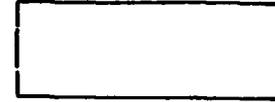


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Contractor Report ARCCD-CR-87004

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ESTABLISH TECHNIQUES FOR CLEANING DEPLETED URANIUM
DERBY IN LIEU OF NITRIC ACID PICKLING

Charles E. Latham-Brown
Nuclear Metals, Inc.
2229 Main Street
Concord, Massachusetts 01742

Douglas Vanderkooi
Project Engineer
ARDC

MAY 1987

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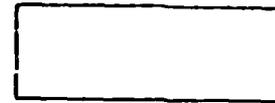
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To David Edgar of Production Base Modernization and William Sharpe of the U.S. Army Research and Development Command, our sincere appreciation for the benefit of their consultations.

INTRODUCTION

OBJECTIVE OF THE PROGRAM

The objective of the efforts described in this report is the development of a method to improve the quality of depleted uranium (DU) derby metal and eliminate the use of nitric acid in the removal of surface inclusions of the derby. This in turn will make cleaner melts containing fewer carbide inclusions and consequently, fewer ultrasonic rejects as well as reduced tool wear during machining. The present pickling procedure results in hazardous nitrous oxide fumes and disposing of the spent solution is costly. Elimination of this operation would be an advance in overall safety and would ultimately result in reduced production costs of the derby metal. An improvement in the quality of cleaning would result in higher yields and consequently reduced penetrator manufacturing costs.

BACKGROUND

As a prelude to the program, a survey was conducted of those facilities involved with the use of molten salts either for the cleaning of derby metal or for the protection of DU metal in processing operations.

From this inquiry we found that only four companies produce depleted uranium derby metal by means of the magnesium reduction of uranium tetrafluoride (UF_4). These are Nuclear Metals/Carolina Metals (CMI), Feed Materials Production Center (NLO), Aerojet Heavy Metals Division, and Eldorado (Canada). The contaminants adhering to the surface of the derby are mainly magnesium fluoride (MgF_2) but some unreacted remaining magnesium metal as well as some finely dispersed uranium may also be present in the MgF_2 . MgF_2 is insoluble in water and in most acids. Removal, therefore, has to be achieved by mechanical means, by heat shock, and in some instances by surface oxidation. Still it is important that the uranium metal is not attacked extensively by the cleaning procedure.

Of the four sources of depleted uranium derby, we are only aware of the Feed Materials Production Center using a molten salt bath as part of its cleaning program. Examples of salt compositions presently in use with uranium are: 25% lithium carbonate, 65% potassium carbonate, and 10% chlorides for heating and rolling at the Rocky Flats Plant of Rockwell International; 21-23% sodium chloride, 31-34% potassium chloride, balance barium chloride at Reactive Metals, Inc. for heating billets for extrusion; and 60% potassium carbonate, 40% lithium carbonate at the Feed Materials Production Center (NLO) for the purpose of heating derbies for magnesium fluoride removal by quenching in water. The salt combination of potassium and lithium carbonates was established by a study conducted at Y-12, Oak Ridge, Tennessee, some years ago. The actual percentages have varied slightly over time as has the operating temperature which is in the range of 1200°F.

The original premise of the salt bath approach was that the thermal expansion of the derby would be such as to loosen the adhesion of the contaminating material on the surface. After heating, the derbies are quenched in water.

The very strong thermal contraction taking place in uranium upon this cooling would then flake off the adhering salts and contaminants. Since only the surface of the derby has to be cooled, it is common practice to immerse a derby until the surface is black, then remove it from the water until the surface is red again from the residual heat and to then quench in water for a second time.

The engineering staff of Nuclear Metals, Inc. and the Project Engineer from ARDC were invited to the Feed Materials Production Center (NLO) for discussions with their staff and to observe their operation. From this visit came the fact that while the salt treatment did assist in their cleaning operation, it still was not a complete answer. The surface irregularities that develop during the reduction reaction were sometimes such that it became necessary to involve both mechanical cleaning and acid pickling to achieve an acceptably clean surface. In addition, a visual inspection and classification of the derbies is carried out in accordance with the requirements of the specific needs of the customer.

Depleted uranium derby is produced for Nuclear Metals, Inc. at Carolina Metals, Inc., a wholly owned subsidiary located in South Carolina. The magnesium reduction of UF_4 is presently carried out in a steel vessel containing a graphite liner. The process results in a relatively clean DU derby with good separation between metal and magnesium fluoride. The derbies are cleaned by a combination of mechanical impacting using a needle gun and immersion of the derby in a nitric acid solution.

The use of nitric acid for a cleaning medium presents the problems of the generation of nitrous oxide fumes which must be properly vented for both health and safety and the disposition of the spent acid solution. These same concerns are common to the facilities of the Feed Materials Production Center (NLO).

The fourth facility producing depleted uranium derby using the technique of magnesium reduction of UF_4 is Eldorado (Canada). The method employed for removal of surface contamination is high temperature oxidation. The derby is placed in a baking oven at $1000^\circ F$ and allowed to oxidize to the extent that the contamination spalls off into a collector tray. This leaves a residual oxide film on the derby which is not further addressed prior to melting. The presumption is that the oxide will float off and be contained in the melt dross rather than becoming trapped in the ingots. In view of the guidelines under which we operate, this is not an acceptable procedure for consideration.

METHODOLOGY AND EXPERIMENTAL WORK

The program was established to examine and evaluate cleaning procedures which would effectively remove the surface contaminants of the derby and in so doing, eliminate the use of the nitric acid pickling bath.

Initially, the method of cleaning concentrated on heating the derby in salt baths to elevated temperatures and quenching in water. Carbonates and chlorides, both singularly and in combination, were investigated. Since chlor-

ides salts are soluble in water, the quenching action, particularly from the elevated temperature, assists in the removal of any surface residue. The carbonates, on the other hand are not water soluble and their removal is dependent on the shrinkage and flaking off of the salt effected by the rapid quenching of the derby.

At the time of the response to the proposal, an alternative method of cleaning by high pressure water was mentioned. The technique is mainly used in the descaling of steel. Nuclear Metals did continue to investigate the conditions under which testing using this method might be evaluated. With the cooperation of Flow Systems, Inc. of Kent, Washington, and after having presented the facts to ARDC, arrangements were made to include this technique as an addition to the program.

The trials and the results will be addressed as we present the data generated in the overall program.

TEST PROGRAM

As a starting point we chose to proceed with those salt compositions known to be used with uranium and to make modifications by minor chemical additions as well as evaluating the effect of temperature variations.

The trials were conducted on a laboratory scale to permit as complete an examination as possible before dealing with a full scale operation. The test pieces were comprised of wedges cut from a standard derby (1400 lbs.) which had not been subjected to any cleaning operation. Each piece was drilled and tapped for a lifting ring as shown in Figure 1. The salt pot was steel and externally heated electrically. Temperature is controlled by built in thermocouples in addition to which a thermocouple was fastened directly to the test piece to determine when the wedge is at temperature. All test wedges were preheated at 300°F for one hour prior to immersion in the bath to insure a completely dry surface. It should be noted that it is essential that no moisture be introduced into any bath of this type as it may cause a violent reaction.

The actual test procedure is illustrated in Figures 2, 3, and 4 where we see the wedge being lowered into the salt, the sample in place for the soak period, and finally the water quench.

The list of conditions to which the pieces, with their mixtures, were subjected can be found in Table 1. In the text, however, we will undertake a detailed examination of each trial in the order in which they were conducted. Having made some examinations of derby material at Carolina Metals, Inc. we were aware of the difficulty of making a good judgement of the cleanliness of the surface without the benefit of acid pickling. We therefore decided to use an etching solution as a tool for evaluating the results of the various salt mixtures and temperature combinations. In the following data presentation when etching is referred to it should be understood that the solution used is as noted:

20% Nitric Acid
Operating Temperature 120°F
Soak time, 5 minutes

The use of three steel salt pots enabled us to change the salt compositions at will.

Test No. 0

Control

This first wedge was not cleaned in any manner prior to being subjected to a five minute etch. It did bring out the fact that many small voids contained material which did not react in the solution. Some deep folds in the outer surface were more clearly outlined. In a few areas where the surface was smooth, the acid brightened the area. This specimen was selected to act as a control comparison. All other specimens were tested in the "as received" condition.

Test No. 1

Salt Mixture Houghton 980
Test temperature - 1100°F
Soak Time - 2 hours

This low temperature resulted in a crust forming on the bath when the wedge was lowered. The wedge acted as a heat sink and we had to raise the temperature to get the bath liquid again. Once the piece was fully immersed the temperature was adjusted. Upon quenching and washing in hot water, no real changes in condition were noted. After being given the 5 minute etch, the MgF_2 showed more clearly. The surface was picked at with a pointed tool to loosen a few large pieces, but there was no sign of material actually spalling off.

Test No. 2

Salt Mixture Houghton 980
Test Temperature - 1400°F
Soak Time - 2 hours

The higher bath temperature resulted in a more fluid solution and posed no problem when lowering the wedge. After the quenching and pickling, it was noted that there was some loosening of the MgF_2 but again no actual flaking. Prior to the etching the surface was dark and dirty looking.

Test No. 3

Salt Mixture 60% K_2CO_3 , 40% Li_2CO_3
Test Temperature - 1200°F
Soak Time - 2 hours

This was the first attempt using the NLO composition. We continued to see a darkened surface upon quenching and did not see any spalling action. Examination after etching showed no real change from the initial inspection.

