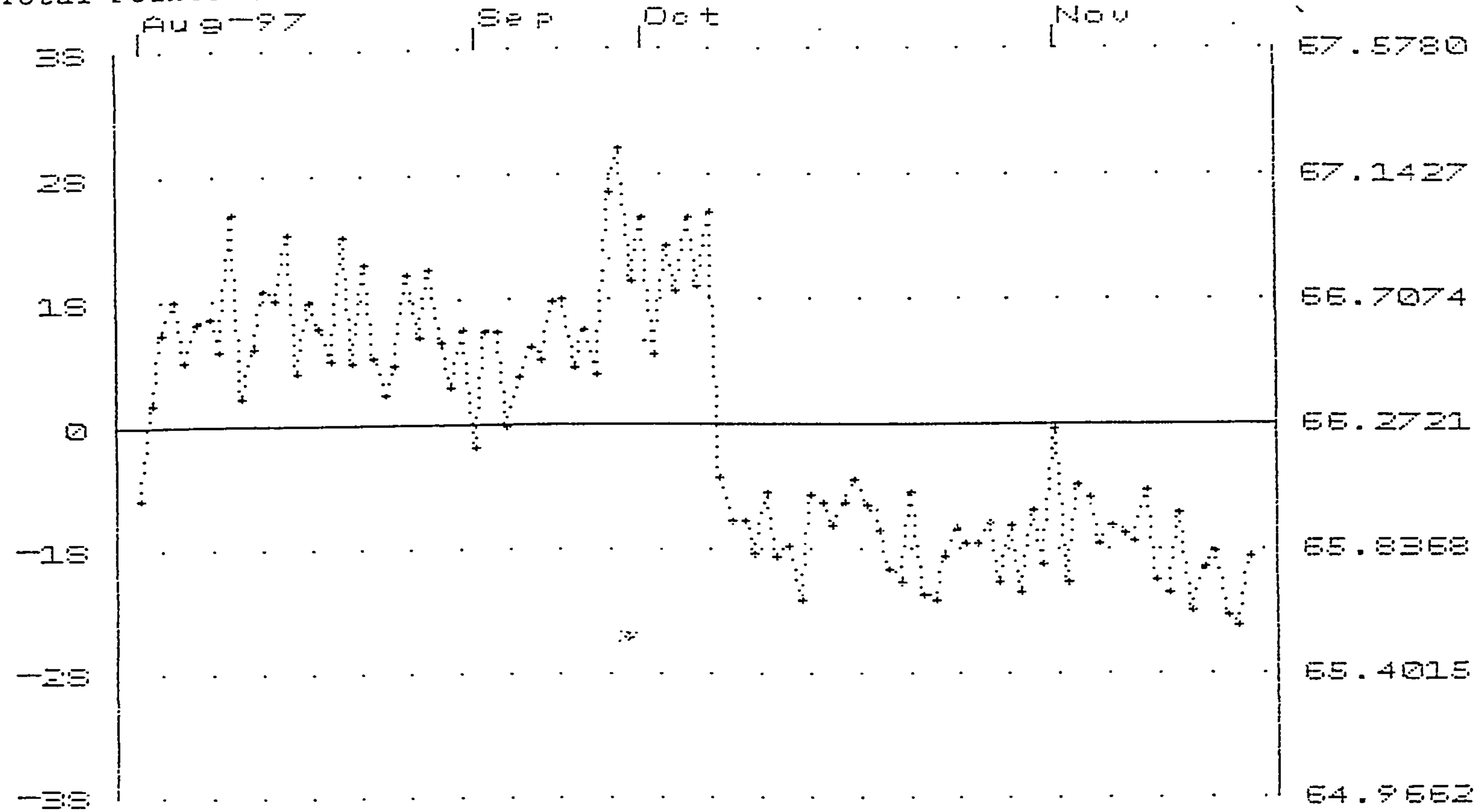
29.09	10 Sep 97	15:54
10.08	11 Sep 97	09:17
14.31	12 Sep 97	09:23
22.38	30 Sep 97	11:20
19.84	30 Sep 97	12:50
30.30	30 Sep 97	14:35
19.25	01 Oct 97	09:45
21.07	02 Oct 97	10:47
10.87	03 Oct 97	07:53
13.37	05 Oct 97	10:57
12.48	06 Oct 97	11:18
23.07	07 Oct 97	07:47
15.49	07 Oct 97	11:28
20.00	07 Oct 97	20:17
13.62	08 Oct 97	06:49
20.10	08 Oct 97	08:33
6.65	09 Oct 97	06:56
15.94	09 Oct 97	10:32
20.96	10 Oct 97	06:46
11.91	13 Oct 97	07:37
16.98	13 Oct 97	16:51
11.43	14 Oct 97	08:41
19.35	15 Oct 97	08:33
19.78	16 Oct 97	08:27
26.73	17 Oct 97	08:27
14.39	18 Oct 97	12:34
10.73	19 Oct 97	15:56
11.03	20 Oct 97	09:02
19.63	21 Oct 97	08:31
21.21	22 Oct 97	08:47
25.29	22 Oct 97	16:46
16.81	23 Oct 97	08:44
36.90	23 Oct 97	20:12
8.60	24 Oct 97	09:05
11.94	24 Oct 97	15:57
29.68	26 Oct 97	02:15
8.26	26 Oct 97	14:18
24.81	26 Oct 97	23:12
14.33	27 Oct 97	09:31
16.99	28 Oct 97	08:07
15.18	29 Oct 97	08:35
17.40	30 Oct 97	09:37
15.56	31 Oct 97	09:26
25.99	02 Nov 97	17:18
16.61	03 Nov 97	08:31
15.06	03 Nov 97	12:29
19.33	04 Nov 97	08:31
19.13	06 Nov 97	09:04
14.87	06 Nov 97	14:28
17.01	07 Nov 97	08:20
18.35	10 Nov 97	08:14
17.81	11 Nov 97	08:23
13.96	11 Nov 97	17:38
26.38	12 Nov 97	08:30
20.93	13 Nov 97	08:16
19.03	13 Nov 97	13:01
25.76	14 Nov 97	08:13
18.51	14 Nov 97	18:06

```
   15.12   15 Nov 97   12:00
   23.73   15 Nov 97   14:50
   14.39   16 Nov 97   21:33
 _______
   18.86   Mean
   5.751   SD
     100   # of points


 5  21   5  60.00    22.38    24.30    20.50 39.010 242.00 0.921168          51
```

Parameter: 3H BACKGROUND
Total Points:100 Valid Points:100 Mean: 15.68 SD: 0.953

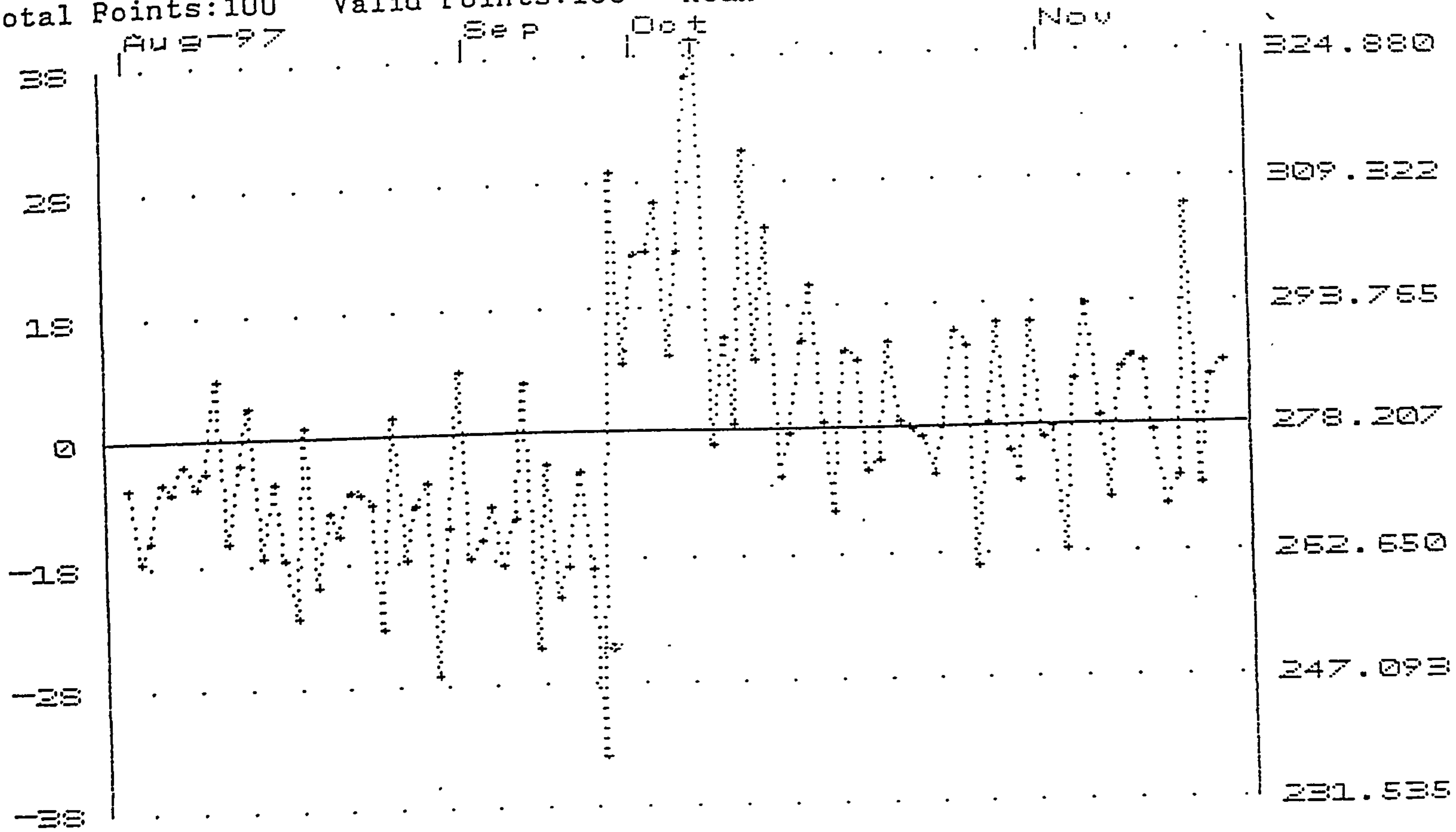


Parameter: 3H EFFICIENCY
Total Points:100 Valid Points:100 Mean: 66.27 SD: 0.435

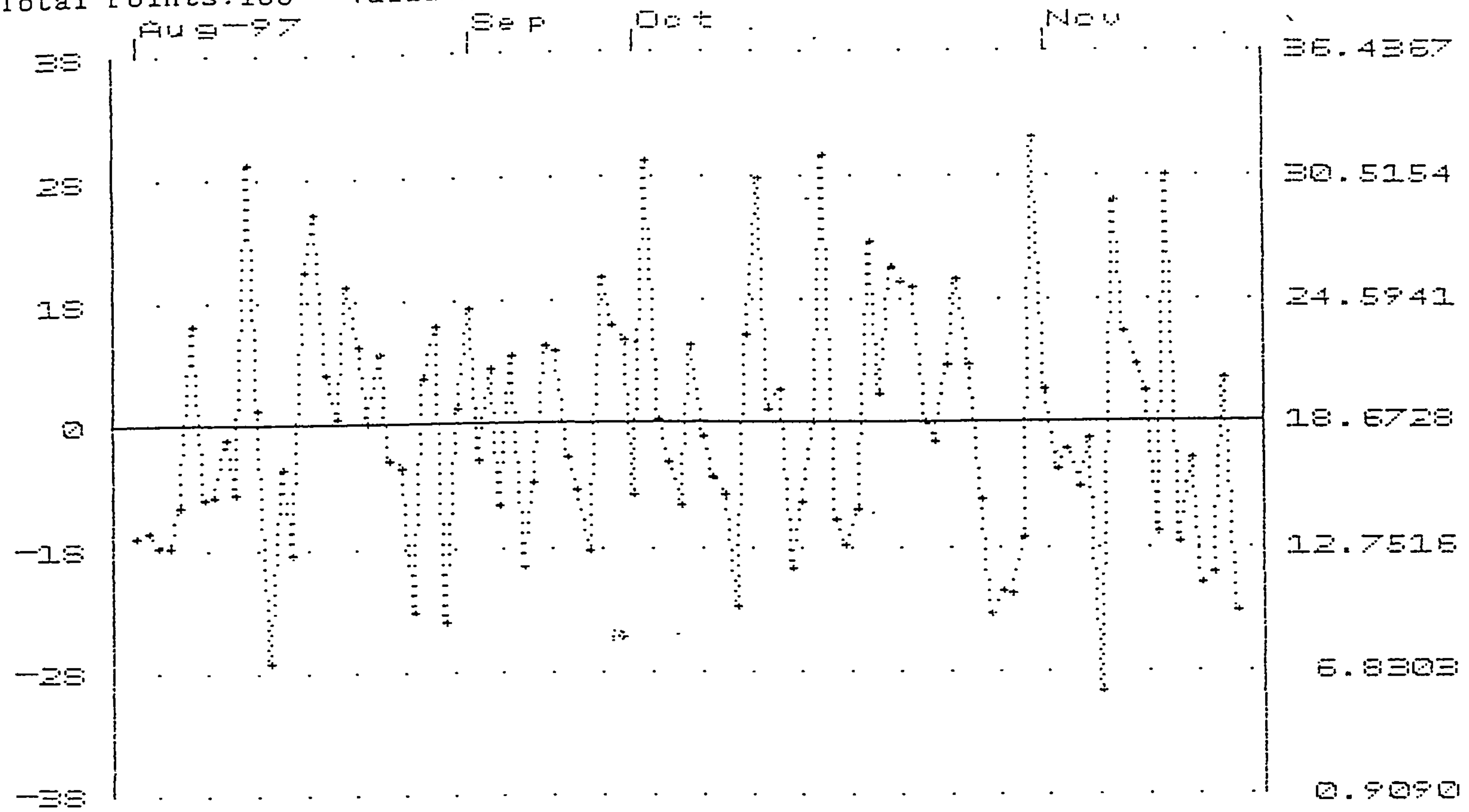


17 Nov 97 09:37 IPA CHARTS Page #1

Parameter: 3H FIGURE OF MERIT (E^2/B)
Total Points:100 Valid Points:100 Mean: 278.21 SD: 15.557

