

## THE RECYCLE OF DEPLETED URANIUM WASTE PRODUCTS BY A HYDROMETALLURGICAL PROCESS

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### ABSTRACT

Nuclear Metals, Inc. has developed a process for recycling uranium scrap materials into high quality metal. The process involves the dissolution of scrap metal in an aqueous solution of 2.4 N HCl and 0.16 N HBF<sub>4</sub>, followed by precipitation of UF<sub>4</sub> through the addition of HF. The precipitated green salt is filtered, washed, dried, and heat treated after which it is suitable for reduction to metal. The product and the process are referred to as Hydromet, since it is a hydrometallurgical approach to producing green salt. Conventionally, green salt is produced by a pyrometallurgical technique. The steps of the process are described and results presented for derbies produced using Hydromet green salt. With proper process selection and appropriate heat treatment, green salt produced by Hydromet is fully equivalent to pyrometallurgical green salt. Hydromet green salt can be reduced to metal using the identical process used for pyromet green salt. Good quality, well-formed derbies can be readily produced.

### INTRODUCTION

The most commonly used method for producing uranium metal is by magnesiothermic reduction of uranium tetrafluoride (UF<sub>4</sub>). The basis for this process is rooted in the technology of the nuclear fuel production cycle. Naturally occurring uranium contains only about 0.7 percent U<sup>235</sup>; the balance is primarily U<sup>238</sup>. To produce reactor grade uranium, the concentration of the fissionable U<sup>235</sup> isotope must be increased to approximately 3 percent. This enrichment is accomplished by gaseous diffusion of uranium hexafluoride (UF<sub>6</sub>). The product of the diffusion process is a stream of UF<sub>6</sub> enriched in U<sup>235</sup> and a stream of UF<sub>6</sub> tails depleted of U<sup>235</sup>.

To produce depleted uranium metal (DU), depleted UF<sub>6</sub> is reduced to UF<sub>4</sub> by hydrogen reduction, and the UF<sub>4</sub> reduced to metal by magnesiothermic reduction. UF<sub>4</sub> is a fine green crystalline powder called green salt. Because hydrogen reduction of UF<sub>6</sub> involves high temperature reaction of gas phases, the product is referred to as pyrometallurgical green salt or pyromet. Green salt is blended with high purity magnesium metal, packed in a retort lined with a graphite crucible, sealed, and placed in a furnace. The retort is heated until spontaneous reaction occurs between the magnesium and the green salt. This reaction is highly exothermic, producing molten uranium metal which settles to the bottom of the crucible and MgF<sub>2</sub> slag, which separates above the melt. The solidified metal or derby is then cleaned, vacuum induction remelted and cast into shapes or billets for further processing.

The manufacture of depleted uranium metal products results in the generation of a number of waste streams such as saw chips, machine turnings, and grinding swarf. Typically this type of scrap is heavily oxidized and contains high amounts of impurities. Direct remelting of this scrap is not feasible. Vacuum induction melting does not effect much purification or

removal of impurities. As a result, specifications for uranium metal usually preclude the use of scrap that can lead to the introduction of oxides and other types of impurities in the metal. Therefore, low grade metal scrap such as turnings, chips and swarf cannot be directly recycled by remelting.

Depleted uranium is classified by the NRC as a low level radioactive material, and as such, all scrap must be disposed according to NRC regulations. Historically DU scrap has been disposed by encapsulation and burial in an approved and licensed burial site. However, regulatory trends and general availability of suitable burial sites will limit the amount of material that can be buried in the future and greatly increase user fees.

An alternative approach for dealing with scrap has been developed by NMI. This process converts scrap DU into high purity UF<sub>4</sub>. The purification inherent in the production of UF<sub>4</sub> by this process makes the green salt suitable for recycle into the process stream for uranium metal production. The process is based on hydrometallurgical techniques for producing UF<sub>4</sub>, and the product is referred to as hydrometallurgical green salt or Hydromet. The quality and characteristics of Hydromet UF<sub>4</sub> are comparable to pyromet UF<sub>4</sub>, and Hydromet can similarly be reduced to metal by magnesiothermic reduction. The Hydromet process is suited to uranium and uranium alloys and has been successfully demonstrated on unalloyed uranium, U-3/4Ti, U-2Mo and U-6Nb alloys.