Test No. 1-4

Salt Mixture 60% K_2CO_3 , 40% Li_2CO_3
Test Temperature - 1200°F
Soak Time - 2 hours

This test specimen had been previously run for two hours at 1100°F in the Houghton 980 salt and was now being run in carbonates to look for signs of improvement. This second exposure did not improve the surface cleanliness.

Test No. 5

Salt Mixture 60% K_2CO_3 , 40% Li_2CO_3
Test Temperature - 1400°F
Soak Time - 2 hours

This wedge section was distinguished by a deep seam in the upper section, a few small holes about midway, and some small chunks of MgF_2 in the lower portion. After quenching it appeared that the small lumps were partially removed. The standard 5 minute etch had virtually no cleaning effect. The time was extended to 45 minutes and still the surface did not clean up. It appeared that the higher temperature resulted in a passivating action. Even the cut surface did not respond to the pickling.

Test No. 6

Salt Mixture 60% K_2CO_3 , 40% Li_2CO_3
Test Temperature - 1200°F
Soak Time - 2 hours

This trial is a repeat of the No. 4 condition using a wedge with no previous exposure. This piece had some large chunks on the surface. Following the salt exposure and quench some softening was obvious, but the heating and quenching cycle still did not result in any spalling action.

Test No. 7

Salt Mixture 57.5% K_2CO_3 , 37.5% Li_2CO_3 , 5% H_3BO_3
Test Temperature - 1200°F
Soak Time - 2 hours

This was the first addition of a material outside of the standard salt mixtures. The most noticeable effect was less adhering of the molten salt to the saw cut surface. The outside of the derby surface was essentially the same.

Test No. 8

Salt Mixture Houghton 980
Test Temperature - 1200°F
Soak Time - 2 hours

There was no visible difference from the starting conditions. The cut surfaces were clean with no attached residue.

Test No. 9

Salt Mixture Houghton 980 + 5% H₃BO₃
Test Temperature - 1200°F
Soak Time - 2 hours

After cleaning, the bottom of the wedge showed some improvement. Minor holes appeared clean, but the large pad of MgF₂ at the top was still present. The quench shock had no effect.

Test No. 0-10

Salt Mixture Houghton 980 + 5% H₃BO₃
Test Temperature - 1400°F
Soak Time - 2 hours

This wedge was also used for the original control etch. After processing, the bottom portion was brighter while the top was dark. The extreme irregularity of the surface trapped the MgF₂. The higher test temperature resulted in the darkening of the saw cut surfaces as had been experienced before in this same temperature range.

Test No. 11

Salt Mixture Houghton 980 + 10% H₃BO₃
Test Temperature - 1400°F
Soak Time - 2 hours

The increase of the boric acid addition while appearing to improve the draining of the salts had no apparent effect on the MgF₂. The higher temperature continues to be detrimental and will not be considered in further tests.

Test No. 12

This was intended to be a salt mixture test, however, the pre-heat furnace control jammed with the result that the temperature climbed to 820°F. The piece had been exposed to air for 20 minutes when the problem was noticed. When the wedge was removed and allowed to air cool, it was noticed that the test surface was better than those done in the salt bath. The wedge was etched for 10 minutes, and we developed a light gold color at the top with a fairly clean surface. The center section, which had a large amount of MgF₂, developed a dark green color whereas before such a surface was black in the salt mixture.

Test No. 13

Salt Mixture 70% K_2CO_3 , 30% Li_2CO_3
Test Temperature - 1200°F
Soak Time - 2 hours

The amount of MgF_2 on the outer face was quite heavy. After the treatment, but before etching, it appeared to have pulled away from the surface. The chunks were removed before etching and the surface etched to a bright color. Other MgF_2 coated sections were unaffected.

Test No. 14

This test was a repeat of No. 13 using the same wedge which had been treated and pickled. The heavy material in a seam at the lower section was unaffected by the first run; therefore, we soaked for an additional 2 hours and quenched again. The deeply imbedded material softened slightly but was not eaten away. No other changes were noted.

Test No. 15

Salt Mixture 70% K_2CO_3 , 30% Li_2CO_3
Test Temperature - 1200°F
Soak Time - 4 hours

While test No. 14 was treated for a total of 4 hours, the exposure to the acid batch between the cycles may have had some effect on the second run. Therefore, a separate run was scheduled for the full 4 hours. Upon examination there was no marked difference that could be attributed to the longer time. The small voids filled with MgF_2 were clearly visible.

Test No. 16

Salt Mixture 70% K_2CO_3 , 30% Li_2CO_3
Test Temperature - 1400°F
Soak Time - 2 hours

While some difficulty had been encountered with the higher temperatures, we needed this same information with the variation of the compositions. The wedge was similar to previous tests having been cut from the same derby. After quenching and cleaning there were no significant differences that would indicate small adjustments in the percentages of carbonates had any effect on the removal of MgF_2 .

Test No. 17

Air Oxidation
Test Temperature - 1000°F
Soak Time - 1 hour

This test was conducted under the conditions used by Eldorado in their derby oxidation cleaning operation. To prevent undo contamination to our furnace, we placed the wedge in an oversized copper can which had been slit and opened on one end. After exposure, the piece was allowed to air cool. It was obvious that some material had flaked off but in our opinion was still not 100% clean. Because of the contamination problem and the results obtained, this is not considered as an acceptable approach.

Test No. 18

Salt Mixture 65% K_2CO_3 , 35% Li_2CO_3
Test Temperature - 1185°F
Soak Time - 1 hour

This salt combination is presently being used by Materials Feed Production Center (NLO) as the first step in their cleaning cycle. After running at the listed condition, the wedge was allowed to air cool rather than quench. The first examination prior to etching indicated no change in surface condition. The piece was then pickled for 5 minutes and only a small change was noted. We therefore concluded that these small changes in composition have no real significance.

Test No. 19

Salt Mixture - Park Chemical Co., K-3
Test Temperature - 1200°F
Soak Time - 1 hour

This time the salt mixture was from Park Chemical Co. and is listed as Type K-3 which is a neutral chloride as is the Houghton 980. These salts are considered as triple chlorides whose composition fall into the following ranges:

Sodium Chloride	21-23%
Potassium Chloride	31-34%
(Barium Chloride)	Balance

These are standard commercial salts and present no problems in handling procedures. This final trial was conducted at 1200°F with a time of 1 hour and allowed to air cool. Upon comparison with the effect of the Houghton 980 salts as listed in previous trials, we were unable to note of any difference in effects.

SALT TRIAL DISCUSSION

In review of the series of experiments it is apparent that no clear cut decision in absolute favor of one particular salt mixture over another was possible. We observed some minor improvements in some instances but under no conditions did we experience the strong flaking off of the magnesium fluoride or the penetration into the voids that was expected from the expansion and contraction of the derby. As was noted, in those areas where the contaminant was deeply enfolded there was some tendency for a softening of the solids.

The etching of the derby slices had no effect other than to allow for better examination of the surfaces, and since the direction of the program was towards the elimination of the use of acid, was not intended to be otherwise.

We can attribute no benefit to the increase in operating temperature over that required to maintain good fluidity of the salt solutions nor is there any suggestion that longer soak times results in any solubility of the magnesium fluoride.

While we did not achieve the firm definition and direction that was expected from the test program we did acquire sufficient data from which to draw upon permitting selection of the salt and operating conditions for the full scale derby experiments.

The ultimate decision as to the selection of the salts and the conditions under which the full scale trials were to be conducted was guided by the fact that two diverse types of salts should be evaluated. The chloride salts have a history of being used in heat treating applications and serve both to protect the surface at elevated temperatures and offering some cleaning action under certain conditions. The carbonate salts, as finally selected, are know to be compatible with the DU and had been used with some small success at other facilities.

Upon careful consideration and reexamination of the test specimens, the decision was made to use the Park Chemical Salt K-3, a triple chloride for one trial, and to use a mixture of 60% K_2CO_3 , 40% Li_2CO_3 for the second mixture.

SALT POT - FULL SCALE TRIALS

During the period while the laboratory trials were underway, a DIPEC search was initiated for a salt pot suitable for processing a full-size derby. The results of this effort directed us to the Army Methods and Material Research Command, located at the Watertown Arsenal, Watertown, Massachusetts. The pot itself was in excellent condition with inside dimensions of 27 inches x 50 inches x 20 inches deep. The unit was slightly radioactive by virtue of the fact that work with uranium had been conducted by the arsenal some years ago. The pot had been rebricked and not used again. The power supply, however, was badly damaged and incomplete. A completely new power supply was designed and built by William Duggan of Nuclear Metals, Inc. under this contract. Heating of the bath is accomplished by immersion blades as illustrated in Figures 5 and 6.

The program using full derbies which are typically 12-1/2 inches diameter by 18 inches long was carried out at Carolina Metals, Inc., a wholly owned subsidiary of Nuclear Metals, Inc. located in Barnwell, South Carolina. Derby metal for all Nuclear Metals, Inc. programs is produced at this facility.

Due to the dimensions of the salt pot we were restricted to processing one derby at a time. The weight of the derby being in the range of 1400 pounds required the design and fabrication of a special cradle of stainless steel

which would permit safe handling with an overhead crane and which could be immersed into the salt baths at elevated temperatures. The cradle and lifting bar is shown in Figure 7.

The program called for four derbies to be processed, two each in two different salt mixtures. Four derbies were taken directly from the knock-out station following their removal from the reduction vessels. These derbies were not cleaned in any manner once having been assigned to the program.

The cycle for testing was identical to that used in the laboratory trials. The derby must first be preheated to insure a completely dry surface. As had been mentioned previously, the accidental introduction of moisture being trapped in a molten bath could result in a most violent reaction.