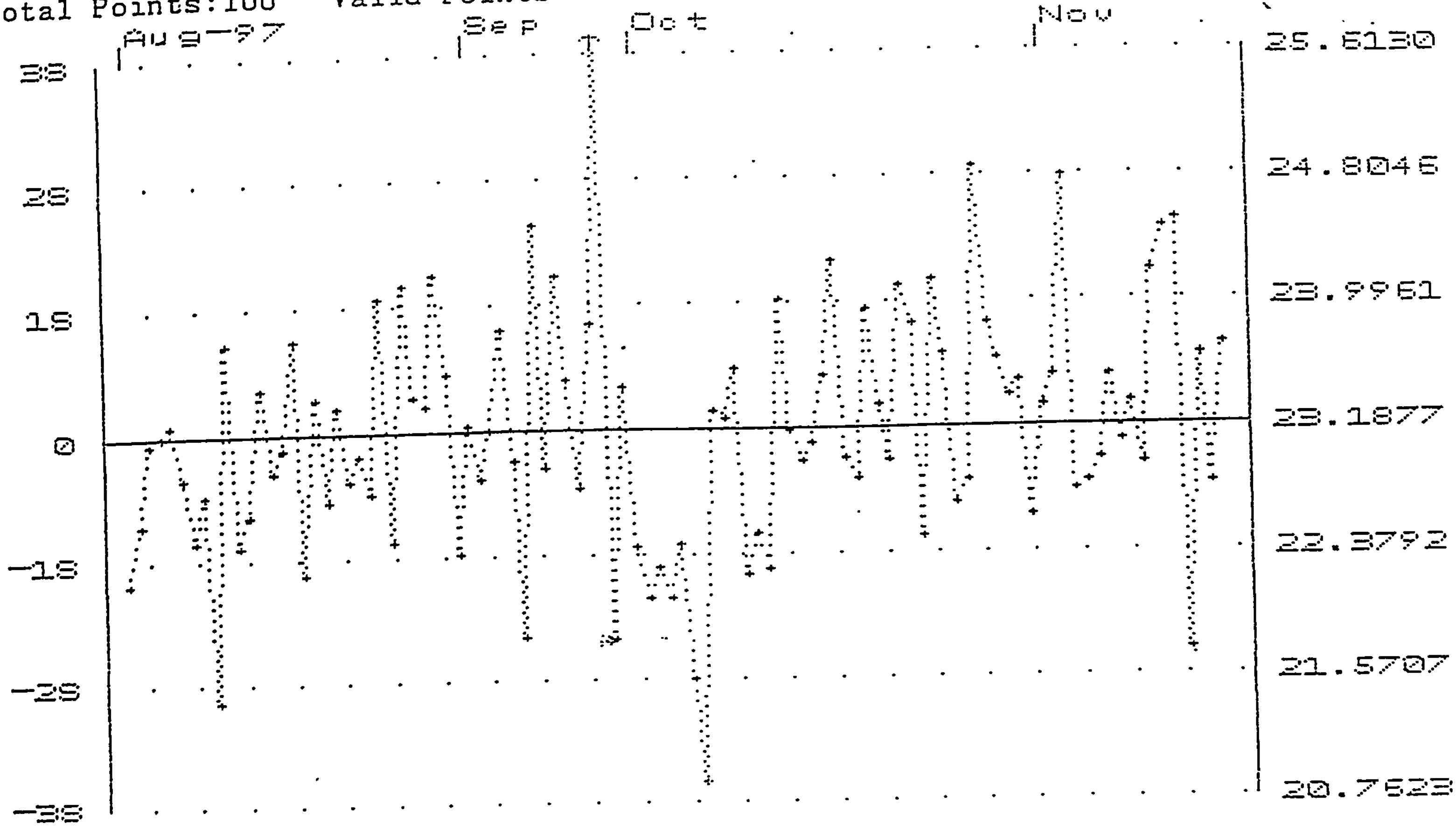


Parameter: 3H CHI-SQUARE
Total Points:100 Valid Points:100 Mean: 18.67 SD: 5.921



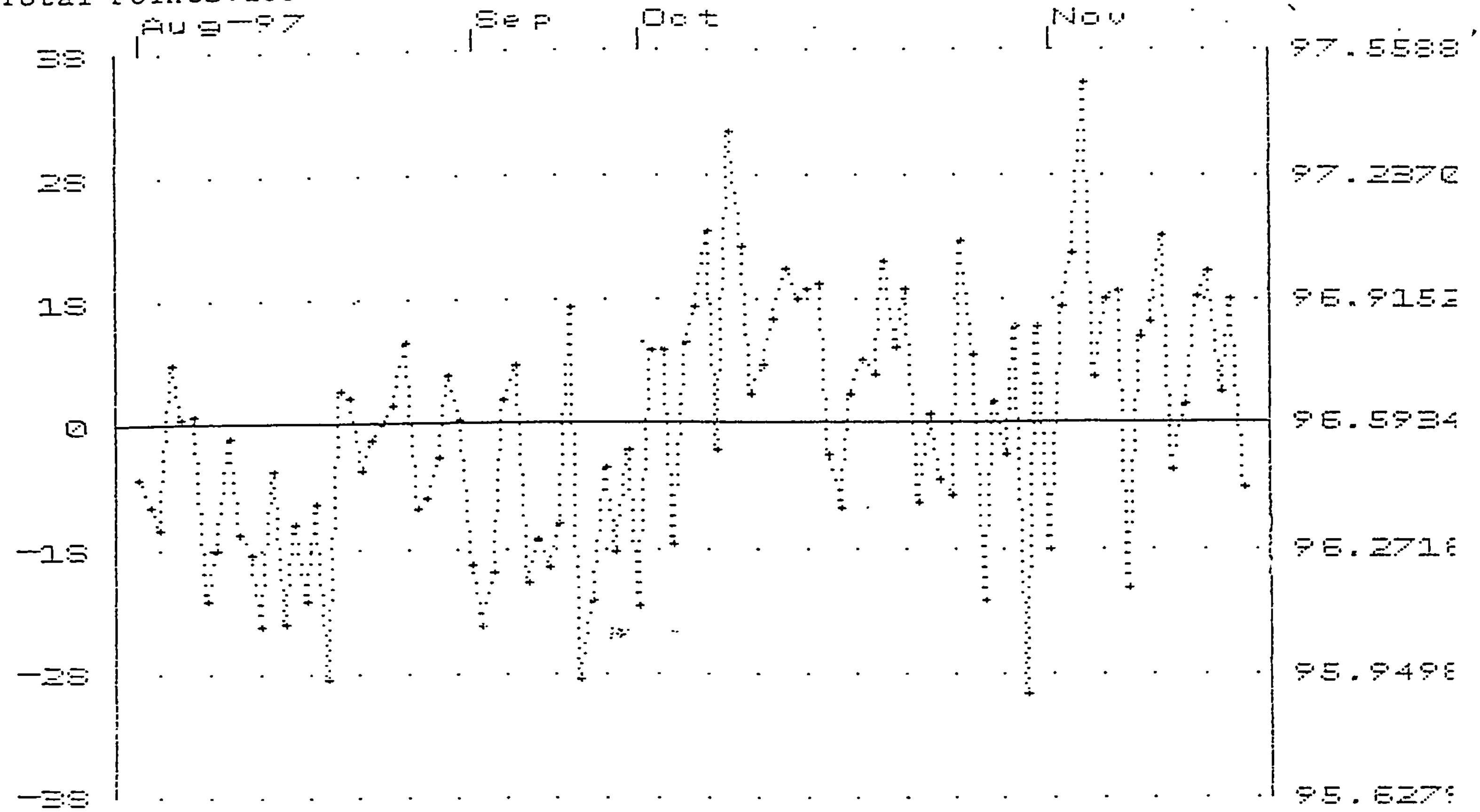
17 Nov 97 09:39 IPA CHARTS Page #1

Parameter: 14C BACKGROUND
Total Points:100 Valid Points:100 Mean: 23.19 SD: 0.808



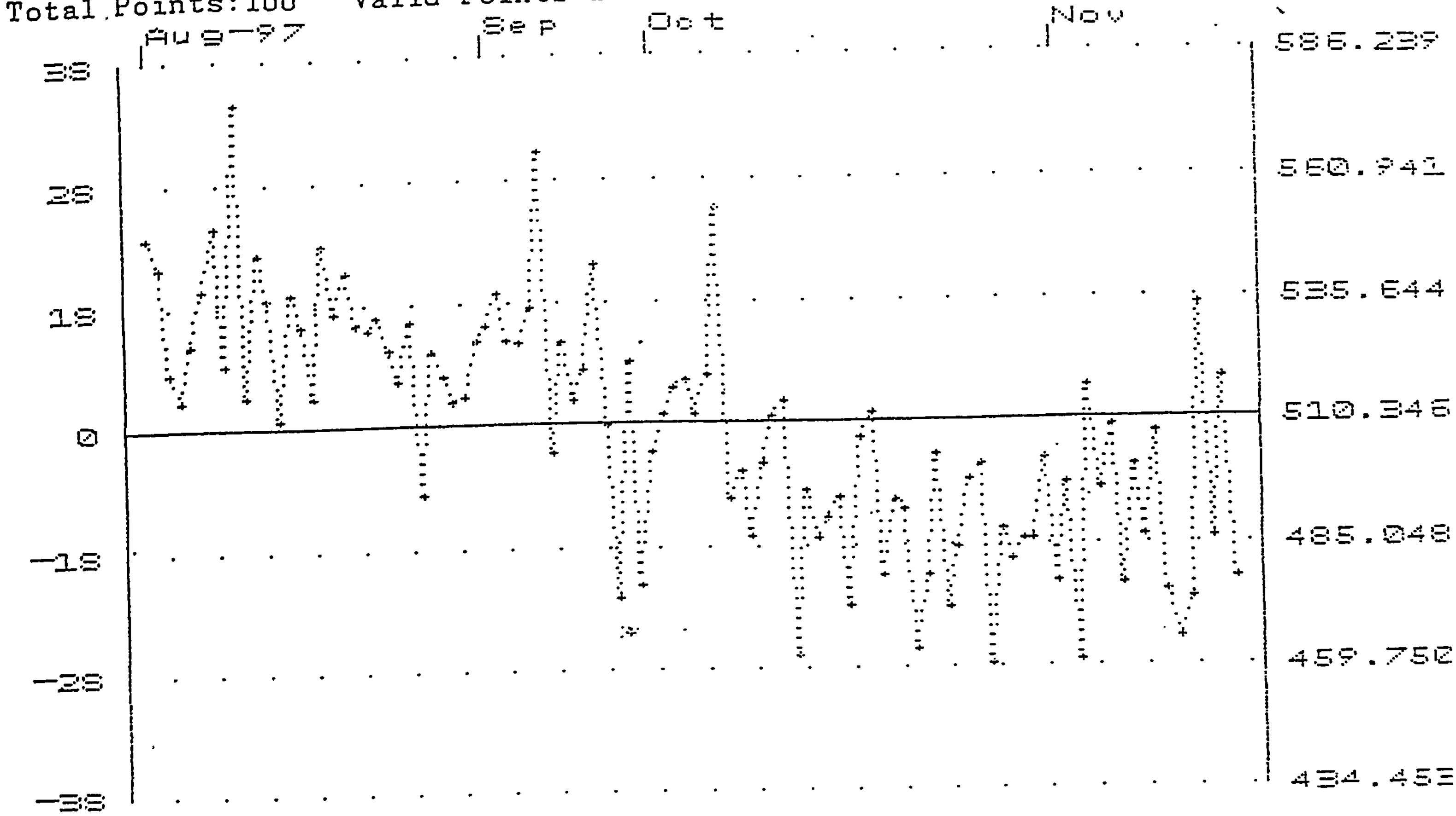
Parameter: 14C EFFICIENCY

Total Points:100 Valid Points:100 Mean: 96.59 SD: 0.322

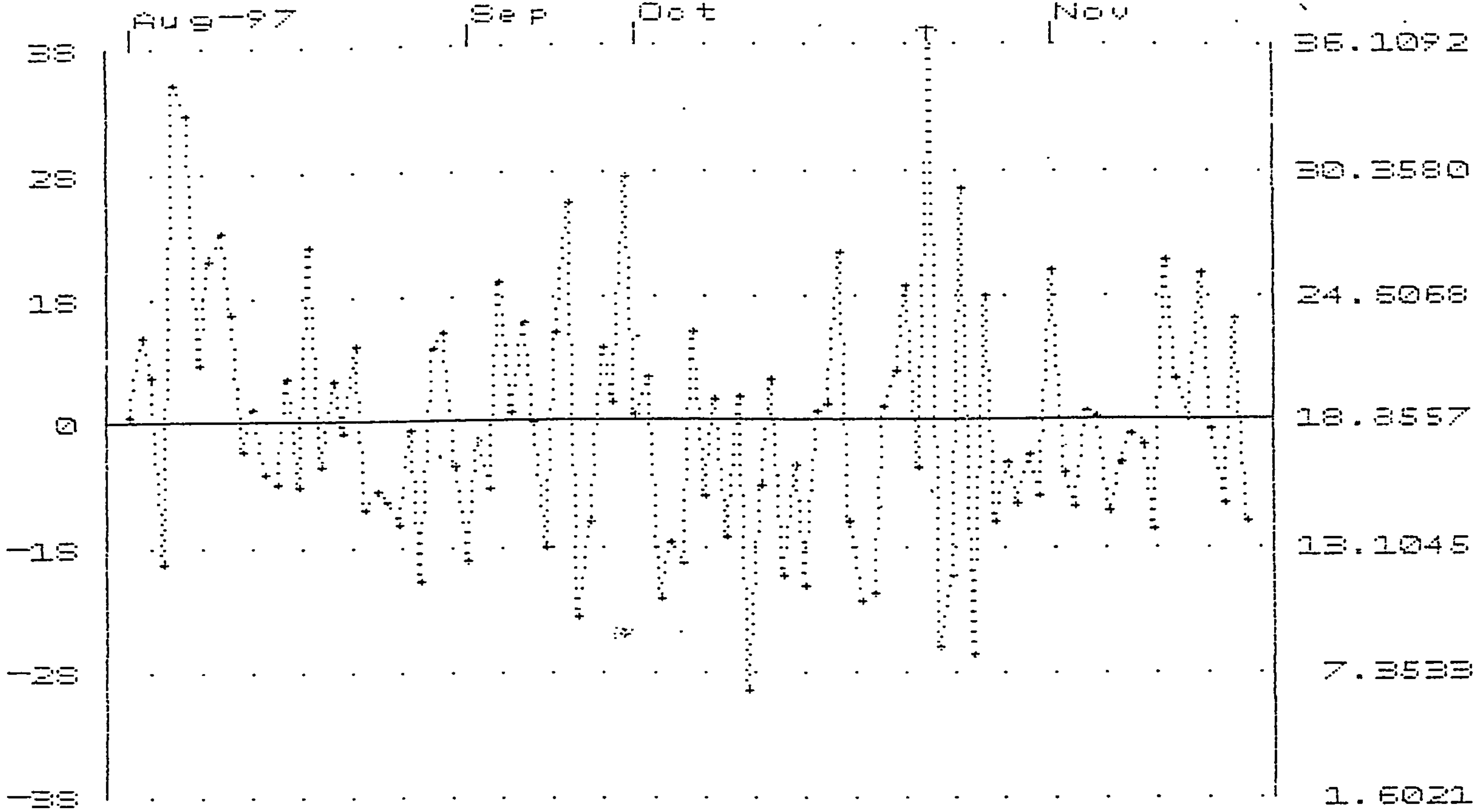


17 Nov 97 09:41 IPA CHARTS

Parameter: 14C FIGURE OF MERIT (E^2/B)
Total Points:100 Valid Points:100 Mean: 510.35 SD: 25.298



Parameter: 14C CHI-SQUARE
Total Points:100 Valid Points:100 Mean: 18.86 SD: 5.751



Fraction Development

11/27/97
DKM

```
                                                              DATE COUNTED:29-AUG-1997
                                                          DATE CALCULATED:02-SEP-1997
                                                                 QA FILE-PE-1141
                                                                 ANALYST:DKM/SPW


                                   GILSON
                              Tc-99 (TevaSpec)
                              ================


 SAMPLE     MD EFFIC.   tSIE    GROSS    DILUTION  WEIGHT      NET       NET     COUNTING
 NUMBER     or FACT.    VALUE  ACTIVITY   FACTOR   FRACTION  ACTIVITY  ACTIVITY  CERTAINTY
            MS CPM/DPM          (CPM)      (ml)      (g)     (pCi/L)   (pCi/g)    (+/-)
 ========= == ======   =====  ========  ========  ========= ========= ========= ========
 BLANK          0.930 284.73    19.30
 CONTROL        0.936 282.78   502.78     0.500     0.000   4.61e+05        NA 5.70e+03
NNDUPLICATE        NA     NA       NA     0.000     0.000         NA        NA       NA
 SPIKE          0.935 278.84   496.97     0.000     0.070         NA  3.26e+03 4.05e+01
NN     NA    MD    NA     NA       NA     0.000     0.000         NA        NA       NA
 970815-002MS   0.934 276.44    24.25     0.000     0.070         NA  9.08e+00 1.22e+01
```