### HYDROMET PROCESS

Hydromet is a process for converting uranium metal into high purity green salt. The process involves dissolving uranium metal in a dilute solution of a non-oxidizing mineral acid such as hydrochloric acid or sulfuric acid, filtering out the insolubles, and precipitating the dissolved uranium as UF<sub>4</sub> through the addition of hydrofluoric acid. The precipitate is filtered,

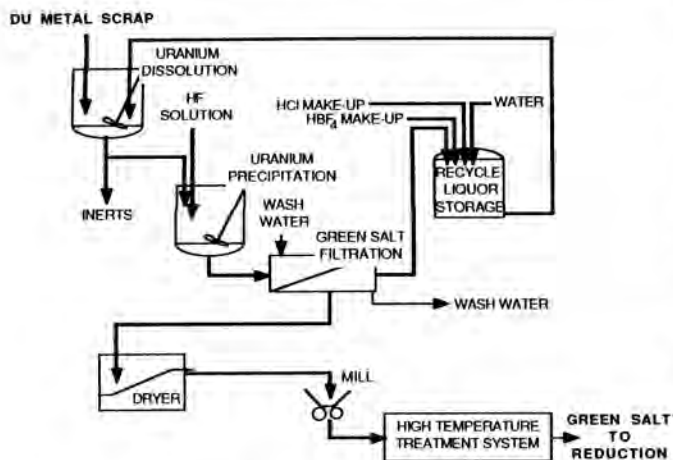
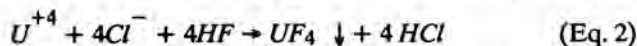
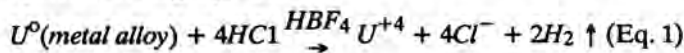


Fig. 1. Hydrometallurgical green salt production process.

washed and dried. The dissolution acid is regenerated and recycled. The Hydromet process inherently purifies the  $UF_4$  of alloying elements, tramp elements and impurities.

The chemistry of the Hydromet process can be described by the following equations:



A schematic of the process is shown in Fig. 1. Key features of the process are covered by several U.S. Patents (1,2,3). Uranium metal is dissolved in an aqueous solution of water, hydrochloric and fluoboric acids producing  $U^{+4}$  and  $Cl^-$  ions and releasing hydrogen gas. Insolubles settle to the bottom of the dissolution tank and are removed by filtering the solution or pumping the bottom of the tank.  $HBF_4$  prevents formation of pyrophoric suboxide sludges and increases the rate of metal dissolution. The liquor is pumped into a second tank where HF is added to precipitate  $UF_4$ . The precipitation reaction is controlled by temperature, rate of HF addition, and amount of stirring in the tank. The  $UF_4$  is filtered from solution and washed to remove impurities. The filtrate is recycled and the filter cake is dried. The dried green salt is milled and either processed directly into metal or heat treated and reduced to metal.

## DISSOLUTION

The objective of the dissolution step is to rapidly and economically dissolve uranium metal producing  $U^{+4}$  ions without generating residual sludge. The formation of  $U^{+4}$  ions as a product of dissolution is critical to the process. Dissolved uranium can only be precipitated from the dissolution liquor from  $U^{+4}$ . Selection of a lixiviant is limited to those that result in formation of  $U^{+4}$  ions, and have little tendency to form sludges or residues. If dissolution produces sludge, a serious safety problem will result. In most cases, sludges contain suboxides that are highly pyrophoric and potentially explosive. Any process that generates sludge is inherently unsafe.

The dissolution behavior of uranium in various acids has been described in a number of references (4,5,6,7). Uranium dissolves rapidly in aqueous hydrochloric acid (HCl), but more slowly in non-oxidizing acids such as sulfuric ( $H_2SO_4$ )

and phosphoric ( $H_3PO_4$ ). Hydrofluoric acid (HF) reacts very slowly with massive pieces of uranium. Nitric acid ( $HNO_3$ ) reacts moderately with massive uranium but explosively with finely divided uranium and alloys containing niobium and zirconium. Nitric acid also produces  $U^{+6}$  ions and thus is not suited to the process. Of the potential lixivants, HCl appears to be the best choice based on dissolution rate and the number of different alloys that can be processed. However, for economic reasons,  $H_2SO_4$  could be used if necessary, although dissolution rates are lower than for HCl.