Preheating was done using a bakeout oven that is normally used to dry reduction vessels. The derby was then loaded into the cradle, Figure 8, and transported by overhead crane to the salt bath. With the mass of the derby being so great, the heating cycle would consist of one hour soaking to allow the bath temperature to stabilize and then to maintain this condition for a two hour period. The assembly is lowered into the bath, Figure 9, and soaked for the prescribed time. Upon removal, the derby is up-ended and quenched as shown in Figure 10. When the outer shell darkened, the derby was lifted clear of the tank allowing the residual heat to expand the shell for a second time. Upon observing the water surface below the derby, we saw no indication of any spalling action of the MgF_2 . After allowing sufficient time, the derby was quenched for the final time. This same procedure is then followed for the second derby.

The first two derbies were heated in the Park Chemical Company's neutral salt K-3. After completing these first trials, the temperature of the bath was then raised so we would have extra time available to bail out the molten salt into 55 gallon steel drums in preparation for charging with the second salt bath which was 60% K_2CO_3 , 40% Li_2CO_3 , Figure 12.

Once the carbonate bath stabilized, the same preparation and charging procedures were followed as with the first mixture. At the time of quenching there were no visible signs of any material flaking from the surface of the derbies.

All four salt cleaned derbies were washed with hot water and brushed to remove any loose particles. The surfaces were carefully examined to determine the effects of the salt immersion and quenching. Neither salt, in our opinion, resulted in an improvement in the surface condition of the derbies that was considered acceptable to permit melting and casting. The decision was therefore made to acid pickle and mechanically remove as necessary the combination of the visibly crusted salts and adhering chunks of MgF_2 . Immersion of the derbies in the salts, in the opinion of the operators, actually made it more difficult to clean the surfaces than the effort normally required at this point in the processing. This confirmed our finding on the laboratory tests.

The processed derbies were shipped to Nuclear Metals, Inc. for melting and further examination. Each derby was melted with qualified recycle material to make a full charge of 800 kg. Standard melting and processing in accordance with M833 specifications were followed up to and including ultrasonic testing, the decision point for comparison with standard procedures.

HIGH PRESSURE WATER CLEANING

As had been briefly mentioned in the early text, consideration of an entirely different method of cleaning, namely high pressure water, was a serious possibility. In the laboratory trials and finally right up to the full size derby cleaning trials, there were no positive indications that the salt baths were a solution to the problem.

During the period of these trials, discussions were held with representatives of Flow Systems, Inc. of Kent, Washington, who make a high pressure (30 to 50 thousand psi) water cleaning jet system. They examined the derby sections on which we had conducted our trials and it was their considered opinion that the removal of contamination from the pockets and seams would be less of a problem than problems they had encountered with other materials. Based on their experience with seemingly more difficult applications of this method, we requested that we be allowed to explore this method under the program.

We first attempted to locate a facility which had a license for uranium which would permit some trials to be conducted before any real commitment. None could be located. With the cooperation of Flow Systems, Inc., we then requested the ARDC and PCO for approval of a rental agreement which would permit the processing and testing to be conducted at Nuclear Metals, Inc. The pumping system and all associated equipment was to be installed in a "cold" area and only a pressure line and the power head wand would be within the restricted area, subject to possible contamination. The request was approved and the equipment installed. A proper steel protective chamber was fabricated within which the actual cleaning would take place.

The high pressure system consists of three major components: (1) the motor driven pump, (2) two parallel filtration units, and (3) the accumulator. These components are shown in Figure 13.

The water pressure intensification system is shown in Figure 14. The hydraulic fluid acting on the large piston results in water pressure intensification equal to the hydraulic fluid pressure times the ratio of the area of the large piston to the small piston. Therefore, the water pressure is controlled by controlling the hydraulic fluid pressure. The inlet water enters the intensifier via the inlet check valve (1) and exits via the outlet check valve (2) to the accumulator (3). At the end of each stroke, the hydraulic fluid flow control valve is shifted to direct the flow to the other side of the hydraulic piston. The accumulator is a large reservoir of pressurized water whose function is to attenuate the pressure pulsation resulting from the reciprocating operation of the pistons and the opening and closing of the check valves.

The cleaning of the derby is quite simple in that the water is delivered by high pressure tubing to a trigger operated nozzle. The unit was on a swivel arm permitting great flexibility in directing the flow.

In cleaning the derbies, only water was used. While it is true that the use of an abrasive could possibly decrease the time, the prime concern was to not subject the depleted uranium to any possible source of contamination which might be carried into the melting operation.

Careful examination of the cleaned surface conducted by Nuclear Metals' personnel familiar with seeing production derbies all concurred that the surface and those areas where there were folds were extremely clean and free of visible contamination. The ARDC Project Engineer was present during the first trials.

Based on our experience at this point with the good cleaning, a judgement was made to continue with the high pressure approach in the proveout phase of the program. By doing so we had at this point established a procedure which eliminated the use of acid which was the prime objective of the investigation.

Figures 15 and 16 illustrate the clean and highly reflective surfaces which result when impacted by high pressure water. These are typical of all the derbies cleaned by this procedure.

DERBY CLEANING VERIFICATION PROVE OUT

Contract requirements were that the optimal process, in this case the high pressure water, be used to prepare derbies for five heats. The five heats were to be processed to finished M833 penetrators per MIL-C-63422 with acceptable penetrators entering the stockpile.

The derbies were taken directly from production with no consideration as to initial surface conditions and were not cleaned in any manner. We were therefore obliged to deal with all possible facets of cleaning from a relatively smooth surface to folds in the metal with entrapped MgF_2 . The cleaning of the derbies was uneventful. By adjusting the position of the derbies in the cleaning chamber we were easily able to blast all faces clean of any foreign matter. As we were under no time constraints, we dried the surface with compressed air which permitted a better inspection of the surface.

For ease in identification and to keep a separate system from standard production material the melts made from the derbies were assigned "M" Lot numbers as follows:

<u>Derby No.</u>	<u>"M" Lot No.</u>
5173	M-65
5175	M-66
5166	M-67
5172	M-68
5174	M-69

A standard melt charge of 800 kg consisted of (1) derby, (2) T1 for alloying, (3) nine chemically acceptable butts, and (4) Class A recycle. Since we wished to maximize the information on this specific cleaning procedure the melts were processed in the following manner.

Heat No.Composition

M-65	Derby 5173 + Ti + Class A recycle only
M-66	Derby 5175 + Ti + M-65 butts + Class A recycle
M-67	Derby 5166 + Ti + M-66 butts + Class A recycle
M-68	Derby 5172 + Ti + M-67 butts + Class A recycle
M-69	Derby 5174 + Ti + M-68 butts + Class A recycle

Using this procedure, any impurities which normally float to the top of the cast ingots would then be carried on to the next melt. While this is only a small sampling, it did give us control of the series.

In keeping with the program, the complete chemistry of every derby is presented in Table 2.

Rod chemistry of lots M-65 through M-69 from which finished penetrators were machined is presented in Table 3.

ULTRASONIC TESTING

Ultrasonic testing of the blanks was done on a four channel unit in the manual mode. This system is manufactured by Automation Industries. The unit addresses the pulse echo which is set at 90 degrees and is a centerline trace for the detection of voids or inclusions. The offset channel detects imperfections of the rod near the surface and is also at 90 degrees to the rod but displaced by 0.27 inch. The last two channels which are left and right shear are set at 45 degrees to the center line and read through the depth of the sample.

In particular with the pulse echo channel, which is oriented at 90 degrees to the blank on the centerline, when the indication of a fault is recorded, one cannot establish whether that fault is a void or inclusion unless a section is taken for metallographic examination. However, during the solidification in the molds, inclusions tend to float to the top. As a result of this, the rear of the extruded rod is expected to have a higher density of inclusions than the front. In line with this data gathered over many production runs representing a very large sampling, it has been clearly established that the last one or two blanks cut from the extruded rod have higher rejection levels. This same type of behavior is indicated in the data presented in Table 4 with respect to this program.

Considering that only 5 heats were involved in the production proveout portion of the program, we find that the type of reject encountered and the percentages are within the scope of standard production material.

CHEMISTRY

All chemistry analyses was carried out in accordance with MIL-C-63422B as directed in the M833 program.

The two different salt baths used for cleaning were analyzed for uranium. Both results were less than 10 ppm. Since each bath was exposed to only two derbies each, this was expected. However, should such baths be subjected to heavy usage, it would be reasonable to assume that baths would have to be monitored and replaced when the uranium content increased appreciably.

The chemistry results reported in Table 2 were obtained by the following sampling technique. In the case of the derbies, a 3/4" hole is drilled into the top surface. The chips generated in the first 1/2" are discarded, then those chips coming from the next 1/2" to 3/4" are carefully collected into a clean container for analysis. This same method is then used on the side surface at approximately the mid-point of the derby.

The rod chemistry as reported in Table 3 is the result of cutting a slice from the extruded rod at the position of the first penetrator blank.

The data reported is all well within the specification for the M833 program and in the normal range of standard production material.

The prove out lots "M" 65, 66, 67, 68 and 69 were machined into finished penetrators and will be delivered to the Army as directed in this program.

METALLOGRAPHY

One butt from each of the prove out melts was sectioned for macro examination. Figures 17, 18, 19, 20, and 21. Evaluation of the material and the amount of oxides and carbides present was the same as has been observed in many other sections of standard production melts. Since we did exercise control in that the individual melts contained the butts from the preceding melt, the original expectation was that we might observe a slight increase in the last melt. This was not the case; therefore, we must consider that either the size of the sampling did not allow for a visible change, or that generally a certain portion goes off with melt dross and impurities do not build beyond the levels seen.