98.74 %
Spike
recovery

```
                                                  DATE COUNTED:29-AUG-1997
                                               DATE CALCULATED:02-SEP-1997
                                                      QA FILE-PE-1141
                                                      ANALYST:DKM/SPW


                              QA DATA SHEET
                              =============

SAMPLE COUNTING TIME IN UNITS OF MINUTES:                    60.0

PACKARD 2550TR LIQ. SCINTILLATION COUNTER #s 1, 2, OR 3:        2

DESIRED ACTIVITY UNITS (1=pCi, 2=uCi, 3=dpm):                   1

DESIRED WEIGHT FRACTION UNITS (1=g, 2=filter):                  1

INTERNAL CONTROL CHART PROGRAM NUMBER:                          0
   ENTER:  0 FOR 160600         3 FOR 160601 (LL mode)
           1 FOR 162010
           2 FOR 162011


INTERNAL CONTROL CHART BATCH NUMBER:                            4



MEASURED CONTROL VALUE, CORRECTED FOR DECAY:            1032.59 DPM/ML

UPPER CONTROL LIMIT IN UNITS OF DPM/ML:                 1073.70

LOWER CONTROL LIMIT IN UNITS OF DPM/ML:                  783.70

*ACTIVITY OF THE SAMPLE THAT WAS DUPLICATED:                 NA   (pCi/g)

*ACTIVITY OF THE SAMPLE THAT WAS SPIKED:              9.08e+00   (pCi/g)

   NA       WAS DUPLICATED, PERCENT DIFFERENCE:              NA        ERR

970815-002   WAS SPIKED, PERCENT RECOVERY:                98.74 %

KNOWN ACTIVITY OF CONTROL, DECAY CORRECTED:             1023.56 DPM/ML
   STANDARD:# R1-128-W
           REFERENCE DATE:        01-JAN-1987



E

           AnaLIS report:
                                 ACTUAL    AMOUNT      SPIKE     PERCENT
                        UNITS    RESULT    SPIKED      RESULT    RECOVERY
                       ========  ========  ========  ========  ========
                          DPM        1.41    511.78    506.72     98.74


20/20 License #4828, Program validation #:SOP160063.R4-T2LG, 09/28/94 DFH
```

DATE COUNTED:29-AUG-1997
DATE CALCULATED:02-SEP-1997
QA FILE-PE-1141
ANALYST:DKM/SPW

GILSON
Tc-99 (TevaSpec)

SAMPLE NUMBER	MD or MS	EFFIC. FACT. CPM/DPM	tSIE VALUE	GROSS ACTIVITY (CPM)	DILUTION FACTOR (ml)	WEIGHT FRACTION (g)	NET ACTIVITY (pCi/L)	NET ACTIVITY (pCi/g)	COUNTING CERTAINTY (+/-)
BLANK		0.930	284.73	19.30					
CONTROL		0.936	282.78	502.78	0.500	0.000	4.61e+05	NA	5.70e+03
NNDUPLICATE		NA	NA	NA	0.000	0.000	NA	NA	NA
SPIKE		0.934	276.70	493.58	0.000	0.070	NA	3.24e+03	4.04e+01
NN NA	MD	NA	NA	NA	0.000	0.000	NA	NA	NA
970815-002	MS	0.935	277.82	23.52	0.000	0.070	NA	3.81e+00	1.21e+01

98.30%
Spike
recovery

```
                                                        DATE COUNTED:29-AUG-1997
                                                     DATE CALCULATED:02-SEP-1997
                                                            QA FILE-PE-1141
                                                            ANALYST:DKM/SPW


                                  QA DATA SHEET
                                  =============


SAMPLE COUNTING TIME IN UNITS OF MINUTES:                    60.0

PACKARD 2550TR LIQ. SCINTILLATION COUNTER #s 1, 2, OR 3:        2

DESIRED ACTIVITY UNITS (1=pCi, 2=uCi, 3=dpm):                    1

DESIRED WEIGHT FRACTION UNITS (1=g, 2=filter):                   1

INTERNAL CONTROL CHART PROGRAM NUMBER:                           0
    ENTER:  0 FOR 160600          3 FOR 160601 (LL mode)
            1 FOR 162010
            2 FOR 162011


INTERNAL CONTROL CHART BATCH NUMBER:                             4



MEASURED CONTROL VALUE, CORRECTED FOR DECAY:               1032.59 DPM/ML

UPPER CONTROL LIMIT IN UNITS OF DPM/ML:                    1073.70

LOWER CONTROL LIMIT IN UNITS OF DPM/ML:                     783.70

*ACTIVITY OF THE SAMPLE THAT WAS DUPLICATED:                    NA   (pCi/g)

*ACTIVITY OF THE SAMPLE THAT WAS SPIKED:                  3.81e+00   (pCi/g)

    NA       WAS DUPLICATED, PERCENT DIFFERENCE:                NA        ERR

970815-002   WAS SPIKED, PERCENT RECOVERY:                   98.30 %

KNOWN ACTIVITY OF CONTROL, DECAY CORRECTED:                1023.56 DPM/ML
   STANDARD:# R1-128-W
            REFERENCE DATE:          01-JAN-1987


E

           AnaLIS report:
                                 ACTUAL    AMOUNT     SPIKE     PERCENT
                        UNITS    RESULT    SPIKED     RESULT    RECOVERY
                      ======== ======== ======== ======== ========
                         DPM       0.59    511.78    503.68      98.30


20/20 License #4828, Program validation #:SOP160063.R4-T2LG, 09/28/94 DFH
```

DATE COUNTED:29-AUG-1997
DATE CALCULATED:02-SEP-1997
QA FILE-PE-1141
ANALYST:DKM/SPW

MANUAL
Tc-99 (TevaSpec)
================

```
 SAMPLE     MD EFFIC.  tSIE   GROSS    DILUTION  WEIGHT      NET       NET     COUNTING
 NUMBER     or FACT.   VALUE  ACTIVITY  FACTOR   FRACTION  ACTIVITY  ACTIVITY  CERTAINTY
            MS CPM/DPM         (CPM)     (ml)      (g)      (pCi/L)   (pCi/g)    (+/-)
 ========= == ====== =====  ========  ========  =========  =========  ========= ========
 BLANK          0.937 284.73    19.30
 CONTROL        0.936 282.78   502.78     0.500      0.000   4.62e+05        NA  5.70e+03
NNDUPLICATE        NA     NA       NA     0.000      0.000         NA        NA        NA
 SPIKE          0.936 281.04   512.97     0.000      0.069         NA  3.43e+03  4.19e+01
NN    NA    MD     NA     NA       NA     0.000      0.000         NA        NA        NA
 970815-002MS   0.937 284.78    23.75     0.000      0.069         NA  4.29e+00  1.24e+01
```

102.16 % Spike
recovery

```
                                                        DATE COUNTED:29-AUG-1997
                                                     DATE CALCULATED:02-SEP-1997
                                                             QA FILE-PE-1141
                                                             ANALYST:DKM/SPW


                                  QA DATA SHEET
                                  =============

SAMPLE COUNTING TIME IN UNITS OF MINUTES:                      60.0

PACKARD 2550TR LIQ. SCINTILLATION COUNTER #s 1, 2, OR 3:          2

DESIRED ACTIVITY UNITS (1=pCi, 2=uCi, 3=dpm):                     1

DESIRED WEIGHT FRACTION UNITS (1=g, 2=filter):                    1

INTERNAL CONTROL CHART PROGRAM NUMBER:                            0
   ENTER:  0 FOR 160600          3 FOR 160601 (LL mode)
           1 FOR 162010
           2 FOR 162011


INTERNAL CONTROL CHART BATCH NUMBER:                              4



MEASURED CONTROL VALUE, CORRECTED FOR DECAY:                1032.85 DPM/ML

UPPER CONTROL LIMIT IN UNITS OF DPM/ML:                     1073.70

LOWER CONTROL LIMIT IN UNITS OF DPM/ML:                      783.70

*ACTIVITY OF THE SAMPLE THAT WAS DUPLICATED:                      NA   (pCi/g)

*ACTIVITY OF THE SAMPLE THAT WAS SPIKED:                   4.29e+00   (pCi/g)

   NA       WAS DUPLICATED, PERCENT DIFFERENCE:                   NA          ERR

970815-002  WAS SPIKED, PERCENT RECOVERY:                    102.16 %

KNOWN ACTIVITY OF CONTROL, DECAY CORRECTED:                 1023.56 DPM/ML
  STANDARD:# R1-128-W
           REFERENCE DATE:          01-JAN-1987


E

            AnaLIS report:
                                     ACTUAL    AMOUNT      SPIKE     PERCENT
                          UNITS      RESULT    SPIKED      RESULT    RECOVERY
                        ========   ========   ========   ========   ========
                          DPM          0.66     511.78     523.48     102.16

20/20 License #4828, Program validation #:SOP160063.R4-T2LG, 09/28/94 DFH
```

DATE COUNTED:29-AUG-1997
DATE CALCULATED:02-SEP-1997
QA FILE-PE-1141
ANALYST:DKM/SPW

```
                                        MANUAL
                                  Tc-99 (TevaSpec)
                                  ================

                                                                                  NET       COUNTING
                MD EFFIC.  tSIE    GROSS     DILUTION   WEIGHT      NET          ACTIVITY   CERTAINTY
  SAMPLE        or FACT.   VALUE   ACTIVITY  FACTOR     FRACTION    ACTIVITY     (pCi/g)    (+/-)
  NUMBER        MS CPM/DPM          (CPM)      (ml)       (g)        (pCi/L)     =========  ========
                                   ========  ========   =========   =========
  =========  == ======  =====         19.30
  BLANK           0.937 284.73       502.78     0.500       0.000   4.62e+05          NA  5.70e+03
   CONTROL        0.936 282.78           NA     0.000       0.000         NA          NA        NA
NNDUPLICATE          NA     NA       504.75     0.000       0.069         NA    3.37e+03  4.15e+01
   SPIKE          0.936 281.54           NA     0.000       0.000         NA          NA        NA
NN      NA     MD    NA     NA        24.33     0.000       0.069         NA    7.59e+00  1.25e+01
  970815-002MS    0.937 289.21
```

100.33% spike recovery

```
                                                           DATE COUNTED:29-AUG-1997
                                                        DATE CALCULATED:02-SEP-1997
                                                                QA FILE-PE-1141
                                                                ANALYST:DKM/SPW

                                     Manual
                                  QA DATA SHEET
                                  =============

SAMPLE COUNTING TIME IN UNITS OF MINUTES:                         60.0

PACKARD 2550TR LIQ. SCINTILLATION COUNTER #s 1, 2, OR 3:           2

DESIRED ACTIVITY UNITS (1=pCi, 2=uCi, 3=dpm):                      1

DESIRED WEIGHT FRACTION UNITS (1=g, 2=filter):                     1

INTERNAL CONTROL CHART PROGRAM NUMBER:                             0
   ENTER:  0 FOR 160600          3 FOR 160601 (LL mode)
           1 FOR 162010
           2 FOR 162011


INTERNAL CONTROL CHART BATCH NUMBER:                               4



MEASURED CONTROL VALUE, CORRECTED FOR DECAY:                 1032.85 DPM/ML

UPPER CONTROL LIMIT IN UNITS OF DPM/ML:                      1073.70

LOWER CONTROL LIMIT IN UNITS OF DPM/ML:                       783.70

*ACTIVITY OF THE SAMPLE THAT WAS DUPLICATED:                       NA    (pCi/g)

*ACTIVITY OF THE SAMPLE THAT WAS SPIKED:                    7.59e+00    (pCi/g)

    NA        WAS DUPLICATED, PERCENT DIFFERENCE:                  NA        ERR  /

970815-002    WAS SPIKED, PERCENT RECOVERY:                   100.33 %

KNOWN ACTIVITY OF CONTROL, DECAY CORRECTED:                  1023.56 DPM/ML
   STANDARD:# R1-128-W
            REFERENCE DATE:          01-JAN-1987


E

            AnaLIS report:
                                 ACTUAL    AMOUNT      SPIKE     PERCENT
                       UNITS     RESULT    SPIKED      RESULT    RECOVERY
                      ========  ========  ========   ========   ========
                        DPM        1.16     511.78     514.63     100.33

20/20 License #4828, Program validation #:SOP160063.R4-T2LG, 09/28/94 DFH
```

```
                                                                  Friday, September 12, 1997, 09:36:52
Descriptive Statistics:

Column   Size  Missing      Mean        Std Dev        Std. Error   Range     Max       Min
Col 1    4     0            6.192       2.555          1.278        5.270     9.080     3.810
Col 2    4     0            12.300      0.183          0.0913       0.400     12.500    12.100

Column   Median   25%       75%         Sum          Sum of Squares ConfidenceSkewness  Kurtosis
Col 1    5.940    4.050     8.335       24.770         172.975      4.066     0.256     -4.221
Col 2    12.300   12.150    12.450      49.200         605.260      0.291     2.916E-014-3.300

ColumnK-S Distance          P Value
Col 1    0.272    0.337
Col 2    0.208    0.606
```

```
Descriptive Statistics:                              Friday, September 12, 1997, 09:40:12

Column   Size  Missing    Mean      Std Dev      Std. Error   Range     Max      Min
Col 1     2      0       31.200      4.384         3.100      6.200    34.300   28.100
Col 2     2      0        8.940      0.0566        0.0400     0.0800    8.980    8.900

Column  Median   25%      75%       Sum       Sum of Squares ConfidenceSkewness  Kurtosis
Col 1   31.200  28.100   34.300    62.400      1966.100     39.389 undefinedundefined
Col 2    8.940   8.900    8.980    17.880       159.850      0.508 undefinedundefined

ColumnK-S Distance       P Value
Col 1     0.260   0.481
Col 2     0.260   0.481
```

```
                                                                   Friday, September 12, 1997, 09:40:12
Descriptive Statistics:

Column   Size  Missing     Mean      Std Dev        Std. Error   Range     Max      Min
Col 1      2      0       31.200      4.384           3.100      6.200    34.300   28.100
Col 2      2      0        8.940      0.0566          0.0400     0.0800    8.980    8.900

Column   Median   25%       75%       Sum        Sum of Squares ConfidenceSkewness  Kurtosis
Col 1    31.200  28.100    34.300    62.400       1966.100      39.389 undefinedundefined
Col 2     8.940   8.900     8.980    17.880        159.850       0.508 undefinedundefined

ColumnK-S Distance          P Value
Col 1       0.260    0.481
Col 2       0.260    0.481
```

Manual:

For Chris

LMES, Inc.
Lockheed Martin Energy Systems, Inc.
Analytical Services Organization

Nuclear Measurements
K-25 Plant M.S.-7458 Oak Ridge, TN. 37830
(615) 574-7584

RADIOCHEMICAL ANALYSIS

LABORATORY PREPARATION SHEET FOR LIQUID SCINTILLATION COUNTING

Prep.# (Circle): [✓ 1] 2 3 Count Time: 60 min.

QA File #: PE-1141

Project/Analysis: DEV / Tc

Ct.Room Rec'd: 8/29/97 by KAC

Count Date: 8/29/97 by KAC

Instr.#/S.#/Protocol#: 2/1039991/3

Package Deadline: ____/____/____

Prep.Date/Analyst: 8/28/97 by DCM

Control Number: 160600

Pipette ID: ______ Daily Check *by*: ______

Microwave Filename: ______

Procedure Number: ______

**Scales ID#:* Sample ID:	**PREP (Circle): Leach Ash Direct HDV Prolabo* Complete Prep. Dilution (*Indicate units: g, fil, ml*)	Cassette# (PID)	Sample Position#
Blank 970815-002B	40 ml 1M HNO_3	25	2
Control (970815-002C	40 ml 1M HNO_3 + 0.5 ml Tc-99		3
Duplicate 970815 002A	0.2749g x 10 ml/40 ml x 10 ml/40 ml		4
Spike 970815-002B	+ 0.5 ml Tc-99		5
Sample 970815-002C			6
Sample 970815-002D	+ 0.5 ml Tc-99		7
INST STD	#15		1

Comments: ______

SUPERVISOR APPROVAL: ______

Gilson Run.