Several experiments were conducted to determine the effect of HCl concentration on the rate of dissolution and amount of residue formed. A range of HCl from 1.2 to 4.3 N was investigated to determine the most suitable concentration. The results indicated that, while high concentrations of HCl increased the rate of dissolution, precipitation of  $UF_4$  from the high concentration solutions was more difficult. Based on these results an optimum HCl concentration of 2.4 N was selected.

Regardless of concentration, straight HCl produces a residue after dissolution that amounts to about 20 percent of the original weight of dissolved metal. This residue is very pyrophoric being composed of uranium suboxides and uranium hydrides. Previous experience with other metals involving fluorides showed that acids such as fluosilicic ( $H_2SiF_6$ ) and fluoboric ( $HBF_4$ ) have unique and useful properties in aiding dissolution in HCl, and preventing or reducing the amount of sludge formation.

To determine the effect of  $H_2SiF_6$  and  $HBF_4$  on preventing residue, additions of  $H_2SiF_6$  ranging from 0.02 to 0.15 M and  $HBF_4$  ranging from 0.08 to 0.39 M were tested in combination with a 2.4 N HCl solution.  $H_2SiF_6$  and  $HBF_4$  contain appreciable amounts of fluorine so that there is a potential for premature precipitation of  $UF_4$  at higher concentrations of these acids. Table I shows the results of dissolution of 0.05 kg pieces of U-3/4Ti in 2.4 N HCl containing either  $HBF_4$  or  $H_2SiF_6$ . The total volume of solution was  $0.0005m^3$ , and temperature was limited to a maximum of 350K. After complete dissolution, the solutions were filtered and the solids dried and weighed. The solids were either green ( $UF_4$ ) or black (pyrophoric uranium compounds). The total time for dissolution was also recorded.

Even though both  $HBF_4$  and  $H_2SiF_6$  reduce the amount of residue compared to straight HCl,  $H_2SiF_6$  is not as desirable for several reasons.  $H_2SiF_6$  has a high activation energy, and the exothermic nature of the reaction raises the temperature of the solution very quickly making for a more difficult process to control. At temperatures below 327K, the dissolution rate with  $HBF_4$  is very slow compared to  $H_2SiF_6$ . However, at temperatures above 327K,  $HBF_4$  produces a more vigorous reaction overall and the total dissolution time is generally less.  $HBF_4$  also appears to have a broader useful range of concentration, between 0.08 to 0.31 N, where very little  $UF_4$  or residue is produced. It is evident from Table I that high concentrations of either  $H_2SiF_6$  or  $HBF_4$  result in premature precipitation and so there is a practical upper limit on the amount of  $H_2SiF_6$  or  $HBF_4$  that can be used.

Additional studies confirmed the effectiveness of  $HBF_4$  in preventing the formation of suboxides and increasing the rate of dissolution. For example, in 0.3 N HCl, the addition of 0.08 N  $HBF_4$  increased the dissolution rate from 0.055%/min to 0.21%/min, nearly a four-fold increase. In all cases a 0.08

to 0.16 N HBF<sub>4</sub> addition reduced the amount of pyrophoric residue to less than 1 percent. Concentrations of HBF<sub>4</sub> greater than 0.24 N, while increasing dissolution rate, are undesirable as the dissolved uranium destroys the BF<sub>4</sub><sup>-</sup> complex and reacts to form UF<sub>4</sub>.

Although HBF<sub>4</sub> is generally more desirable than H<sub>2</sub>SiF<sub>6</sub>, the possibility of contamination with boron may require that H<sub>2</sub>SiF<sub>6</sub> be used. The exception is the dissolution of U-Nb alloys. In this case, H<sub>2</sub>SiF<sub>6</sub> does not work. H<sub>2</sub>SiF<sub>6</sub> decomposes in the dissolution reaction and hydrolyses to form a sludge containing insoluble silicic acid and niobium pentoxide containing 25 to 35 percent silica.