Micro specimens were taken from the very end of three of the butts as shown in Figures 22, 23, and 24. Examination shows the material to have a large amount of oxides and carbides with some areas of an unidentified material. This is in keeping with other examinations of production butt ends and appears to be about the same level.

DISCUSSIONS AND CONCLUSIONS

From the experimental work conducted under this program, it has been demonstrated that the heating of DU derby in various types of salt bath combinations for the dual purpose of effecting some degree of cleaning from the expansion and contraction of the metal and obtaining some degree of solubility was not effective. Only minor differences could be attributed to this approach, all of which in themselves were not adequate to justify the melting of a derby without a further mechanical or chemical cleaning operation.

At this time, other facilities which produce DU metal by the magnesium reduction method use a combination of molten salt, mechanical and chemical approach, or surface oxidation at elevated temperatures.

As defined, the purpose of this program was to develop a cleaning procedure which would eliminate the use of acid cleaning methods and their accompanying problems. The alternative approach presented by Nuclear Metals, Inc. of removing surface contamination by the use of high pressure water was predicated on a like technique being used successfully in other industrial applications for surface cleaning. The very high pressures made possible by this system coupled with the flexibility of direction of the discharge wand enabled the operator to attack the crevices and folds allowing the stream to cut deeply into the entrapped foreign material of whatever type. With other techniques, this entrapped material could only be removed by extensive picking before it could be loosened and possibly removed. Since the entire derby is subject to the acid bath, this results in a considerable amount of metal being dissolved and wasted.

With high pressure water, metal loss in the cleaning operation is no longer of concern, and since the cleaning operation involves only water without the use of any abrasive additives, there is no risk of contamination of the metal. It was our experience that derby metal cleaned in this manner developed a matte finish and color not unlike that of stainless steel. No discoloration occurred upon storage of over a two week period.

Finally, since the objective of cleaning without the use of acid has been demonstrated and the melting and processing in accordance with the M833 specifications has resulted in acceptable penetrator production, we strongly recommend the implementation of the high pressure water method of cleaning into the production process.

There are commercial filtering systems available which have the capability of removing solid contaminants to such a fine degree as to permit the recycling of the water. This approach when used in conjunction with this operation would permit the consolidation of the contaminating material consisting mainly of MgF_2 with small amounts of DU, thereby greatly reducing disposal costs.

Table 1. Experimental Salt Trials

Specimen No.	Salt Mixture	Temp. (°F)	Time at Temp. (hrs)	Quenched
0	Etched Control	RT	-	-
1	Houghton 980 (3 Cl)	1100	2	Y
2	Houghton 980 (3 Cl)	1400	2	Y
8	Houghton 980 (3 Cl)	1200	2	Y
9	Houghton 980 + 5% H ₃ BO ₃	1200	2	Y
0-10	Houghton 980 + 5% H ₃ BO ₃	1400	2	Y
11	Houghton 980 + 10% H ₃ BO ₃	1400	2	Y
3	60% K ₂ CO ₃ + 40% Li ₂ CO ₃	1200	2	Y
1-4	60% K ₂ CO ₃ + 40% Li ₂ CO ₃ (2nd time)	1200	2	Y
5	60% K ₂ CO ₃ + 40% Li ₂ CO ₃	1400	2	Y
6	60% K ₂ CO ₃ + 40% Li ₂ CO ₃ (repeat of No. 1-4)	1200	2	Y
7	60% K ₂ CO ₃ + 40% Li ₂ CO ₃ + 5% H ₃ BO ₃	1200	2	Y
18	65% K ₂ CO ₃ + 35% Li ₂ CO ₃ (present NLO)	1185	1	Air cool
13	70% K ₂ CO ₃ + 30% Li ₂ CO ₃ (3 etches)	1200	2	Y
14	70% K ₂ CO ₃ + 30% Li ₂ CO ₃	1200	2	Y
15	70% K ₂ CO ₃ + 30% Li ₂ CO ₃	1200	4	Y
16	70% K ₂ CO ₃ + 30% Li ₂ CO ₃	1400	2	Y
17	Oxidized - (Eldorado method)	1000	1	Air cool
12	Oxidized - (Furnace problem)	820	20 min.	Air cool
19	K-3 Neutral (3 Cl)	1200	1	Air cool

Table 2. Derby Chemistry

Derby No.	"M" Lot	(ppm)					
		C	Fe	Ni	Cu	Si	
Salt Trials							
3692	45	13	17				
3696	46	49	5				
3693	47	19	5				
3697	48	37	13				
High Pressure Water Trials							
4729	52	35	29				
4739	53	68	8				
Production Prove Out							
5173	65	23	11	20	7	25	Top
		36	7	20	8	27	Side
5175	66	28	10	33	11	22	Top
		25	12	39	11	27	Side
5166	67	35	10	24	10	11	Top
		31	9	24	10	14	Side
5172	68	43	12	24	9	39	Top
		22	8	27	9	14	Side
5174	69	38	11	19	5	30	Top
		44	8	14	5	30	Side

Table 3. Rod Chemistry

"M" Lot	w/o		(ppm)				
	Ti	C	H ₂	Fe	Ni	Cu	Si
Salt Trials							
45	0.73	0.003	0.4	18	8	3	45
46	0.72	0.003	0.3	7	5	2	49
47	0.73	0.004	0.2	10	9	1	32
48	0.74	0.003	0.4	12	6	2	54
High Pressure Water Trials							
52	0.55	0.002	0.3	30	6	5	47
53	0.71	0.002	0.4	13	5	7	56
Production Prove Out							
65	0.75	0.003	0.2	18	17	7	49
66	0.74	0.003	0.2	16	29	11	61
67	0.73	0.004	0.5	19	22	10	31
68	0.73	0.003	0.4	21	23	8	27
69	0.73	0.005	0.5	14	14	8	35

Table 4. Ultrasonic Test Data

Lot No.	Lot Size	Percent Reject	Blank No.	Pulse Echo	Rejects		Offset
					Left Shear	Right Shear	
Salt Trials							
M-45	110	1.8	4-8 6-13	X	X		
M-46	113	1.7	6-13 7-7	X		X	
M-47	113	0.8	1-13	X			
M-48	111	0.9	5-13		X		
High Pressure Water Trials							
M-52	103	0.9	1-1			X	
M-53		2.7	4-12 5-10 5-12	X X X			
Production Prove Out							
M-65	113	0.0					
M-66	112	7.1	1-7 2-13 3-10 5-11 5-12 5-13 6-13 7-12	X X X X X X	X X	X X X X	
M-67	110	3.6	1-13 2-13 6-12 9-13	X X X X	X X	X X	
M-68	112	2.6	1-8 3-10 7-9		X	X X	
M-69	113	1.7	4-11 6-6	X		X	

Table 4. Ultrasonic Test Data (continued)

Lot No.	Lot Size	Percent Reject	Blank No.	Pulse Echo	Rejects		Offset
					Left Shear	Right Shear	
Production Comparison Lots							
589	112	4.4	3-13	X			
			8-10			X	
			8-13	X		X	
			9-13	X			
			6-13	X			
572	114	3.5	1-13	X			
			2-13	X			
			5-13	X			X
			8-13	X			
619	110	1.8	3-5	X			
			7-1				X