LMES, inc.
Lockheed Martin Energy Systems, Inc.
Analytical Services Organization

Nuclear Measurements
K-25 Plant M.S.-7454 Oak Ridge, TN 37830
(615) 574-7384

RADIOCHEMICAL ANALYSIS

LABORATORY PREPARATION SHEET FOR LIQUID SCINTILLATION COUNTING

Prep.# (Circle) [✓ 1] 2 3 Count Time: 60 min.

QA File #: TE-1141

Project/Analysis: ______/______

Ct.Room Rec'd: ___/___/___ by______

Count Date: ___/___/___ by______

Instr.#/S.#/Protocol#: ___/______/___

Package Deadline: ______/______/______

Prep.Date/Analyst: ___/___/___ by______

Control Number: 16

Pipette ID: ________ Daily Check *by*: ______

Microwave Filename: ____________

Procedure Number: ____________

**Scales ID#:* Sample ID:	**PREP (Circle): Leach Ash Direct HDV Prolabo* Complete Prep. Dilution (*Indicate units: g, fil, ml*)	Cassette# (PID)	Sample Position#
Blank 970815-Blk	40ml 1m HNO$_3$		
Control (970815-Con)	40ml 1m HNO$_3$ + 0.5ml Tc-99		
~~Duplicate 9~~ -			
~~Spike 9~~ -			
970815-001A	0.2801g x 10ml / 40ml x 10ml / 40ml		
970815-001B	↓ +0.5ml Tc-99		
970815-001C	↓		
970815-001D	+0.5ml Tc-99		

Comments: ______________________________

SUPERVISOR APPROVAL: ______________

PB-1141

```
29 Aug 97  20:17                                                     Page #1
Protocol #: 3                 Tc-99/Teva UG                User : K25 Radiochemistr

Time:  60.00
Data Mode: DPM                      Nuclide: TC_TEVA_UG      Quench Set: TC_TEVA
Background Subtract: Manual

               LL       UL      LCR      2S%       BKG
Region A:     0.0 -   293        0       0.0      0.00
Region B:     6.0 -   293        0       0.0      0.00
Region C:     293 -  2000        0       0.0      0.00

Quench Indicator: tSIE/AEC
   Ext Std Terminator: Count

Packard Liquid Scintillation 2550TR-#2  SN 103999
Luminescence Correction On
Heterogeneity Monitor On
Color Quench Correction On


 P# PID  S#   TIME    CPMA     DPM1     CPMB     SIS    tSIE      EFF.  FLAG  LUM
  3  25   1  60.00 2431.38  2595.97  2406.70  152.34  284.90  0.936599          0 Ind
  3  25   2  60.00   19.30    20.61    17.85  70.471  284.73  0.936573 chris' 18 Bk
  3  25   3  60.00  502.78   537.02   490.12  87.415  282.78  0.936239 donald  1 Cnd
  3  25   4  60.00   24.33    25.97    22.53  72.983  289.21  0.937122       {12-002
  3  25   5  60.00  504.75   539.27   491.00  86.254  281.54  0.935996 002A  { 1-002
                                                                        set
  3  25   6  60.00   23.75    25.36    22.17  77.440  284.78  0.936580         10-002
  3  25   7  60.00  512.97   548.10   501.22  88.418  281.04  0.935892          1-002
                                                                  - - - - - - - - - -
  3  25   8  60.00   18.65    20.06 ←  17.43  72.708  268.35  0.929806          9-Blk
  3  25   9  60.00  494.68   528.24   481.85  87.242  284.09  0.936470          0 Con
  3  25  10  60.00   24.25    25.96    22.55  81.394  276.44  0.934274 Gilson   5-00
  3  25  11  60.00  496.97   531.30   484.88  84.414  278.84  0.935387          0-00
  3  25  12  60.00   23.52    25.15    22.37  77.881  277.82  0.934974          6-00
  3   2  13  60.00  493.58   528.23   481.28  84.241  276.70  0.934408          0-00
```

Developmental

```
                                                                     Page #1
07 Aug 97  16:02                  TOTAL ACTIVITY AEC          User : K-25 Site Radiochem.
Protocol #:10

Time:  20.00                           Nuclide: TOTACT UG          Quench Set: TOTAL UG
Data Mode: DPM
Background Subtract: None

               LL       UL      LCR      2S%       BKG
Region A:     0.0 -  2000        0       0.0       0.00
Region B:     6.0 -   293        0       0.0       0.00
Region C:     293 -  2000        0       0.0       0.00

Quench Indicator: tSIE/AEC
   Ext Std Terminator: Count

Packard Liquid Scintillation Counter 2550 #2
Luminescence Correction On
Heterogeneity Monitor On
Color Quench Correction On

 P#  PID   S#   TIME      CPMA      CPMB      CPMC      DPM1   tSIE    EFF  FLAG   LUM
 10   28   1   20.00     26.55     16.60      7.15     28.08  225.77  .946          59 B
 10   28   2   20.00     24.65     16.60      5.80     26.08  232.77  .945          20 N:metal
 10   28   3   20.00     29.35     16.85      6.55     31.88  149.71  .921          54 4MD:1
 10   28   4   20.00     25.20     15.35      7.55     26.74  212.35  .942          30 1MD:1 (1)
 10   28   5   20.00     25.35     17.35      7.00     26.88  214.62  .943          28 1MD:1(2)
 10   28   6   20.00     26.20     16.75      6.90     27.78  214.96  .943          26 1MD:1(3)
```

2nd
Run
Gilson

10/27/97
DF

DATE COUNTED:02-SEP-1997
DATE CALCULATED:04-SEP-1997
QA FILE-PE-1141
ANALYST:DKM/SPW

Tc-99 (TevaSpec)

SAMPLE NUMBER	MD or MS	EFFIC. FACT. CPM/DPM	tSIE VALUE	GROSS ACTIVITY (CPM)	DILUTION FACTOR (ml)	WEIGHT FRACTION (g)	NET ACTIVITY (pCi/L)	NET ACTIVITY (pCi/g)	COUNTING CERTAINTY (+/-)
BLANK		0.924	271.07	22.50					
CONTROL		0.931	290.30	495.02	0.500	0.000	4.57e+05	NA	4.02e+03
NNDUPLICATE		NA	NA	NA	0.000	NA	NA	NA	NA
SPIKE		0.929	284.06	501.83	0.000	0.070	NA	3.32e+03	2.89e+01
NN970815-002MD		NA	NA	NA	NA	NA	NA	NA	NA
970815-002MS		0.929	283.09	26.80	0.000	0.070	NA	2.81e+01	8.90e+00

DATE COUNTED:02-SEP-1997
DATE CALCULATED:04-SEP-1997
QA FILE-PE-1141
ANALYST:DKM/SPW

QA DATA SHEET

SAMPLE COUNTING TIME IN UNITS OF MINUTES: 120.0

PACKARD 2550TR LIQ. SCINTILLATION COUNTER #s 1, 2, OR 3: 3

DESIRED ACTIVITY UNITS (1=pCi, 2=uCi, 3=dpm): 1

DESIRED WEIGHT FRACTION UNITS (1=g, 2=filter): 1

INTERNAL CONTROL CHART PROGRAM NUMBER: 0
ENTER: 0 FOR 160600 3 FOR 160601 (LL mode)
1 FOR 162010
2 FOR 162011

INTERNAL CONTROL CHART BATCH NUMBER: 4

MEASURED CONTROL VALUE, CORRECTED FOR DECAY: 1015.12 DPM/ML

UPPER CONTROL LIMIT IN UNITS OF DPM/ML: 1073.70

LOWER CONTROL LIMIT IN UNITS OF DPM/ML: 783.70

*ACTIVITY OF THE SAMPLE THAT WAS DUPLICATED: NA (pCi/g)

*ACTIVITY OF THE SAMPLE THAT WAS SPIKED: 2.81e+01 (pCi/g)

970815-002 WAS DUPLICATED, PERCENT DIFFERENCE: NA ERR

970815-002 WAS SPIKED, PERCENT RECOVERY: 99.89 %

KNOWN ACTIVITY OF CONTROL, DECAY CORRECTED: 1023.56 DPM/ML
STANDARD:# R1-128-W
REFERENCE DATE: 01-JAN-1987

E

AnaLIS report:

UNITS	ACTUAL RESULT	AMOUNT SPIKED	SPIKE RESULT	PERCENT RECOVERY
DPM	4.37	511.78	515.57	99.89

20/20 License #4828, Program validation #:SOP160063.R4-T2LG, 09/28/94 DFH

DATE COUNTED:02-SEP-1997
DATE CALCULATED:04-SEP-1997
QA FILE-PE-1141
ANALYST:DKM/SPW

Tc-99 (TevaSpec)

SAMPLE NUMBER	MD or MS	EFFIC. FACT. CPM/DPM	tSIE VALUE	GROSS ACTIVITY (CPM)	DILUTION FACTOR (ml)	WEIGHT FRACTION (g)	NET ACTIVITY (pCi/L)	NET ACTIVITY (pCi/g)	COUNTING CERTAINTY (+/-)
BLANK		0.924	271.07	22.50					
CONTROL		0.931	290.30	495.02	0.500	0.000	4.57e+05	NA	4.02e+03
NNDUPLICATE		NA	NA	NA	0.000	0.000	NA	NA	NA
SPIKE		0.929	284.09	499.15	0.000	0.070	NA	3.30e+03	2.89e+01
NN NA	MD	NA	NA	NA	0.000	0.070	NA	NA	NA
970815-002	MS	0.928	280.53	27.64	0.000	0.070	NA	3.43e+01	8.98e+00

```
                                                      DATE COUNTED:02-SEP-1997
                                                   DATE CALCULATED:04-SEP-1997
                                                           QA FILE-PE-1141
                                                           ANALYST:DKM/SPW



                                 QA DATA SHEET
                                 =============

 SAMPLE COUNTING TIME IN UNITS OF MINUTES:                     120.0

 PACKARD 2550TR LIQ. SCINTILLATION COUNTER #s 1, 2, OR 3:        3

 DESIRED ACTIVITY UNITS (1=pCi, 2=uCi, 3=dpm):                   1

 DESIRED WEIGHT FRACTION UNITS (1=g, 2=filter):                  1

 INTERNAL CONTROL CHART PROGRAM NUMBER:                          0
     ENTER:  0 FOR 160600          3 FOR 160601 (LL mode)
             1 FOR 162010
             2 FOR 162011


 INTERNAL CONTROL CHART BATCH NUMBER:                            4



 MEASURED CONTROL VALUE, CORRECTED FOR DECAY:                1015.12 DPM/ML

 UPPER CONTROL LIMIT IN UNITS OF DPM/ML:                     1073.70

 LOWER CONTROL LIMIT IN UNITS OF DPM/ML:                      783.70

*ACTIVITY OF THE SAMPLE THAT WAS DUPLICATED:                      NA   (pCi/g)

*ACTIVITY OF THE SAMPLE THAT WAS SPIKED:                    3.43e+01   (pCi/g)

    NA       WAS DUPLICATED, PERCENT DIFFERENCE:                  NA        ERR

 970815-002  WAS SPIKED, PERCENT RECOVERY:                     99.13 %

 KNOWN ACTIVITY OF CONTROL, DECAY CORRECTED:                 1023.56 DPM/ML
    STANDARD:# R1-128-W
             REFERENCE DATE:        01-JAN-1987


 E

          AnaLIS report:
                                   ACTUAL    AMOUNT      SPIKE      PERCENT
                         UNITS     RESULT    SPIKED      RESULT     RECOVERY
                        ========  ========  ========   ========   ========
                          DPM        5.33     511.78     512.69      99.13

 20/20 License #4828, Program validation #:SOP160063.R4-T2LG, 09/28/94 DFH
```

DATE COUNTED:02-SEP-1997
DATE CALCULATED:04-SEP-1997
QA FILE-PE-1141
ANALYST:DKM/SPW

Tc-99 (TevaSpec)

SAMPLE NUMBER	MD or MS	EFFIC. FACT. CPM/DPM	tSIE VALUE	GROSS ACTIVITY (CPM)	DILUTION FACTOR (ml)	WEIGHT FRACTION (g)	NET ACTIVITY (pCi/L)	NET ACTIVITY (pCi/g)	COUNTING CERTAINTY (+/-)
BLANK		0.924	271.07	22.50					
CONTROL		0.931	290.30	495.02	0.500	0.000	4.57e+05	NA	4.02e+03
DUPLICATE		0.929	283.09	26.80	0.000	0.070	NA	2.81e+01	8.90e+00
SPIKE		0.929	284.09	499.15	0.000	0.070	NA	3.30e+03	2.89e+01
970815-002	MD	0.928	280.53	27.64	0.000	0.070	NA	3.43e+01	8.98e+00
970815-002	MS	0.928	280.53	27.64	0.000	0.070	NA	3.43e+01	8.98e+00

DATE COUNTED:02-SEP-1997
DATE CALCULATED:04-SEP-1997
QA FILE-PE-1141
ANALYST:DKM/SPW

QA DATA SHEET

SAMPLE COUNTING TIME IN UNITS OF MINUTES:	120.0
PACKARD 2550TR LIQ. SCINTILLATION COUNTER #s 1, 2, OR 3:	3
DESIRED ACTIVITY UNITS (1=pCi, 2=uCi, 3=dpm):	1
DESIRED WEIGHT FRACTION UNITS (1=g, 2=filter):	1
INTERNAL CONTROL CHART PROGRAM NUMBER: ENTER: 0 FOR 160600 1 FOR 162010 2 FOR 162011 3 FOR 160601 (LL mode)	0
INTERNAL CONTROL CHART BATCH NUMBER:	4
MEASURED CONTROL VALUE, CORRECTED FOR DECAY:	1015.12 DPM/ML
UPPER CONTROL LIMIT IN UNITS OF DPM/ML:	1073.70
LOWER CONTROL LIMIT IN UNITS OF DPM/ML:	783.70
*ACTIVITY OF THE SAMPLE THAT WAS DUPLICATED:	3.43e+01 (pCi/g)
*ACTIVITY OF THE SAMPLE THAT WAS SPIKED:	3.43e+01 (pCi/g)
970815-002 WAS DUPLICATED, PERCENT DIFFERENCE:	19.86 %
970815-002 WAS SPIKED, PERCENT RECOVERY:	99.13 %
KNOWN ACTIVITY OF CONTROL, DECAY CORRECTED: STANDARD:# R1-128-W REFERENCE DATE: 01-JAN-1987	1023.56 DPM/ML

AnaLIS report:

UNITS	ACTUAL RESULT	AMOUNT SPIKED	SPIKE RESULT	PERCENT RECOVERY
DPM	5.33	511.78	512.69	99.13

20/20 License #4828, Program validation #:SOP160063.R4-T2LG, 09/28/94 DFH

Gilson

```
02 Sep 97  18:19             ALPHA/BETA - 1.21                              Page #1
Protocol #: 4                 Tc-99/TEVA UG               User : K-25 Radiochemistry

Time: 120.00
Data Mode: DPM                  Nuclide: TC_TEVA_UG     Quench Set: TC_TEVA
Background Subtract: Manual

               LL      UL     LCR    2S%     BKG
Region A:     0.0 -   293      0     0.0     0.00
Region B:     6.0 -   293      0     0.0     0.00
Region C:     293 -  2000      0     0.0     0.00

Quench Indicator: tSIE/AEC
   Ext Std Terminator: Count
Packard Liquid Scintillation 2550/TR 402234
Luminescence Correction On
Heterogeneity Monitor On
Color Quench Correction On
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

  P#   S# PID   TIME     CPMA     DPM1     CPMB     CPMC    tSIE     EFF FLAG   LUM
   4    1  27 120.00    22.50    24.35    21.00     9.74 271.07 0.9239D        12
   4    2  27 120.00   495.02   531.72   483.89    10.11 290.30 0.9310 C        1
   4    3  27 120.00    27.64    29.79    25.52     9.53 280.53 0.9278A         8
   4    4  27 120.00   499.15   537.19   488.51    10.22 284.09 0.9292B -spike  0
   4    5  27 120.00    26.80    28.85    25.10    10.08 283.09 0.9288 C        7
   4    6  27 120.00   501.83   540.09   490.38     9.65 284.06 0.9292 D -spike  0
```