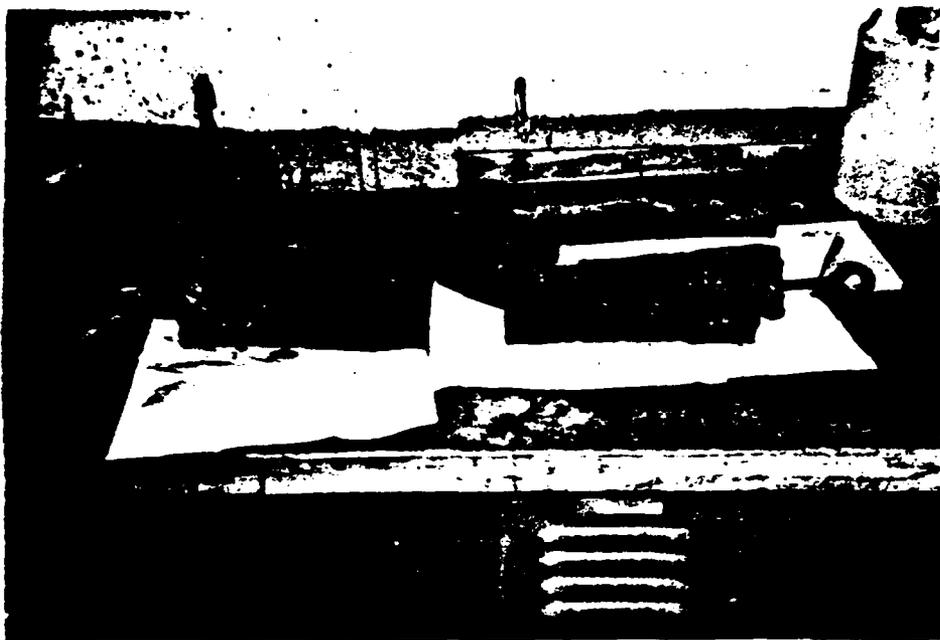


Figure 1. Derby Wedge Test Piece

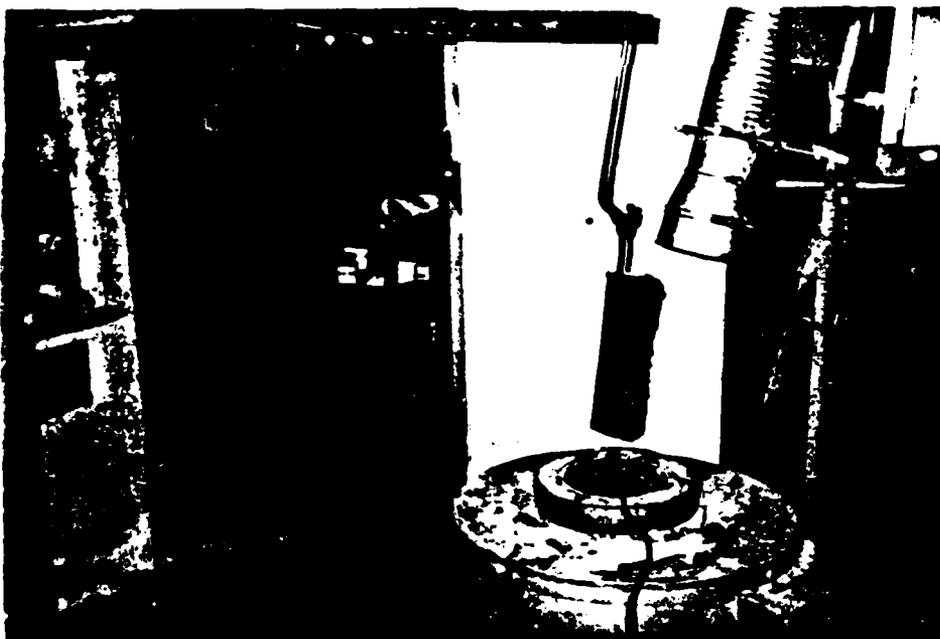


Figure 2. DU Wedge into Salt



Figure 3. DU Wedge Immersed in Salt

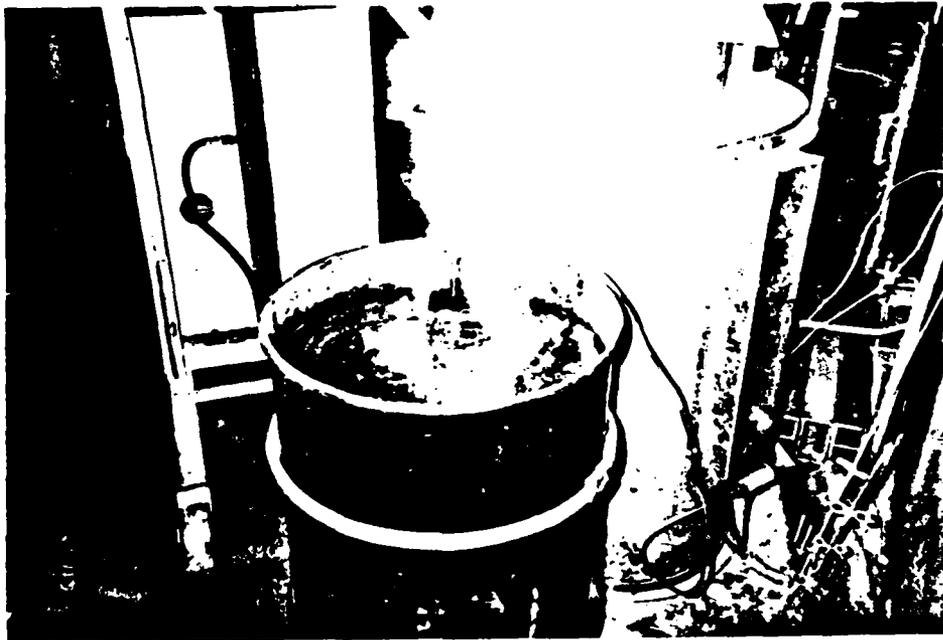


Figure 4. Water Quench

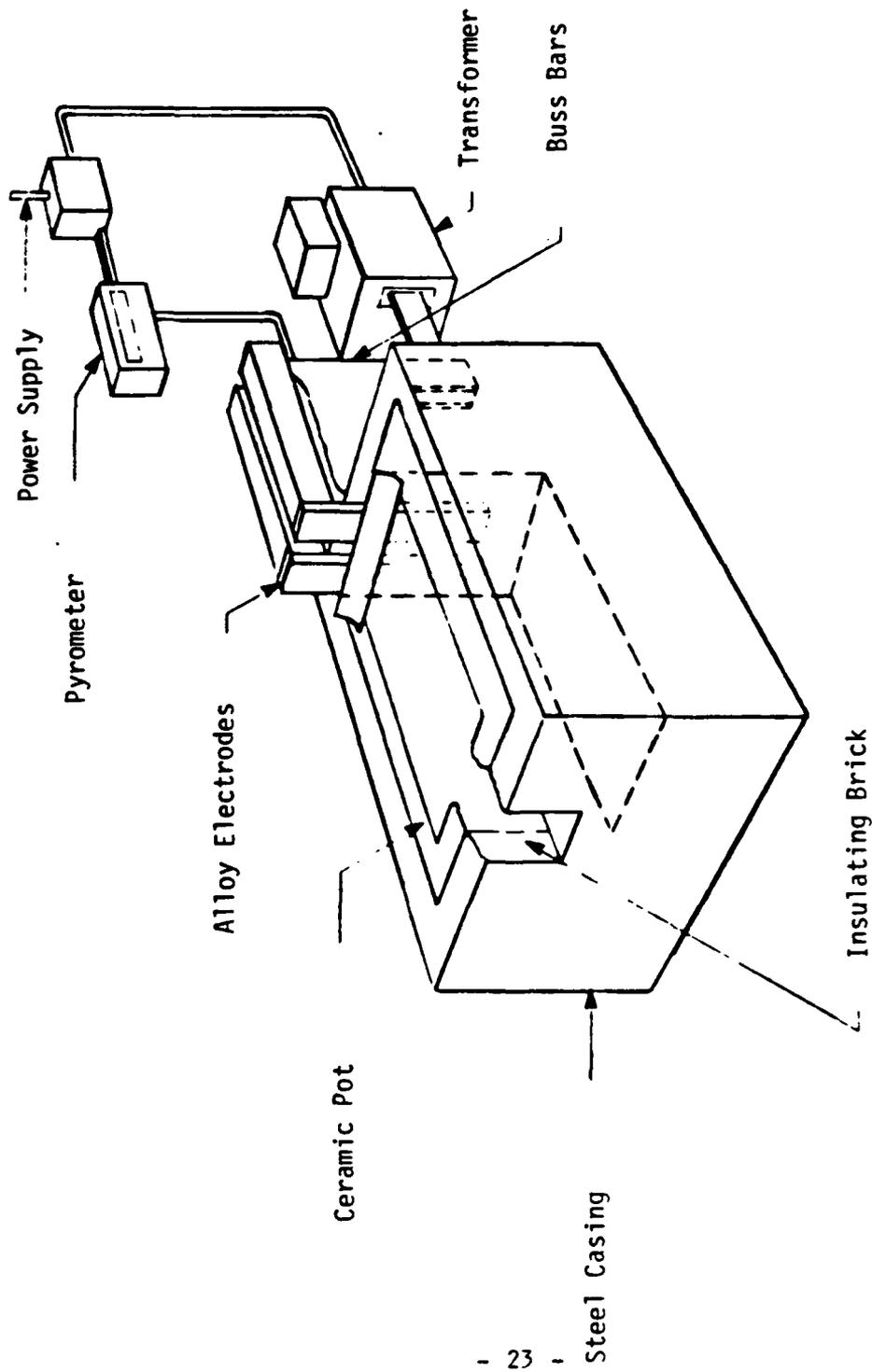


Figure 5. Internally Heated Salt Bath

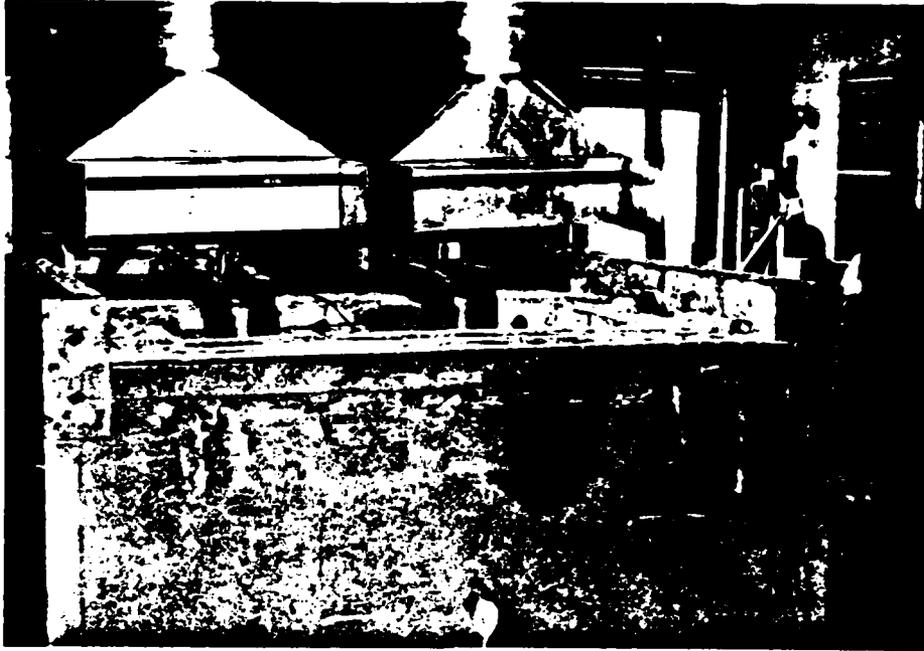


Figure 6. Salt Bath at CMI

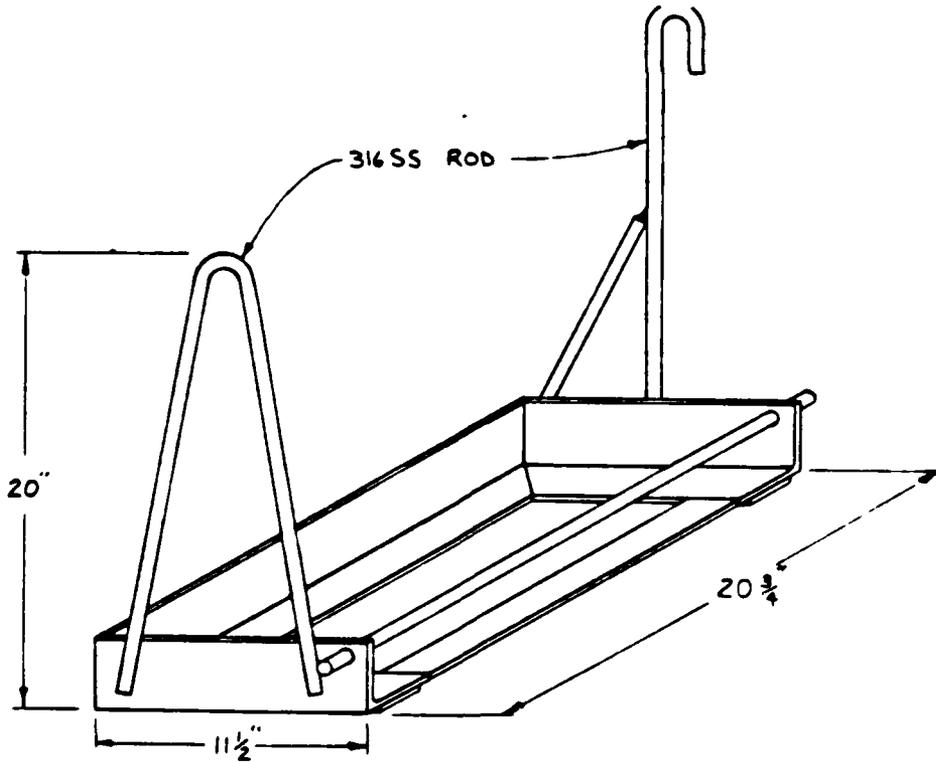


Figure 7. Derby Cradle

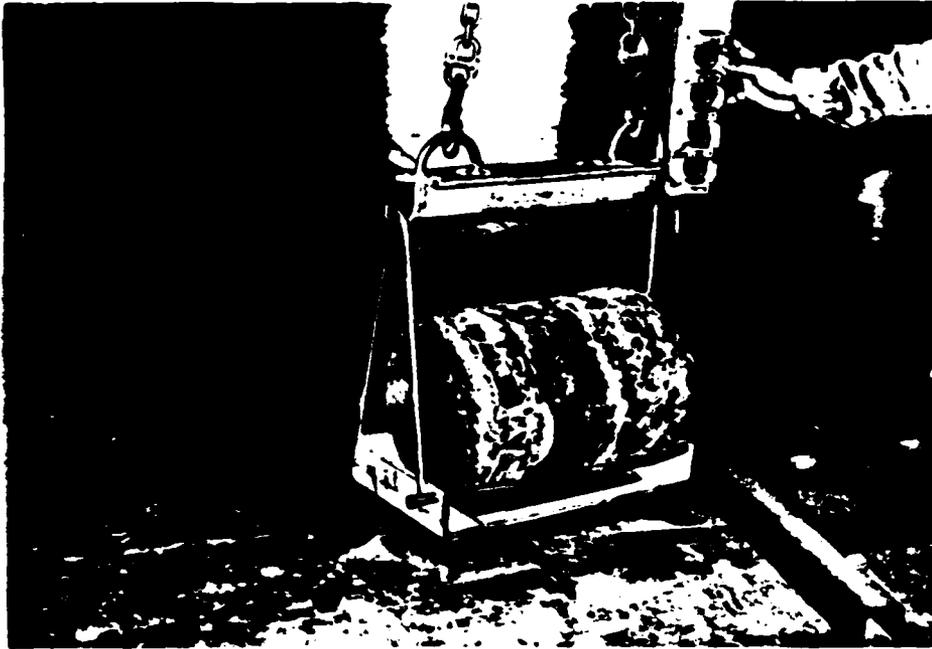


Figure 8. Derby Handling



Figure 9. Salt Pot Loading



Figure 10. First Derby Quench

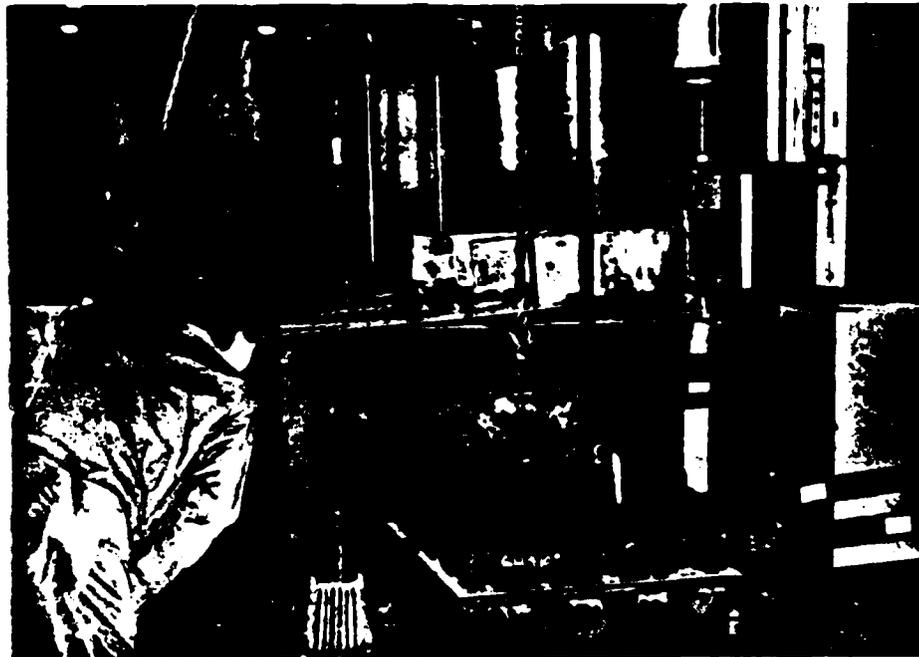