ICP/MS Data

.2609 ×

[15]

115

Analysis of ^{99}Tc in Ni Metal

9/22/97 Specific Activity ^{99}Tc = 2.669×10^{-5} ug/dpm

Blank Mean = 48.89 dpm/L spike 952.47 dpm/L

S_B = 2.214 dpm/L

$\text{Yield} = \frac{952.47 - 48.89}{942.63} \times 100 = 95.86$

Result 48.89/0.9586 = 51.00 dpm/L

MDA = 4.65(2.21)/0.9586 = 10.72 dpm/L

* Control (I forgot to add Control to the Control spi

Yield = 911.67/942.63 *100 = 96.72 %

Result = 181.76/0.9672 = 187.93 dpm/L

True Value = 187.15 dpm/L

% Bias = 187.93 − 187.15 / 187.15 ×100 = +0.42

MDA = 4.65(221)/0.9672 = 10.63 dpm/L

Sample $\text{Yield} = \frac{1091.35 - 250.65}{942.63} \times 100 = 89.19\%$

Result = 250.65/0.8919 (2) = 562.08 dpm/L

36.73 % 1.00 pCi/gm

= 562.08 dpm/L (2.669×10^{-5} ug/dpm)(0.0049/0.2251

= 0.002176 ug/g = 2.176 ppb

Error = 15.33 dpm/L (2.669×10^{-5} ug/dpm)(0.094 L/0.22

= 0.00005936 ug/g = 0.05936 ppb

$\text{MDA} = \left(\frac{4.65(2.21)}{0.8919}\right)\left(2.669 \times 10^{-5} \text{ ug/dpm}\right)\left(\frac{0.094}{0.2}\right.$

= 0.00003549 ug/g = 0.03549 ppb

0.04461 ppb

Lab

To: Valerie MacNair
Cc: Steve Sarten
Subject: Ni Results from Ni cathodes stripped from Ti Base

Sample # 002752A July 24, 1998

Ni cathode was sectioned off in 9 equal portions. Sections were counted using a Ludlum 2224 with a alpha/beta scintillation detector. The counted section was marked and a 100 cm 2 area cut from the quadrant and cut into small pieces. Twenty ml of HNO3 was added to the samples and heated until dissolved. Water was added and the solution evaporated down to 15 ml at 60 degrees C to reduce the nitric acid concentration. The sample was quantitatively rinsed into a 100 ml volumetric flash and diluted to 100 ml with distilled water. One ml of the solution was removed and added to 12 ml of a liquid scintillation cocktail and counted for 100 minutes in the Packard 2700 TR counter.

Sample #	Sample weight	Net CPM	Bq/g
1	3.3925	8.74	4.52
2	3.3682	10.50	5.46
3	2.5815	7.19	4.89
4	3.3963	8.20	4.23
5	3.0158	7.02	4.08
6	2.9657	8.64	5.11
7	3.4928	16.89	8.48
8	3.8090	19.16	8.82
9	3.5318	13.99	6.95
10	3.5426	9.21	4.33
			5.69 (4.66)

Sample B (from the backside of Base) Sampled and counted as in A.
002752B July 29,1998

Sample #	Sample Weight	Net CPM	Bq/g
1	3.7675	8.01	3.73
2	2.9325	8.60	5.15
3	3.6843	9.31	4.43
4	3.0726	8.62	4.68
5	3.3547	9.14	4.54
6	3.3039	8.02	4.05
7	3.7473	8.75	3.89
8	3.1548	7.34	4.97
9	2.6881	8.10	5.29
10	4.1284	11.69	4.97
			4.46
Cementation 1	3.1269	1,852	2,306
Cementation 2 (a) 1.00 ml sample	2.8363	1,219	1,674
Cementation 2 (b) 0.50 ml sample		617.7	1,696
Cementation 2 (c) 2.00 ml		2,461	1690

Tc-99 Results from Ni Cathode: Lab. # 002752A July 23, 1998

Model 2224 alpha/beta scintillator 100 cm2 Area
SN 125601 Cal. Date : 12-11-97
Alpha eff. : 18.5% Beta eff. : 11.4%

Area #											
1	259	251	246	274	238	217	232	233	247	246	244.3
2	245	225	252	247	231	247	229	240242		217	237.5
3	243	261	250	253	225	256	238	241	276	240	248.3
4	257	240	220	234	260	233	232	240	242	220	237.8
5	210	233	236	245	265	268	243	254	226	242	242.2
6	234	249	227	260	244	255	264	239	247	232	245.1
7	216	250	251	246	269	252	237	246	238	250	245.5
8	227	241	270	236	247	247	244	259	244	254	246.9
9	244	229	236	263	256	242	262	229	233	251	244.5
								AVERAGE			243.6
Bkg.	231	251	240	223	219	245	212	256	236	235	237.9
	230	262	254	240	231	254	234	246	227	231	

Net CPM	5.7
DPM	50.0
Bq	0.83
Bq/gram	0.37

Tc-99 Results from Ni Cathode: Lab. # 002752~~8~~B July 23, 1998

Model 2224 alpha/beta scintillator 100 cm2 Area
SN 146728 Cal. Date : 4-24-98
Alpha eff. : 20.6% Beta eff. : 13.0%

Area #											
1	397	444	412	414	425	402	419	383	427	418	413.2
2	436	426	396	419	408	410	392	390	425	444	413.7
3	420	392	368	442	457	401	419	422	397	379	408.9
4	402	417	396	366	413	385	434	401	420	420	404.5
5	404	404	423	416	414	437	366	402	400	419	407.4
6	375	435	396	442	395	427	409	418	394	399	408.1
7	386	374	421	404	393	432	429	381	403	411	402.7
8	394	408	404	435	443	400	390	363	387	426	404.2
9	392	418	406	411	390	426	441	401	393	399	407.0
								AVERAGE			407.7
Bkg.	335	317	342	360	338	334	347	312	305	276	326.6

Net CPM	81.1
DPM	624.2
Bq	10.40
Bq/gram	4.66

Tc-99 Results from Ni Cathode: Lab. # 002858A July 30, 1998
Thicker Material

Model 2224 alpha/beta scintillator 100 cm2 Area
SN 125578 Cal. Date : 5-26-98
Alpha eff. : 20.1% Beta eff. : 9.1%

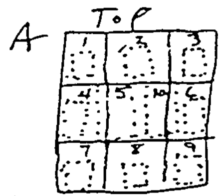
Area #											
1	707	715	760	689	698	745	687	699	702	633	702.8
2	755	768	724	690	723	746	714	756	741	707	737.4
3	688	641	661	670	646	636	655	649	679	672	659.7
4	771	743	753	718	760	739	710	726	755	751	742.6
5	746	713	765	775	769	724	746	744	718	760	746.0
6	695	698	670	644	689	709	707	702	687	689	689.0
7	772	799	771	768	820	738	765	806	800	779	781.8
8	762	769	720	770	751	771	777	751	768	737	757.6
9	7478	787	768	738	763	764	776	784	765	782	767.4
								AVERAGE			732.0
Bkg.	290	271	289	268	242	279	240	274	273	259	270.0
	242	271	282	260	281	258	285	278	276	275	
									Net CPM		462.0
									DPM		5,076.9
									Bq		84.6
									Bq/gram		37.9

2.981 g from cathode dissolved in 20 ml HNO3: Diluted to 100 ml, 1 ml counted in LSC.

Sample 1: 30 net cpm Sample 27.8 net cpm Background = 21.5 cpm
Ave: 28.9 net cpm or 30 dpm/ml = 1,020 dpm/g or 17.0Bq/g.
Anolyte= 60.4 net cpm = 64 dpm = 1.06 Bq/ml Catholyte = 14.7 cpm = 15 dpm = 0.25 Bq/ml

002752 A

7-24-98



Ni Anodes stripped from Ti Base.

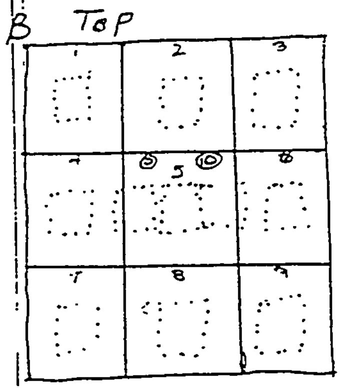
100 cm^2 section removed from quadrant and cut into small pieces. 20 ml HNO_3 added to sample and heated until dissolved. H_2O added and evaporated down to ~15 ml. Quantitatively rinsed into 100 ml volumetric flask and diluted to 100 ml with dist. H_2O. 1 ml removed and added to LSC cocktail.

Bq/g.	Weight	Net cpm
4.52	3.3925	8.74
5.46	3-3682	10.50
4.89	2.5815	7.19
4.23	3.3963	8.20
4.08	3.0158	7.02
5.11	2.9657	8.64
8.48	3.4928 ~~3.5426~~	16.89
8.82	3.8090	19.16
6.95	2.5318	13.99
4.33	3.5426	9.21

e 5.69

002752 B

7-29-



Sampled As Above - Analyzed as Above

	Weight	Net c/m	Bq/g
1	3.7675	8.01	3.73
2	2.9325	8.60	5.15
3	3.6843	9.31	4.43
4	3.0726	8.62	4.68
5	3.3547	9.14	4.54
6	3.3039	8.02	4.05
7	3.7473	8.75	3.89
8	3.1548	7.34	3.88
9	4.1284	11.69	4.97

		Net c/m	Bq/
Cementation 1	3.1269	1,852	2,30
Cementation 2	(a) 2.8363 (1ml)	1,219	1,67
	(b) 0.5 ml	617.7	1,69
	(c) 2.0 ml	2,461	1,69

```
24 Jul 1998  22:28          ALPHA/BETA - 1.08                           Page
Protocol #: 2               Tc-99                                  User  :  Tc-9
```

002752A

```
Time: 100.00
Data Mode: CPM                        Nuclide: MANUAL
Background Subtract: None

              LL       UL    LCR    2S%      BKG
Region A:    0.0 -    200     0     0.0      0.00
Region B:    4.0 -    200     0     0.0      0.00
Region C:    0.0 -    0.0     0     0.0      0.00

Quench Indicator: tSIE/AEC
   Ext Std Terminator: Count
Tc-99 in Ni solution
```

S#	TIME	CPMA	DPM1	SIS	FLAG	Bq/g
1	100.00	30.12		59.725	8.74	10.03
2	100.00	31.88		59.729	10.5	12.14
3	100.00	28.57		60.555	7.19	10.85
4	100.00	29.58		56.369	8.20	9.40
5	100.00	28.40		58.850	7.02	9.06
6	100.00	30.02		59.629	8.64	11.35
7	100.00	38.27		59.241	16.89	18.83
8	100.00	40.54		56.433	19.16	~~21.07~~ 19.59
9	100.00	35.37		60.557	13.99	15.58
10	100.00	30.59		57.715	9.21	9.62
11	100.00	891.06		50.716	Anolyte	12.63 Ave Bq/g
12	100.00	21.38		66.843	Bkgnd	

```
SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 27-Jul-1998 06:27
   C14 Eff (0-156 keV) = 95.99 %
H3 IPA DATA PROCESSED - 27-Jul-1998 06:28
   H3 Eff (0-18.6 keV) = 67.60 %
BKG IPA DATA PROCESSED - 27-Jul-1998 07:29
   Bkg (0-18.6 keV) = 15.43 cpm
   Bkg (0-156 keV)  = 23.50 cpm
   C14 E^2/B (1-156 keV)    = 523.08
   H3  E^2/B (1-18.6 keV)   = 302.99
```

```
29 Jul 1998  21:55          ALPHA/BETA - 1.08                      Page
Protocol #: 2               Tc-99                                  User : Tc-9
```

002752 B

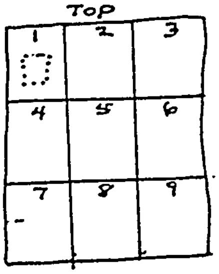
```
Time:  60.00
Data Mode: CPM                   Nuclide: MANUAL
Background Subtract: None

              LL      UL    LCR    2S%     BKG
Region A:    0.0 -   200     0     0.0    0.00
Region B:    4.0 -   200     0     0.0    0.00
Region C:    0.0 -   0.0     0     0.0    0.00

Quench Indicator: tSIE/AEC
   Ext Std Terminator: Count
Tc-99 in Ni solution
```

Weight (g)	S#	TIME	CPMA	DPM1	SIS	FLAG	
	1	60.00	22.09		66.061	BKgnd	
~~3.1296~~	2	60.00	1864.61		68.677	Com-1 1 ml	
3.1269	3	60.00	1880.37		67.654		
2.8363	4	60.00	639.01		84.384	0.5 ml	CEM-2
	5	60.00	1240.56		71.754	1.0 ml	
	6	60.00	2482.43		53.507	2.0 ml	
3.7675	7	60.00	29.27		60.415	1	
2.9325	8	60.00	29.86		59.594	2	
3.6343	9	60.00	30.57		57.166	3	
3.0726	10	60.00	29.88		57.922	4	
3.3547	11	60.00	30.40		56.436	5	002752 B
3.3039	12	60.00	29.28		56.838	6	1 ml of 100 ml added
3.7473	13	60.00	30.01		55.792	7	to LSC solution
3.1584	14	60.00	28.60		58.374	8	
[illegible].6881 ~~4.1284~~	15	60.00	29.36		56.462	9	
~~2.0551~~	16	60.00	32.95		54.734	10	
4.1284	17	60.00	20.43		63.590	BKgnd	
3.3838 g			30.02		Ave: 21.26		

Net 8.76

4.31 Bq/g

A



002752A

Model 2224 α-β⁻ Scintillator
100 cm²
SN 125601
α eff. : 18.5%
β⁻ eff. : 11.4%
Cal. Date Dec 11,'97

7-23-98

											β⁻
1.	4 259	7 251	6 246	8 274	4 238	3 217	5 232	6 233	8 247	7 246	244.3
2.	6 245	7 225	4 252	10 247	5 231	6 241	9 229	5 240	6 242	6 217	237.5
3.	3 243	8 261	4 250	5 253	2 225	5 256	6 238	6 241	7 276	6 240	248.3
4.	7 257	5 240	3 220	7 234	5 260	3 233	6 232	8 240	6 242	6 220	237.8
5.	6 210	9 233	7 236	4 245	5 265	6 268	6 243	8 254	5 226	6 242	242.2
6.	6 234	5 249	6 227	5 260	4 244	5 255	8 264	6 239	5 247	3 232	245.1
7.	1 216	10 250	6 251	6 246	5 269	6 252	2 237	4 246	8 238	8 250	245.5
8.	7 227	4 241	7 270	6 236	9 247	6 247	7 244	2 259	4 244	9 254	246.9
9.	7 244	3 229	9 236	7 263	6 256	4 242	10 262	5 229	8 233	4 251	2445
											243.6
Bkgnd	5 231	4 251	6 246	8 223	7 219	9 245	4 212	5 256	5 236	3 235	234.8
	5 230	4 262	4 254	4 240	2 231	6 254	10 234	7 246	8 227	6 231	240.9
											237.9

Net c/m : 5.7
dpm : 49.7

Top
B

1	2	3
4	5	6
7	8	9

#002752B

LUDLUM 2224
SN·146728
Cal. Date: 4-24-98
B ~~eff~~: 13.0%
α Eff: 20.6%

													B⁻ only
BKGND	29	0 335	0 317	3 345	1 361	0 338	0 334	0 347	0 312	1 306	0 276	0.5 327.1	326.6
	30												
	31	1 397	1 444	0 412	0 414	2 425	1 402	1 419	1 383	1 427	1 418	0.9 414.1	86.6
	32	0 436	1 426	1 396	4 419	1 408	1 410	0 392	0 390	1 425	1 444	0.9 414.6	87.1
	33	1 420	0 392	3 368	0 442	0 457	1 401	1 419	1 422	0 397	1 379	0.8 409.7	82.3
	34	2 402	2 417	0 396	3 366	0 413	0 385	0 434	1 401	1 420	0 420	0.9 405.4	77.9
	35	0 404	2 404	1 423	2 416	2 414	0 437	1 366	2 402	0 400	1 419	1.1 408.5	80.8
	36	1 375	1 435	0 396	2 442	0 395	1 427	1 409	2 418	0 394	1 399	0.9 409.0	81.5
	37	2 386	0 374	1 421	0 404	1 393	0 432	0 429	0 381	1 403	2 411	0.7 403.4	76.1
	38	0 394	1 408	1 404	1 435	2 443	0 400	1 390	0 363	1 387	1 426	0.8 405.0	77.6
	39	0 392	1 418	1 406	1 411	0 390	1 426	2 441	1 401	0 393		0.7 407.7	80.4
	40												
:241 1:125601 11.4 eff :18.5 eff	41	10 269	6 313	3 300	3 269	2 304	7 312	4 287	9 297	6 295	5 267	5.5 291.3	64.5
	42	7 295	8 300	5 269	3 302	3 274	5 264	6 312	4 287	7 289	5 280	5.3 287.1	
	43	4 293	6 308	7 298	7 268	6 291	3 285	4 268	3 273	3 273	4 284	4.7 284.	
	44	9 298	3 308	2 283	5 307	4 270	3 309	5 298	3 308	10 310	6 273	5.0 296.4	
	45	0 284	6 289	5 325	6 297	9 339	3 274	5 269	4 295	8 291	2 347	4.7 301.0	/
	46	4 274	5 329	3 294	5 320	4 288	2 285	0 306	4 290	4 299	3 308	3.4 299.3	
	47	4 265 ~~415~~	6 288 ~~[illegible]~~	4 305 ~~391~~	6 290	7 312	5 292	4 310	4 279	5 287	7 310	5.2 293.8	
	48	7 303	6 308	7 298	5 269	6 291	8 294	6 325	3 291	3 285	7 316	5.8 298.0	
	49	4 292	5 321	6 288	5 273	9 310	5 268	6 302	4 287	8 310	10 340	6.2 289.1	
	50												
Background		2 195	4 204	2 211	3 212	2 234	3 243	3 259	4 241	6 234	2 235	3.1 226.8	(Am)
	52	4 248	8 289	6 242	3 269	3 253	8 238	5 268	7 268	3 286	3 258	5.0 261.9	(Pm)
	53												
	54												
	55												

NOT Ⓐ

1	2	3
4	5	6
7	8	9

SN 125601
α : 18.5%
β : 11.4%

PREPARED BY 7-24-98
DATE only β

1	4 263,	7 258,	6 252,	8 282,	4 238,	3 220,	5 237,	6 239,	8 255,	7 253	5.8 249.7	243.9	6.
2	6 251,	7 232,	4 256,	10 257,	5 236	6 247,	9 238,	5 245,	6 248	4 223	6.4 243.3	236.9	-0
3	3 246	8 269	6 246	4 254	5 258	2 225	5 261	6 244	6 247	7 276	5.2 252.6	247.4	
4	7 264	5 245	3 223	7 241	5 265	3 236	6 238	8 248	6 248	6 226	5.6 243.4	247.8	
5	6 210	9 242	7 243	4 249	5 270	6 274	6 249	8 262	5 231	6 248	6.2 247.8	241.6	
6	6 240	5 254	6 233	5 255	4 248	5 260	8 272	6 245	5 252	3 235	5.3 250.4	245.1	
7	1 217	10 260	6 257	6 252	5 274	6 258	2 259	4 250	8 246	9 256	5.7 250.9	245 2	
8	7 234	4 245	7 277	6 242	9 256	6 253	7 251	2 261	4 248	9 263	[6.1] 253.0	246.9	
9	7 251	3 232	9 235	7 270	6 262	4 246	10 272	5 234	8 241	4 255	6.3 249.8	243.5	

β only
237.6

Bkgnd 236, 256, 245, 228, 225, 251, 218, 262, 242, 24
230, 266, 258, 244, 233, 260, 244, 253, 235, 237
5 4 4 4 2 6 10 7, 8 6

243.2
5.6

	Net β	α		Net α
1	6.3	5.8	-5.6	0.2
2	-0.7	6.4		0.8
3	9.8	5.2		-0.4
4	10.2	5.6		0
5	4.0	6.2		0.6
6	7.5	5.3		-0.3
7	7.6	5.7		0.1
8	9.2	6.1		0.4
9	5.9	6.3		0.7
Ave	6.2			
Background	243.2	5.6		

002752 C

SN: $\frac{2224}{125601}$
α eff.: 18.5%
β⁻ eff: 11.4%

											β⁻	α
	5	7	4	4	5	6	2	10	6	4		
Bkgnd	256	258	256	233	230	253	239	271	259	235	249.0	5.5
	7	4	10	4	5	7	2	4	7	1		
1.	291	284	280	288	301	280	311	289	280	303	290.7	5.1
	2	4	3	6	3	3	2	3	6	5		
2.	274	299	290	269	295	292	261	258	282	282	280.2	3.7
	5	7	0	4	8	6	4	7	10	3		
3.	277	298	291	253	263	281	280	296	270	283	279.2	5.4
	9	7	4	5	11	4	10	4	5	1		
4.	278	295	282	255	289	276	251	288	307	258	277.9	6.0
	4	4	3	3	4	8	0	2	8	6		
5.	306	278	308	287	305	298	292	289	323	276	296.2	4.2
	3	5	5	6	6	4	7	3	9	8		
6.	297	270	325	302	283	307	309	298	328	311	303.0	5.6
	5	4	4	4	7	7	6	3	2	9		
7.	279	283	311	291	322	294	294	291	300	276	294.1	5.1
	7	4	5	2	4	6	5	6	2	2		
8.	331	325	297	286	305	272	339	806	333	299	309.3	4.3
	3	6	8	4	2	7	5	3	3	3		
9.	299	300	281	308	309	314	298	283	289	303	298.4	4.4
	8	6	8	5	5	6	6	4	8	3		
Bkgnd.	237	292	227	257	227	250	229	247	277	240	248.3	5.9
											287.8	4.38
								Bkgnd			248.7	5.7
								Net c/m			39.1	
								dpm			343	

PREPARED BY	
DATE	

Material Disposition Plan :

Process Equipment - Electrical Enterprise

Processing of Pipes, etc : May ?? Readiness Review

Material receipt May 31 - early June.

Corrective Actions from DOE.

Project Plan for receiving Material.

Status of Draft of Report

Procurement

Transportation :

Waste Management

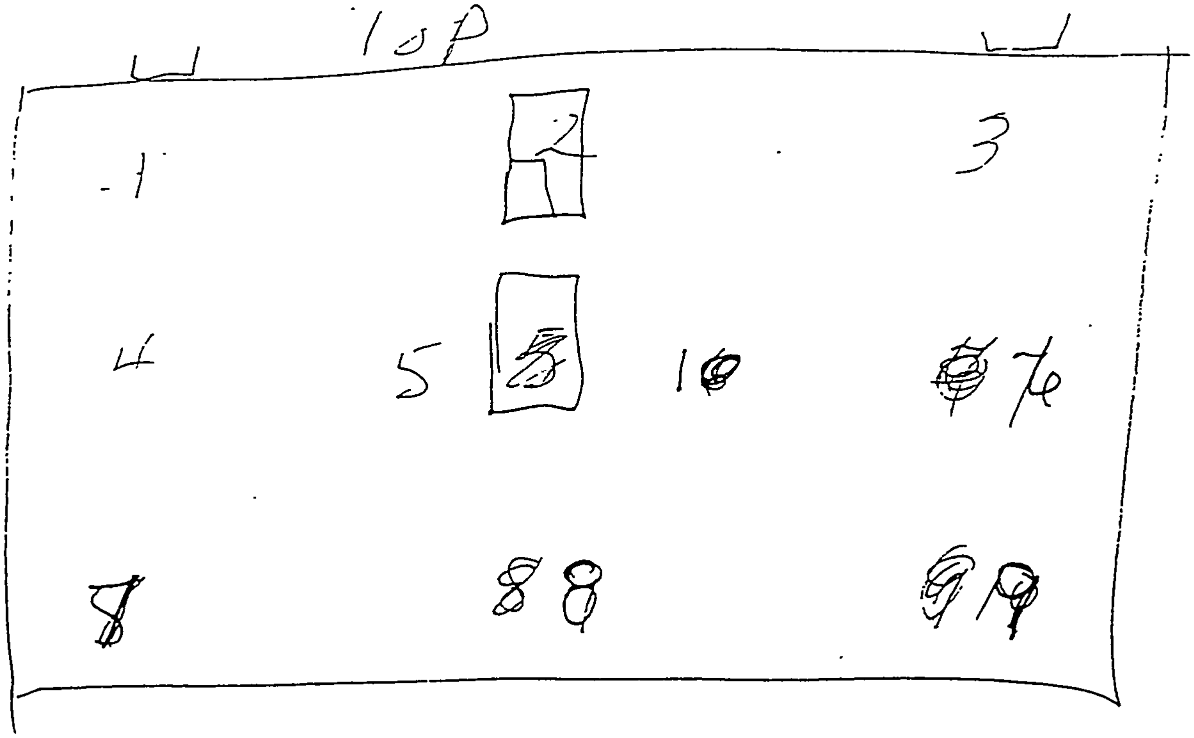
Ni - Pretreatment

Keep records of work done here.

Training requests

Calibration + Procedures :

1.



2222

Ni Anode

Thicker material

1	2	3	Top
4	5	6	
7	8	9	

222H Cal. Date: 5-26-98

SN 125578

α eff: 20.19%

β^- " : 9.1%

#002858A

8-6-98

β^-

	2	4	4	1	2	3	1	1	2	1	~~8~~					
1	707	715	760	682	698	745	687	699	702	633	~~728~~			702.8	2.1	702.8 ±34
	3	1	2	2	2	3	2	2	1	2	2	3	1			
2	715	708	750	755	768	724	690	773	746	714	756	741	707			737.4 ±28
	0	1	4	0	1	0	2	1	1	1						
3	688	641	661	670	646	636	655	649	679	472						659.7 ±17
	0	1	4	3	2	1	1	0	2	2						
4	771	743	753	718	760	739	710	726	755	751						742.6 ±19
	3	2	1	3	2	1	2	1	1	1						
5	746	713	765	775	769	724	746	744	718	760						746.0 ±22
	2	1	1	2	4	0	1	2	1	1						
6	695	698	670	644	689	709	707	702	687	689						689.0 ±19
	0	1	3	2	1	2	0	1	1	2						
7	772	799	771	768	822	738	765	806	800	779						781.8 ±22
	1	1	3	5	3	1	1	0	1	3						
8	762	769	720	770	751	771	777	751	768	737						757.6 ±18
	2	3	2	1	1	2	5	4	1	1						
9	747	787	768	738	763	764	776	784	765	782						767.4 ±16

Ave 732 ± 40

	3	1	1	1	3	0	1	1	1	2		
Backgrd	290	271	289	268	242	279	240	274	273	259	270 ±15	268.5
	4	5	0	1	1	1	0	2	1	2		
	242	271	282	260	281	258	285	278	276	275		271.8

2.981 g dissolved in 20 ml HNO3. Diluted to 100 ml 1 ml Counted for 1 hour

					dpm/g
Sample 1	51.5 cpm	30 net cpm	28.9	30 dpm/ml	1,020
Sample 2	49.3 cpm	27.8			
Anolyte	81.9 cpm	60.4			
Catholyte	36.2 cpm	14.7			

```
10 Aug 1998  18:13          ALPHA/BETA - 1.08                          Page
Protocol #: 2               Tc-99                                  User : Tc-

Time:  60.00
Data Mode: CPM                    Nuclide: MANUAL
Background Subtract: None

               LL      UL    LCR    25%      BKG
Region A:     0.0 -   200      0    0.0     0.00
Region B:     4.0 -   200      0    0.0     0.00
Region C:     0.0 -   0.0      0    0.0     0.00

Quench Indicator: tSIE/AEC
   Ext Std Terminator: Count
Tc-99 in Ni solution


  S#    TIME      CPMA      DPM1      SIS  FLAG
   1   60.00     51.50              64.137
   2   60.00     49.30              63.128
```

002858A

5 60.00 21.46 65.994 BKgnd

```
17 Sep 1998  18:43          ALPHA/BETA - 1.08 .                      Page #:
Protocol #:10               Tc-99/U-238:a/b  3.0g dissolved            User : MSC l
                                             2ml of 100ml dilution added to
Time:  60.00                                 cocktail.
Data Mode: CPM                  Nuclide: MANUAL
Background Subtract: None

              LL     UL    LCR    2S%     BKG
Region A:    0.0 -  250     0     0.0     0.00
Region B:    100 -  500     0     0.0     0.00
Region C:    0.0 -  0.0     0     0.0     0.00

Quench Indicator: tSIE/AEC
   Ext Std Terminator: Count
Alpha/Beta analysis of Tc-99:U-238

Net cpm S#   TIME     CPMA     CPMB      DPM1      SIS  FLAG       | Bq/g
         1   60.00    25.89    14.90               97.542  Stock Ni |
 7.03    2   60.00    32.92    14.88               82.998  1A       | 1.95
 7.24    3   60.00    33.13    14.90               83.623  1B       | 2.01
 9.25    4   60.00    35.14    15.64               83.383  1C       | 2.57     2.18
 7.39    5   60.00    33.28    14.48               83.353  1D       | 2.05
 8.30    6   60.00    34.19    16.12               84.404  1E       | 2.31
 7.83    7   60.00    33.72    15.45               86.125  1F       | 2.18
 6.28    8   60.00    32.17    15.10               82.493  2A       | 1.74
 5.42    9   60.00    31.31    13.63               79.366  2B       | 1.50
 4.81   10   60.00    30.70    14.16               82.285  2C       | 1.34     1.71
 7.12   11   60.00    33.01    14.92               82.646  2D       | 1.98
 8.96   12   60.00    34.85    15.07               81.352  2E       | 2.49
 4.43   13   60.00    30.32    13.88               81.812  2F       | 1.23
        14   60.00    28.80    13.82               172.64  BKgnd    |
```

LB5100-W Low Background Counting System -- Smear Analysis

Date: 9/15/98
Counting Unit id: 1
Data file name: [60S1C000.XLD]SMEARAB2
Batch Ended: 9/15/98 5:51
Crosstalk Correction: Not Applied

Alpha activity action level (DPM): 20.00
Beta activity action level (DPM): 200.00
Certainty level for MDA and flags: 95.00%
High Voltage Setting: 1350

Application Revision: 2.1.4

Application Version: Standard

Alpha efficiency log file: Th230ab
Alpha Efficiency: 33.07%
Alpha to Beta Crosstalk: 16.54%
Alpha Background (CPM): 0.6
Alpha Correction Factor: 1.000

Beta efficiency log file: Tc99ab
Beta Efficiency: 25.82%
Beta into Alpha Crosstalk: 3.71%