Figure 11. Second Quench and Inspection

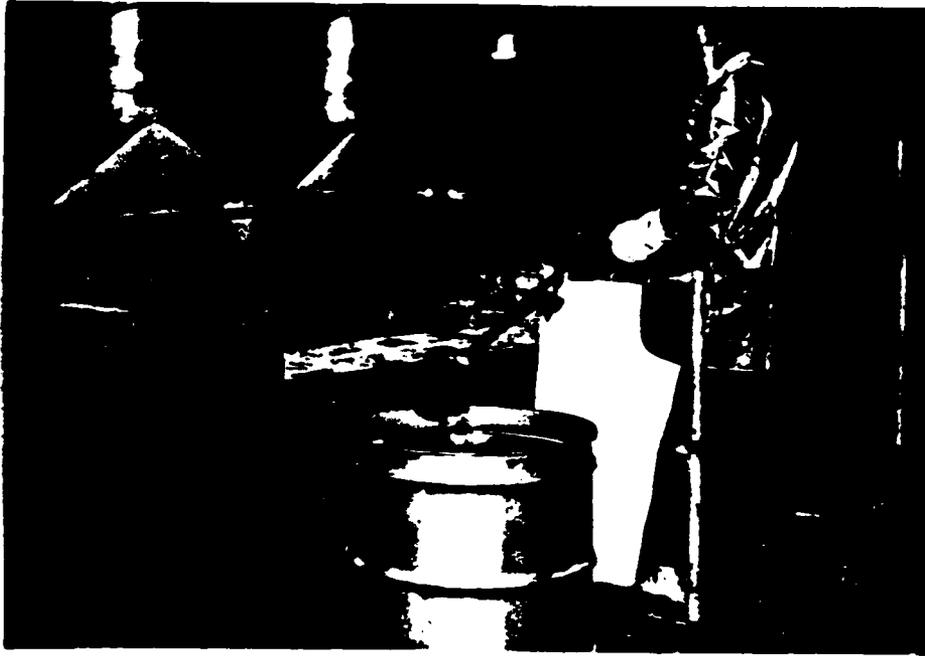


Figure 12. Bailing Molten Salt for Second Bath

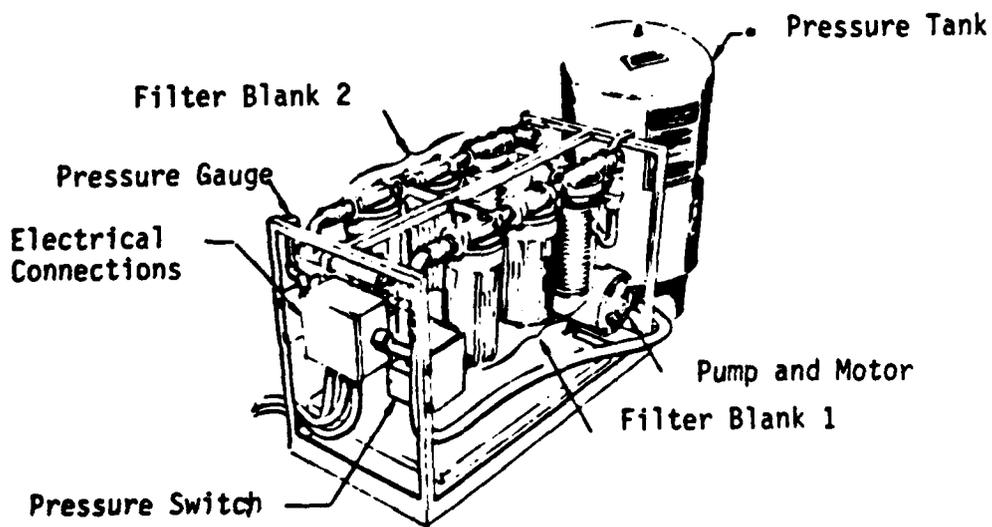
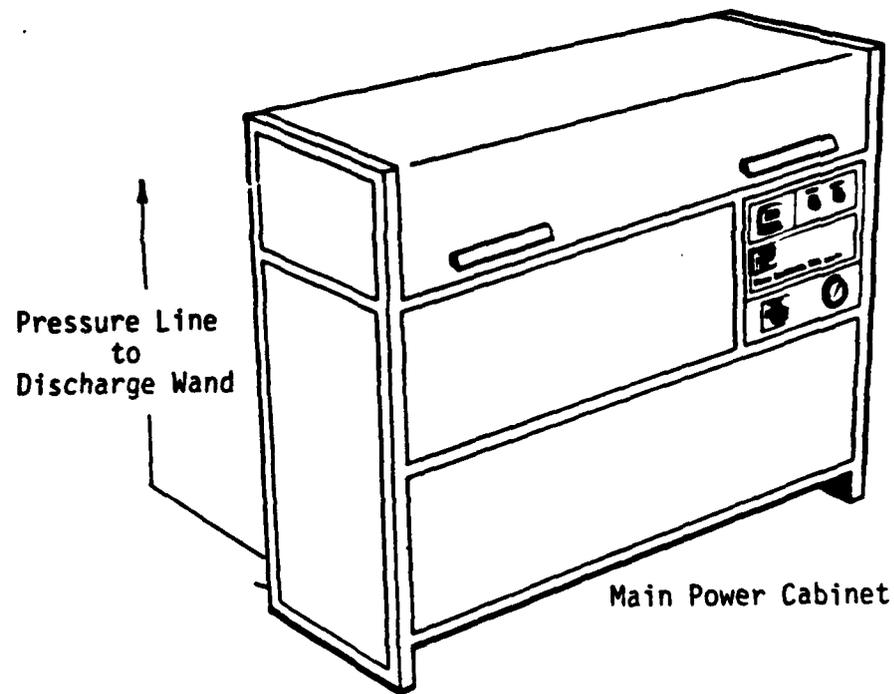


Figure 13. High Pressure Water System

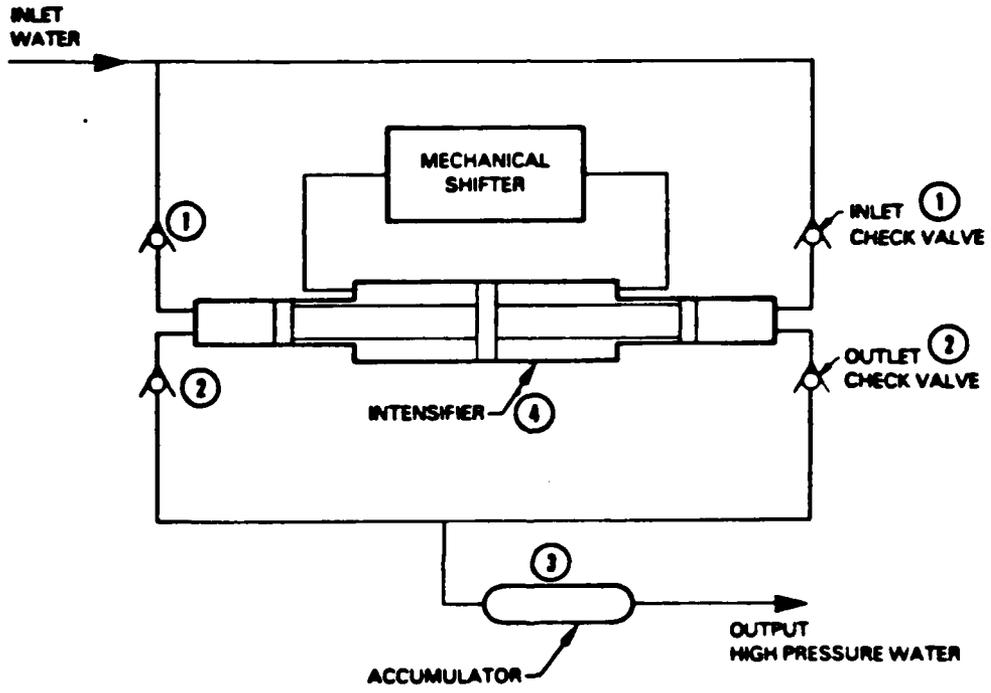
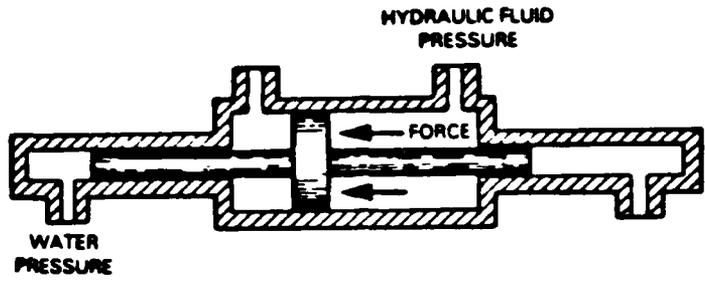