Beta Background (CPM): 4.3
Beta Correction Factor: 1.000

Batch ID: Ni shavings 9/14/98 - ETTP

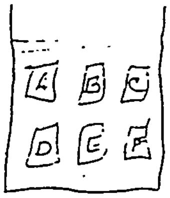
		Alpha Activity				Beta Activity				Count	Alpha	Beta	Completion
	Carrier	DPM	1σ	flags	MDA	DPM	1σ	flags	MDA	time (min)	CPM	CPM	Date - Time
Blank	-91	-0.907	0.77	<MDA	2.77	-4.45	2.69	<MDA	9.22	60.00	-0.300	-1.15	9/14/98 17:48
1A	- 92	-0.605	0.78	<MDA	2.77	24.53	3.07	<AL	9.22	60.00	-0.200	6.33	9/14/98 18:48
1B	- 93	0.050	0.80	<MDA	2.77	24.98	3.08	<AL	9.22	60.00	0.017	6.45	9/14/98 19:48
1C	- 94	-0.756	0.78	<MDA	2.77	26.21	3.09	<AL	9.22	60.00	-0.250	6.77	9/14/98 20:49
1D	-95	-0.252	0.79	<MDA	2.77	24.33	3.07	<AL	9.22	60.00	-0.083	6.28	9/14/98 21:49
1E	-96	-0.050	0.80	<MDA	2.77	27.75	3.12	<AL	9.22	60.00	-0.017	7.17	9/14/98 22:49
1F	- 97	-0.101	0.80	<MDA	2.77	29.69	3.14	<AL	9.22	60.00	-0.033	7.67	9/14/98 23:49
2A	- 98	-0.302	0.79	<MDA	2.77	25.50	3.08	<AL	9.22	60.00	-0.100	6.58	9/15/98 0:49
2B	-99	-0.605	0.78	<MDA	2.77	28.14	3.12	<AL	9.22	60.00	-0.200	7.27	9/15/98 1:50
2C	-100	-0.655	0.78	<MDA	2.77	25.11	3.08	<AL	9.22	60.00	-0.217	6.48	9/15/98 2:50
2D	- 71	0.252	0.81	<MDA	2.77	29.76	3.14	<AL	9.22	60.00	0.083	7.68	9/15/98 3:50
2E	- 72	-0.252	0.79	<MDA	2.77	31.30	3.17	<AL	9.22	60.00	-0.083	8.08	9/15/98 4:50
2F	-73	-0.403	0.79	<MDA	2.77	23.49	3.06	<AL	9.22	60.00	-0.133	6.07	9/15/98 5:51

3 grams of drillings from 2 cathodes: 6 sites from each cathode drilled to determine uniformity of Tc. deposition

9/15/98

12560! 8 Sept 98

2.232 g/100 cm²



A α	A β	B α	B β	C α	C β
3	304	4	339	7	311
5	334	8	303	6	329
9	267	5	310	7	318
3	298	8	303	6	315
5	322	6	311	3	322
4	317	7	322	5	290
4.83	307	6.33	314.67	5.67	314.17
	.848 dpm		65.89 dpm		61.65 dpm

#1

Background α	Background β
11	317
9	327
2	301
3	278
6	304
7	314
11	287
6	279
5	320
4	342
6.4	306.9

D α	D β	E α	E β	F α	F β
6	322	6	306	9	343
5	324	7	331	4	339
9	326	5	311	3	348
6	307	7	335	7	328
6	291	6	305	3	318
8	358	3	332	3	312
6.67	321.33	5.67	320	4.83	331.33
	122.37 dpm		111.09 dpm		207.17 dpm

	Net cpm	dpm	Bf/
A	0.18	0.85	0.0c
B		65.89	0.4
C		61.65	0.4
D		122.37	0.9
E		111.09	0.8
F		207.17	1.5

Ave ¹⁄

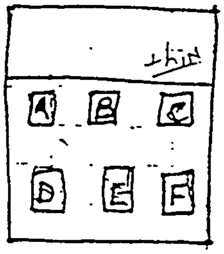
A α	A β	B α	B β	C α	C β
6	348	3	380	4	351
1	351	2	376	3	355
4	364	4	377	6	352
2	366	5	361	5	348
8	361	4	399	11	346
	358		380.6		350.4
	433.3 dpm		624.98 dpm		348.88 dpm

#2

	Net cpm	dpm	Bf/
A		433.3	3.2
B		625	4.6
C		368.9	2.7
D		709.8	5.3
E		582.4	4.3
F		433.3	3.2

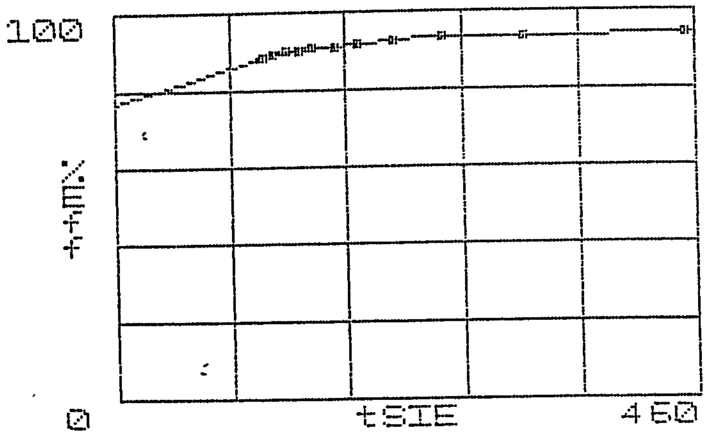
Ave. 3.9

D α	D β	E α	E β	F α	F β
2	409	4	396	3	373
1	402	6	385	3	356
4	354	5	402	4	350
3	414	7	380	4	327
8	374	6	315	5	384
	390.6		375.6		358
	709.78 dpm		582.58		433.33 dpm



Very Rough

```
21 Oct 1998  18:21          ALPHA/BETA - 1.08                        Page #
Protocol #: 6               Tc-99/Ni                           User : RSMRP

Time:  60.00
Data Mode: DPM               Nuclide: TC-99/NI       Quench Set: TC-99 QC
Background Subtract: None

               LL     UL    LCR    2S%     BKG
Region A:    0.0 -   293     0     0.0     0.00
Region B:    2.0 -   293     0     0.0     0.00
Region C:    0.0 -   0.0     0     0.0     0.00

Quench Indicator: tSIE/AEC
   Ext Std Terminator: Count
Ni in Nitric Acid:Nominal 1g/25mL
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal
```



```
tSIE    %Eff
452.85  94.17
326.05  93.21
262.00  93.69
222.04  92.34
193.79  91.83
175.40  90.98
157.38  91.14
145.19  90.27
135.44  89.72
125.28  89.06
116.84  88.24
```

S#	SMPL_ID	TIME	CPMA	CPMB	DPM1	tSIE	
1	Tc-99/Ni BLANK1	60.00	29.29	29.26	32.83	127.55	
2	11000dpm Control	60.00	10341.6	10319.3	10983.6	451.20	
3	Ni Plate CHT-9-10-6 ABC	60.00	37.77	37.72	45.87	57.445	107%, [illegible]

25 [illegible]

2.9 bq/g	5	"								
1.3 bq/g	6	"	"	#3L CAT#1-A	60.00	31.60	31.54	38.90	46.401	1.01g/25ml × 2ml
2.7 bq/g	7	"	"	#3L CAT#1-B	60.00	36.38	36.34	45.84	27.730	1.00g/25ml × 2ml
4.4 bq/g	8	"	"	#3L CAT#1-C	60.00	44.65	44.63	54.89	47.375	1.04g/25ml × 2ml

S#3 $45.87\,dpm - 32.83\,dpm = 13.04\,\frac{d}{min} \times \frac{1\,min}{60\,sec} \times \frac{25\,ml}{1.07\,g} \times \frac{1}{2\,ml} = 2.5$ beq/g

S#4 $45.81\,dpm - 32.83\,dpm = 12.98\,\frac{d}{min} \times \frac{1\,min}{60\,sec} \times \frac{25\,ml}{1.11\,g} \times \frac{1}{2\,ml} = 2.4$ beq/g

S#5 $47.64\,dpm - 32.83\,dpm = 14.81\,\frac{d}{min} \times \frac{1\,min}{60\,sec} \times \frac{25\,ml}{1.06\,g} \times \frac{1}{2\,ml} = 2.9$ beq/g

2.6 Bq/g

S#6 $38.90\,dpm - 32.83\,dpm = 6.07\,\frac{d}{min} \times \frac{1\,min}{60\,sec} \times \frac{25\,ml}{1.01\,g} \times \frac{1}{2\,ml} = 1.3$ beq/g

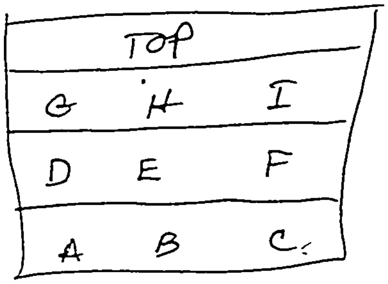
S#7 $45.84\,dpm - 32.83\,dpm = 13.01\,\frac{d}{min} \times \frac{1\,min}{60\,sec} \times \frac{25\,ml}{1.00\,g} \times \frac{1}{2\,ml} = 2.7$ beq/g

S#8 $54.89\,dpm - 32.83\,dpm = 22.06\,\frac{d}{min} \times \frac{1\,min}{60\,sec} \times \frac{25\,ml}{1.04\,g} \times \frac{1}{2\,ml} = 4.4$ beq/g

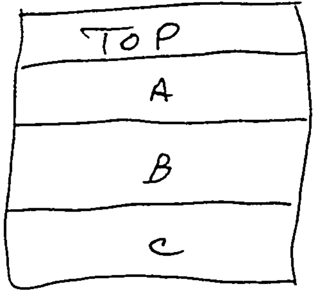
2.8 Bq/g

AVE

CH-9-10-8



3L CATH#1



LB5100-W Low Background Counting System -- Smear Analysis

Date: 10/15/98
Counting Unit id: 1
Data file name: [10S1J002.XLD]SMEARAB2
Batch Ended: 10/15/98 16:47
Crosstalk Correction: Not Applied

Alpha activity action level (DPM): 20.00
Beta activity action level (DPM): 200.00
Certainty level for MDA and flags: 95.00%
High Voltage Setting: 1350

Application Revision: 2.1.4

Application Version: Standard

Alpha efficiency log file: Th230ab
Alpha Efficiency: 33.07%
Alpha to Beta Crosstalk: 16.54%
Alpha Background (CPM): 0.6
Alpha Correction Factor: 1.000

Beta efficiency log file: Tc99ab
Beta Efficiency: 25.82%
Beta into Alpha Crosstalk: 3.71%

Beta Background (CPM): 4.3
Beta Correction Factor: 1.000

Batch ID: Ni CATHODE SHAVINGS 10-5 & 10-15

			Alpha Activity				Beta Activity				Count	Alpha	Beta	Completion	
		Carrier	DPM	1σ	flags	MDA	DPM	1σ	flags	MDA	time (min)	CPM	CPM	Date - Time	
IT-9-10-8	G, H, I	1	0.907	1.17	<MDA	4.27	24.01	4.74	<AL	12.88	10.00	0.300	6.20	10/15/98 15:56	1.06 g
"	A, B, C	2	0.605	1.13	<MDA	4.27	27.88	4.91	<AL	12.88	10.00	0.200	7.20	10/15/98 16:06	1.07 g
"	D, E, F	3	0.000	1.05	<MDA	4.27	25.17	4.79	<AL	12.88	10.00	0.000	6.50	10/15/98 16:16	1.11 g
3L CAT #1	A	4	0.605	1.13	<MDA	4.27	32.14	5.08	<AL	12.88	10.00	0.200	8.30	10/15/98 16:27	1.26 g
"	B	5	-0.302	1.00	<MDA	4.27	17.43	4.45	<AL	12.88	10.00	-0.100	4.50	10/15/98 16:37	1.23 g
"	C	6	-0.302	1.00	<MDA	4.27	27.50	4.89	<AL	12.88	10.00	-0.100	7.10	10/15/98 16:47	1.21 g

CHT-9-10-8 $\frac{77.06}{3.[illegible]\,g}$ 23.78 dpm/g
= 0.40 Bq/g

STACY M REDWINE
10-15-98

3LCAT # 1 $\frac{77.07}{3.70\,g}$ = 20.83 dpm/g
= 0.35 Bq/g

CHT-9-10-8

Section	β^-	α
A	316 ± 20	4.2 ±
B	338 ± 16	6.3 ±
C	333 ± 25	5.5 ±
D	326 ± 19	4.9 ±
E	327 ± 20	4.9 ±
F	329 ± 15	6.5 ±
G	323 ± 13	4.3 ±
H	340 ± 24	7.2 ±
I	327 ± 18	6.9 ±
Bkgnd	290 ± 9	5.2 ± 2.4
1 SD	281 - 299	2.8 - 7.6
2 SD	272 - 308	

Steve Sarten

From: Lab
Sent: 11. mai 1999 08:25
To: Steve Sarten
Cc: Dianna Walker
Subject: FW: Tc-99 And Nickel Anolyte/Catholyte/Starter Plate Results

I left off the Nickel results from the 1st e-mail. Here they are!

Sample ID	Tc-99	Nickel (ppm)
Anolyte 5/7/99 3:00 PM	2.68 Bq/mL	104,300
Catholyte 5/7/99 3:00 PM	<0.06 Bq/mL	64,800
Anolyte 5/8/99 6:30 AM	2.70 Bq/mL	90,400
Catholye 5/8/99 6:30 AM	<0.06 Bq/mL	67,800
Anolyte 5/10/99 6:30 AM	2.54 Bq/mL	96,300
Catholyte 5/10/99 6:30 AM	<0.06 Bq/mL	56,400
Starter Plate-right side	1.83 Bq/g	
Starter Plate-left side	1.59 Bq/g	

Steve Sarten

From:	Lab
Sent:	7. mai 1999 08:39
To:	Steve Sarten
Cc:	Dianna Walker
Subject:	Tc-99 Results on Anolyte/Catholytes May 5-6

Sample ID	Tc-99 (Bq/mL)
Anolyte 5/5/99 3:30 PM	2.17
Catholyte 5/5/99 3:30 AM	<0.06
Anolyte 5/6/99 6:15 AM	2.27
Catholyte 5/6/99 6:15 AM	<0.06
Anolyte 5/6/99 2:30 PM	2.44
Catholyte 5/6/99 2:30 PM	<0.06

Steve Sarten

From:	Lab
Sent:	6. mai 1999 08:34
To:	Steve Sarten
Cc:	Dianna Walker
Subject:	Tc-99 Results on Anolyte/Catholyte 5/4-5/99

Below are the final results (long count) of samples analyzed yesterday with a shorter screening count times:

Sample ID	Tc-99 (Bq/mL)
Anolyte 5/4/99 3:30 PM	2.02
Anolyte 5/5/99 6:30 AM	2.08
Catholyte 5/5/99 6:30 AM	0.11

From: Lab
Sent: 5. mai 1999 14:52
To: Steve Sarten
Cc: Dianna Walker
Subject: Tc-99 Screen Results on 5/4-5/5 Anolytes

These results are from a 15 min count instead of the usual hour count time. This will give you an idea of the approximate Tc-99 activity.

Sample ID	Tc-99 (Bq/mL)
Anolyte 5/4/99 3:30 PM	1.95
Anolyte 5/5/99 6:30 AM	1.99
Catholyte 5/5/99 6:30 AM	0.13

Call if you have any questions.

Harold x248

Steve Sarten

From:	Lab
Sent:	5. mai 1999 10:07
To:	Steve Sarten
Cc:	Dianna Walker
Subject:	Tc-99 Resutls for Anolytes 4/30 thru 5/4

Sample ID	Tc-99 (bq/mL)
Anolyte 4/30/99 3:30 PM	2.16
Anolyte 5/3/99 6:15 AM	2.14
Anolyte 5/3/99 3:30 PM	2.10
Anolyte 5/4/99 6:15 AM	1.95

Steve Sarten

From:	Lab
Sent:	3. mai 1999 12:35
To:	Steve Sarten
Cc:	Dianna Walker
Subject:	Anolyte Tc-99 Results

Sample ID	Tc-99 (bq/mL)
Anolyte 4/29/99 3:30 PM	2.53
Anolyte 4/30/99 6:15 AM	2.51

Steve Sarten

From:	Lab
Sent:	27. april 1999 11:19
To:	Steve Sarten
Cc:	Dianna Walker
Subject:	Nickel plating solution Analytical Lab Results

Sample Description	Tc-99 (Bq/mL)
Charcoal H2O 4/24/99	0.17
Anolyte 4/23/99 3:30 PM	5.61
Catholyte 4/23/99 3:30 PM	<0.06
Anolyte 4/24/99 6:30 AM	5.53
Anolyte 4/24/99 11:30 AM	4.15
Catholyte #1 4/24/99	0.06
Anolyte 4/26/99 6:30 AM	2.09
Catholyte 4/26/99 6:30 AM	<0.06
CAT#1L plate	2.73 Bq/g
CAT#1R plate	3.29 Bq/g

Let us know if you have questions.

Harold

Steve Sarten

From: Lab
Sent: 23. april 1999 13:53
To: Steve Sarten
Cc: Dianna Walker
Subject: Tc-99 Results on Anolyte Samples

Results from recent Anolyte samples:

Sample Description	Tc-99 (bq/mL)
Anolyte 4/21/99 3:30 PM	5.56
Anolyte 4/22/99 6:30 AM	5.52

Results below are short counts results to give you an idea of your approximate Tc-99 activity:

Anolyte 4.22/99 3:30 PM	5.65
Anolyte 4/23/99 7:00 AM	2.65
Catholyte #1 4/23/99 7:30 AM	<MDA

Will count this later batch to give you a more definitive activity on Monday.

Harold

From:	Lab
Sent:	22. april 1999 09:06
To:	Steve Sarten
Cc:	Dianna Walker
Subject:	Anolyte Analytical Results

Steve,

Below are the results of several anolyte batches:

Sample Description	Tc-99 (bq/mL)	Ni (ppm)
Anolyte-Before 4/17/99 7:00AM	14.41	72,900
Anolyte-After 4/17/99 11:30AM	15.15	73,400
Anolyte-Before 4/19/99 6:30AM	15.22	74,700
Anolyte 4/19/99 3:30PM	14.78	N/A
Anolyte 4/20/99 7:00AM	15.50	N/A
Anolyte 4/20/99 3:30PM	15.83	N/A
Anolyte 4/21/99 6:30AM	15.29	N/A
Anolyte 4/21/99 10:00AM	15.35	N/A

If you have any questions, Let Elbert or me know.

Harold

From: Lab
Sent: 6. april 1999 13:09
To: Steve Sarten
Cc: Dianna Walker
Subject: Tc-99 Results for Anolyte/Catholyte 4/6/99 Samples

Dianna,

Please forward a Hard copy of these results to Sam Fritts.

Sample #	Tc-99
Anolyte-Before 4/6/99	15.2 bq/mL
Anolyte-After "	15.4 bq/mL
Catholyte#1 "	1.7 bq/mL
Catholyte#2 "	0.8 bq/mL

Thanks,

Harold Waddle

From: Lab
Sent: 5. april 1999 14:33
To: Steve Sarten
Subject: Anolyte/Catholyte Results from 4-5-99

Steve,

Reported below are the Tc-99 Results from sample brought in 4/5/99 AM:

Sample	Tc-99
Anolyte	15.5 bq/mL
Catholyte #1	1.2 bq/mL
Catholyte #2	0.7 bq/mL
Anolyte-Before	15.2 bq/mL
Anolyte-After	15.3 bq/mL

Let me know if you have any questions about the results.

Harold

From: Lab
Sent: 2. april 1999 08:16
To: Steve Sarten
Subject: Nickel Starter Plate Tc-99 Results for RFA#004630

Steve,

Below are the results you so anxiously await. The QC ran did very well. The blanks that were used were batched along with these samples. We normally use an older blank and it was in close proximity to these two new blanks. The spike on #1 Right sample had a 95.8% Tc-99 recovery.

Sample Description	Tc-99 Results
#1 Right	29.8 bq/g
#1 Left	22.1 bq/g
#2 Right	7.8 bq/g
#2 Left	8.1 bq/g

Elbert is out today for Good Friday. If you have any questions, please call.

Harold Waddle

From:	Lab
Sent:	24. mars 1999 10:51
To:	Steve Sarten
Cc:	Tom Muth; Valerie MacNair
Subject:	Tc-99 in Anolyte and Anodes

Concentrations of Tc-99 in Anolyte from 3-22-99 and Anodes #1, #3, & #5 from 3-20-99 are as follows:

Anolyte	3-22-99 A.M.	15.5 Bq/ml
Anode # 1	3-20-99 A.M.	578.3 Bq/g
Anode # 3	3-20-99 A.M.	654.1 Bq/g
Anode # 5	3-20-99 A.M.	544.9 Bq/g

The table of elemental concentrations in the above samples is in your mailbox.
Call me if you have any questions.

Elbert Carlton
Analytical Services

Steve Sarten

From: Lab
Sent: 19. mars 1999 12:22
To: Steve Sarten
Cc: Tom Muth; Valerie MacNair
Subject: Anolyte/catholytes

Enclosed are the results to Tc-99 in anlyyte and catholyte for 3-17-99.

3-17-99 A.M.

Anolyte	16.34 Bq/ml
Catholyte # 1	0.03 Bq/ml
Catholyte # 2	0.06
Catholyte # 3	0.12
Catholyte # 4	0

3-17-99 P.M.

Catholyte # 1	1.77 Bq/ml
Catholyte # 2	0.26 Bq/ml
Catholyte # 3	0.34 Bq/ml
Catholyte # 4	0.22 Bq/ml

ElbertCarlton
Laboratory Services

Steve Sarten

From: Lab
Sent: 18. mars 1999 17:25
To: Steve Sarten
Cc: Tom Muth; Valerie MacNair
Subject: Tc concentrations in solutions from 3-13 thru 3-17 AM

Steve,
Enclosed are the concentrations of Tc-99 in anclytes/catholytes and starter sheets.

3-13-99 A.M.

Anolyte	13.5 Bq/ml
Catholyte 1	0
Catholyte 3	0.05 Bq/ml
Catholyte 4	0

3-15-99 A.M.

Anolyte	15.25 Bq/ml
Catholyte # 1	0
Catholyte # 2	0.009 Bq/ml
Catholyte # 3	0.030 Bq/ml
Catholyte # 4	0

3-16-99 A.M.

Anolyte	15.93 Bq/ml	
Catholyte # 1	0.33 Bq/ml	
Catholyte # 2	0.10 Bq/ml	
Catholyte # 3	0.12 Bq/ml	
Catholyte # 4	0.01 Bq/ml	
SS 1	0.96 Bq/g	– Clean Ni
2 R SS	56.70 Bq/g	
4 R SS	40.06 Bq/g	
Pilot Sample	1.95 Bq/g	
Nickel Starter	30.07 Bq/g	

3-17-99 A.M.

Anolyte	16.30 Bq/ml
Catholyte #1	0
Catholyte # 2	0.05 Bq/ml
Catholyte # 3	0.09 Bq/ml
Catholyte # 4	0

From:	Lab
Sent:	15. mars 1999 15:37
To:	Steve Sarten
Cc:	Tom Muth; Valerie MacNair
Subject:	Comparison of Tc-99 in Ni using LSC and LB5100

Steve,
The Tc-99 determined by the two procedures are as follows:

Approximately 1 gram of Ni was removed by drilling into each of four sheets, spread uniformly in a planchet and counted for 10 minutes in the LB5100 alpha/beta counter.
The samples were then transferred to individual beakers. 10 ml of concentrated HNO3 was added to each and heated to dissolve the Nickel. After dissolution, distilled water was added and evaporated to reduce the NO2 concentration. The final volume was made up to 25 ml with distilled water and mixed thoroughly. One ml was removed from each flask and added to a 15 ml aliquot of scintillation cocktail and counted for 60 minutes.

Cathode	LSC	LB5100	
# 1 (1.0037 g Ni)	14.21 Bq/g	1.09 Bq/g	
# 2 (1.0011 g Ni)	30.48 Bq/g	3.27 Bq/g	
# 3 (1.0127 g Ni)	22.45 Bq/g	2.04 Bq/g	
# 4 (1.0013 g Ni)	13.36 Bq/g	1.77 Bq/g	
# 4 (1.0013 g Ni)	12.99 Bq/g	———	Duplicate

Steve Sarten

From:	Lab
Sent:	15. mars 1999 15:04
To:	Steve Sarten
Cc:	Tom Muth; Valerie MacNair
Subject:	Tc-99 in Catholytes/Anolytes

Steve,

Enclosed are the results for anolytes/catholytes from 3-12-99 A.M.

Virgin Ni (1.0045 g/25 ml HNO3) 2 ml	38.82 dpm	
Catholyte # 02 3-12-99 A.M. 1 ml	359.3 dpm	5.34 Bq/ml
Anolyte (before) 3-12-99 A.M. 1 ml	858.1 dpm	13.7 Bq/ml
Anolyte (after) 3-12-99 A.M. 1 ml	846.9 dpm	13.5 Bq/ml
Catholyte # 01 3-12-99 A.M. 1 ml	401.2 dpm	6.0 Bq/ml
Catholyte # 1 3-12-99 A.M. 1 ml	63.9 dpm	0.42 Bq/ml
Catholyte # 2 3-12-99 A.M. 1 ml	107.1 dpm	1.15 Bq/ml
Catholyte # 4 3-12-99 A.M. 1 ml	79.3 dpm	0.68 Bq/ml
Catholyte # 3 + 100 dpm Tc spike	178.0 dpm	

Steve Sarten

From: Lab
Sent: 3. mars 1999 10:09
To: Steve Sarten
Subject: Anolyte/Catholyte analyses for Tc-99

Concentrations for Tc-99 in Anolyte and Catholyte, and Cathodes are shown in the following table.

2-26-99 PM	Bq/ml
Anolyte	9.24
Catholyte - 1	0.05
Catholyte - 2	0.19
Catholyte - 3	0.05
Catholyte - 4	0.52

2-27-99 AM	Bq/ml
Anolyte	9.27
Catholyte - 1	0.12
Catholyte - 2	0.18
Catholyte - 3	0.05
Catholyte - 4	0.00

2-27-99 PM	Bq/ml
Anolyte	10.32
Catholyte - 1	0.31
Catholyte - 2	0.39
Catholyte - 3	0.15
Catholyte - 4	0.05

2-28-99 AM	Bq/ml
Anolyte	10.87
Catholyte - 1	0.10
Catholyte - 2	0.18
Catholyte - 3	0.09
Catholyte - 4	0.00

2-28-99 PM	Bq/ml
Anolyte	11.05
Catholyte - 1	0.14
Catholyte - 2	0.30
Catholyte - 3	0.18
Catholyte - 4	0.00

3-1-99 AM	Bq/ml
Anolyte	11.10

Catholyte - 1	0.03
Catholyte - 2	0.20
Catholyte - 3	0.20
Catholyte - 4	0.08

3-2-99 AM	Bq/ml
Anolyte	12.18
Catholyte - 1	0.66
Catholyte - 2	0.50
Catholyte - 3	0.33
Catholyte - 4	0.00
Anolyte - Before	31.33
Anolyte - After	14.86

3-1-99	Bq/g
Cathode # 1	20.55
Cathode # 2	17.52
Cathode # 3	7.41
Cathode # 4	6.82

Steve Sarten

From: Lab
Sent: 1. mars 1999 09:19
To: Steve Sarten
Subject: Anolyte-Catholyte Concentrations

Steve, The concentrations of Tc-99 in Anolyte and Catholytes for 2-25-99 PM and 2-26-99 AM are as follows.

2-25-99 5:30 PM	Tc-99 Bq/ml
Anolyte	8.43
Catholyte - 1	0.14
Catholyte - 2	0.21
Catholyte - 3	0.08
Catholyte - 4	0.06

2-26-99 6:00 AM	
Anolyte	8.27
Catholyte - 1	0.09
Catholyte - 2	0.09
Catholyte - 3	0.12
Catholyte - 4	0.02
Anolyte Before	8.08
Anolyte After	8.55

Cementation # 3 (1.75 g)	Bq/g
(1 ml of 28 ml)	2,500
(2 ml of 28 ml)	2,130

From: Lab
Sent: 26. februar 1999 09:34
To: Steve Sarten
Cc: Valerie MacNair
Subject: Tc-99 in Anolytes-Catholytes

Steve, the concentrations in the Anolytes and Catholytes for 2-24-99 PM and 2-25-99 AM are given below:

2-24-99 5:30 PM	Tc-99 (Bq/ml)
Anolyte	6.96
Catholyte - 1	0.32
Catholyte - 2	0.24
Catholyte - 3	0.09
Catholyte - 4	0.03

2-25-99 6:00 AM	Tc-99 (Bq/ml)
Anolyte	7.64
Catholyte - 1	0.42
Catholyte - 2	0.30
Catholyte - 3	0.08
Catholyte - 4	0.04

Steve Sarten

From: Lab
Sent: 25. februar 1999 14:31
To: Steve Sarten
Cc: Valerie MacNair
Subject: Anolyte-Catholytes

The following values were determined for Tc-99 in Ni materials:

Sample #	Bq/ml or gram
4L-1 new membrane (1.0343 g Ni)	7.80
4L-1 " " " dup.	8.45
4L-2 " " (1.0465 g Ni)	8.06
Anolyte 2-23-99 (6:00 am)	7.04
Catholyte - 1 2-23-99 (6:00 am)	1.56
Catholyte - 2 " "	4.59
Catholyte - 3 " "	0.99
Catholyte - 4 " "	1.07

Anolyte and catholyte recounted using Ultima Gold a/b cocktail

Anolyte	6.92
Catholyte - 1	1.56
Catholyte - 2	4.65
Catholyte - 3	1.04
Catholyte - 4	1.11
Water from pump	0.05

Ni from 4L before Washing	8.38
Ni from 4L after Washing	5.81
Anolyte 2-23-99 PM collection	6.88
Catholyte - 1 2-23-99 PM collection	0.23
Catholyte - 2 " " "	0.57
Catholyte - 3 " " "	0.14
Catholyte - 4 " " "	0.08
Anolyte 2-24-99 AM Collection	6.73
Catholyte - 1 " " "	0.01
Catholyte - 2 " " "	0.12
Catholyte - 3 " " "	0.03
Catholyte - 4 " " "	0.03

Steve Sarten

From:	Lab
Sent:	22. februar 1999 12:33
To:	Steve Sarten
Cc:	Valerie MacNair
Subject:	Tc-99 concentrations in Anolyte and Catholyte solutions

The concentrations in the Anolyte and Catholyte samples from 2-18-99 and 2-19-99 are as follows:

Anolyte 5:50 PM 2-18-99:	5.87 Bq/ml
Catholyte 1 5:50 PM 2-18-99:	0.01 Bq/ml
Catholyte 2	0
Catholyte 3	0.01 Bq/ml
Catholyte 4	0

Anolyte 5:30 AM 2-19-99	6.34 Bq/ml
Catholyte 1 5:30 AM 2-19-99	0.28 Bq/ml
Catholyte 2	0
Catholyte 3	0
Catholyte 4	0

Anolyte PM 2-19-99	6.80 Bq/ml
Catholyte 1 PM 2-19-99	0
Catholyte 2	0
Catholyte 3	0
Catholyte 4	0

Ni - 1L	12.5 Bq/g
Ni - 1R	9.7 Bq/g
Ni - 2L	6.7 Bq/g
Ni - 2R	9.4 Bq/g