Figure 14. High Pressure Water Intensification System

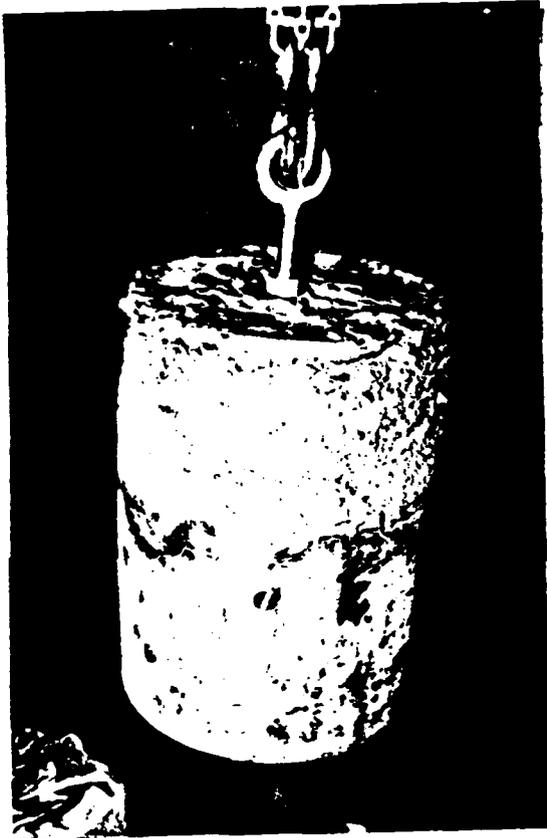


Figure 15. M-65



Figure 16. M-66

Derbies Cleaned by High Pressure Water Only



Figure 17. Butt M-65

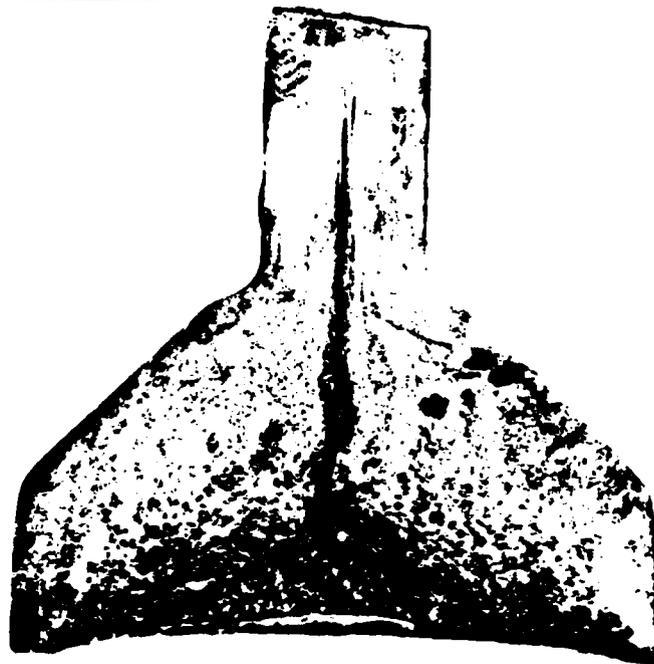


Figure 18. Butt M-66

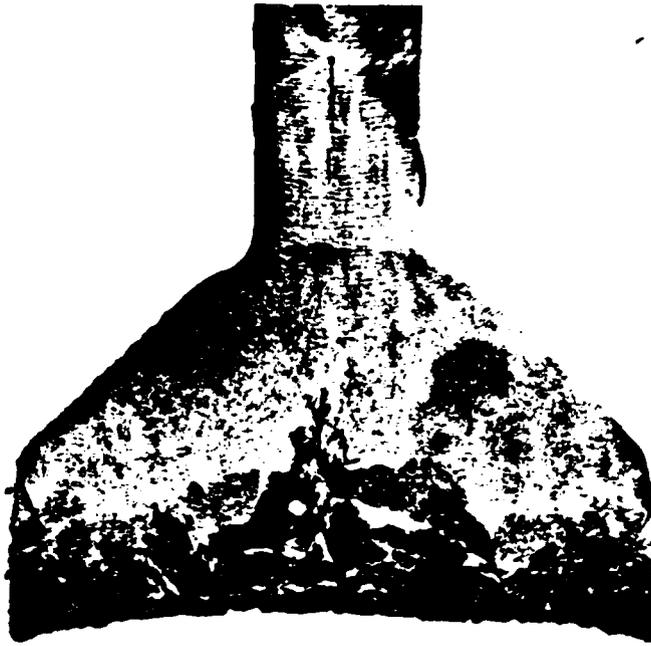


Figure 19. Butt M-67

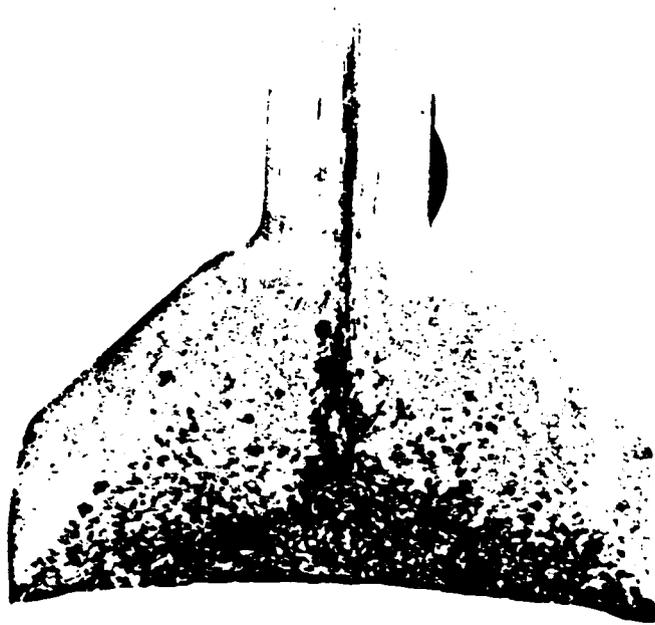


Figure 20. Butt M-68



Figure 21. Butt M-69

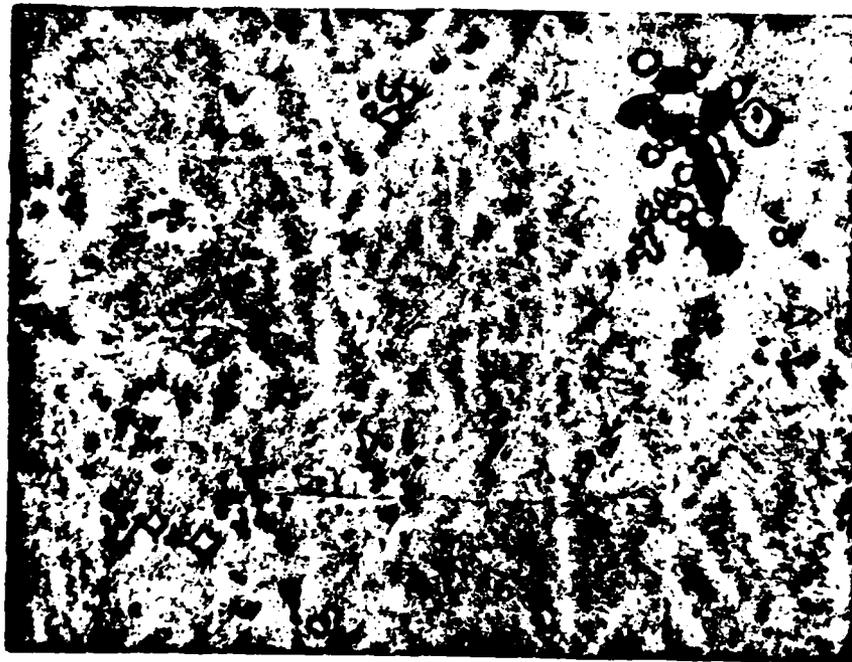


Figure 22. Photomicrograph M-65, 500X Typical Area



Figure 23. Photomicrograph M-66, 200X Typical Area

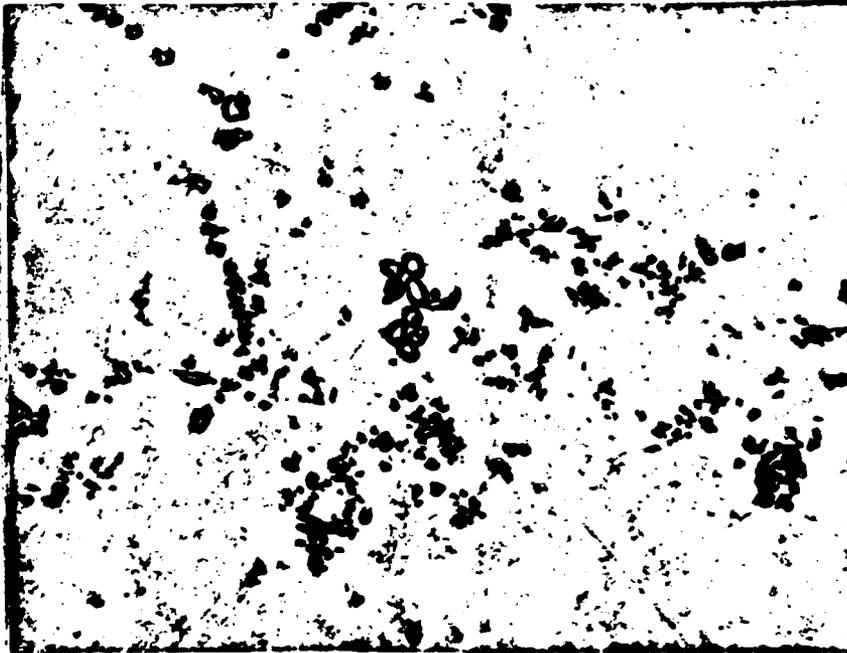


Figure 24. Photomicrograph M-68, 250X Typical Area

APPENDIX A

HEALTH AND SAFETY

This qualitative safety evaluation report has been prepared to satisfy a Contract Data Requirement for Contract No. DAAK10-84-C-0087.

The safety evaluation conducted as a part of this effort was administered through the Nuclear Metals, Inc. (NMI) Health and Radiation Safety Program. The specific operation/operator(s) was issued a Radiological Work Permit (RWP) in order to identify the actual and potential radiation hazards associated with the particular operation and the precautionary measures (including the use of protective equipment) to reduce the potential for exposure to these hazards.

The safety related facilities and equipment designed to reduce the potential and severity of any hazards occurring from this effort included:

- Proper protective clothing, gloves, and face shields when engaged in any operation utilizing either acids, molten salts, or high pressure water in the derby cleaning operations.
- Monitoring by air sampling of the areas in which the cleaning operations were conducted.
- Periodic surveillance by personnel of the Health and Safety Department as to the procedures used in the program.
- Once cleaned, the derby required no special handling or storage facility.

In review of the safety related measurements taken during this effort and the observations of the Safety Department personnel, that data indicates that the procedures used in the program do not result in a substantial increase in the airborne concentrations of depleted uranium in the work areas and that the operators' external exposure was maintained well within established guidelines. The results of air sampling and measurements of operators' external exposure were less than 10 percent of our license limit for airborne concentrations and external exposures respectively.

All procedures and health and safety related evaluations were conducted in compliance with our Source Material License SMB-179 issued by the U.S. Nuclear Regulatory Commission dated 23 May 1984.

APPENDIX B

ECONOMIC ANALYSIS

The result of this MM&T recently completed by Nuclear Metals, Inc. supports the adoption of this process of cleaning DU derby with high pressure water spray. The elimination of Nitric Acid from the derby cleaning process has two immediate benefits. First is the acid cost and subsequent waste processing of this media. Second is the DU saved which is normally consumed by the acid during the soaking cycle. The acid eats away the DU surface of pockets or folds which entraps contaminants, specifically MgF_2 , and allows the contaminants to fall away or to be removed by mechanical agitation. The process while effective is not discrete and an average of fifteen (15) pounds of DU is consumed by the acid for each derby cleaned.

The additional 15 pounds of DU derby weight would reduce the recycle material weight by 15 pounds for each melt charge. The recycle material savings will facilitate an additional 100% recycle melt at the 10,000 core MOB rate production per month.

The current acid pickling station would be replaced by a closed loop high pressure water cleaning station. NMI has solicited and worked with Flow Systems, of Kent, Washington to develop specification outlines for the process application. The equipment required to achieve the cleanliness desired and to support the product through put required would cost approximately \$112,650 installed. This system would consist of a Dual Intensifier, 40,000 psi Waterknife pump, Water Booster and Filtration System, Stand Plumbing Package and Jetwand Cleaning Assembly with plumbing package. The water cleaning station would physically replace the inline production acid etch station and utilize the material handling system.

The Economic Analysis is based on a production (MOB) rate of 10,000 cores per month. The following are the elements of cost savings:

<u>Material</u>	<u>Current</u>	<u>Proposed</u>	<u>Delta</u>
Total Derby w/overhead	\$462,136	\$458,298	\$ 3,838
<u>Other Direct Cost</u>			
Waste Disposal	<u>118,124</u>	<u>116,359</u>	<u>1,765</u>
Total Savings (A)			5,603
MOB Rate (B)B			10,000
Savings Per Core (C)			.5603
A/B = C			
Facilities Dollars (D)			\$112,560
Payback in Cores (E)			201,053
D/C = E			
Payback in Months at MOB Rate (F)			20.1
E/B = F			

ASSUMPTIONS

The total labor requirement to support the current acid pickling process and mechanical agitation to remove contaminates and the labor to support this process change would be offsetting.

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