While HCl was selected as the best acid for dissolution, the use of H<sub>2</sub>SO<sub>4</sub> was also investigated for economic reasons. H<sub>2</sub>SO<sub>4</sub> concentrations in the range of 2.58 to 4.33 N with 0.08 to 0.16 N HBF<sub>4</sub> were found to produce acceptable dissolution rates, and very little residue. The dissolution rate was lower with H<sub>2</sub>SO<sub>4</sub> compared to 2.4 N HCl with 0.16 N HBF<sub>4</sub>. For massive pieces of uranium, the rate with H<sub>2</sub>SO<sub>4</sub> was approximately three times lower and, unlike HCl, increasing the concentration of H<sub>2</sub>SO<sub>4</sub> does not increase the dissolution rate. However, for chips and turnings the effect is much lower and for practical purposes either 2.58 to 4.33 N H<sub>2</sub>SO<sub>4</sub> with 0.16 N HBF<sub>4</sub> or 2.5 N HCl with 0.16 N HBF<sub>4</sub> are suitable.

Temperature has a significant effect on the rate of dissolution. With either lixiviant, reaction is slow at room temperature but accelerated by heating above 322K. At a temperature of 333K, the reaction generally proceeds to completion in less than one hour. Typically, dissolutions yield above 95 percent on a uranium metal basis, as calculated from the concentration of uranium in solution.

#### FILTRATION FOR REMOVAL OF INERTS

After dissolution, the pregnant liquor is filtered or centrifuged to remove insoluble inerts. These inerts include undissolved uranium oxides, grinding abrasives and machining residues. For finely divided scrap such as grinding swarf, more than 15 percent of the weight charged into the dissolution tank may be in the form of surface oxides and grinding abrasives. Filtration is necessary to prevent these inerts from being processed into the green salt and becoming a source of contamination. The inerts are treated by neutralization and oxidation prior to disposal. The disposal volume of inerts is relatively small representing only 5 percent of the original volume of uranium scrap that would require burial if not processed through Hydromet. Further work to remove residual uranium from the inerts could eliminate the need for burial of this material.

#### PRECIPITATION OF UF<sub>4</sub>

After the pregnant liquor is filtered, it is pumped into a second tank where HF is added to precipitate UF<sub>4</sub>. The U<sup>+4</sup> in solution can be almost completely precipitated in the presence of the stoichiometric amount of fluoride ion. To determine the amount of HF need for precipitation, the solution is analyzed for U concentration and the total mass of uranium calculated from the volume of available solution. For each kg of uranium, 0.67 kg of 50% HF (w/w) is required to stoichiometrically precipitate UF<sub>4</sub>. Before making the HF addition, the solution is heated to at least 327K. An amount of HF, equal to approximately 110 percent the stoichiometric requirement,

is then added to the solution at a rapid rate while the tank is stirred vigorously.

Precipitation is essentially completed within 5 minutes after which the UF<sub>4</sub> slurry is pumped through a filter press. The filtrate is recovered and recycled back to the dissolution tank. The hydrogen ions from the added HF regenerate the HCl making it suitable for recycle in dissolving more uranium scrap. A bleed stream may be taken on the filtrate to control contaminants if required. Prior to discharging, the UF<sub>4</sub> filter cake is washed to remove most soluble contaminants and impurities. Washing with three volume changes of water effectively removes most of the soluble metallic impurities such as Ti, Mo, Fe, Ni, Cu and Si.

It has been found that the quality of the precipitate is influenced primarily by the rate of HF addition, the bath temperature at the time of addition, and the degree in agitation of the tank. Depending on the precipitation conditions, different hydrates of UF<sub>4</sub> can be produced such as UF<sub>4</sub> • 3/4H<sub>2</sub>O and UF<sub>4</sub> • 2H<sub>2</sub>O. The type of hydrate largely determines the ease of filtering the UF<sub>4</sub> precipitate and potentially its suitability for reduction to metal. The aim is to produce lower hydrates that are denser and more easily filtered. When UF<sub>4</sub> is precipitated at a temperature of approximately 327K, the addition rate is less than 5 minutes, and the bath is well agitated, a dry filter cake is produced that has an apparent bulk density of at least 1400 kg/m<sup>3</sup> with less than 5 percent hydrated water.

Important characteristics of green salt that determine its suitability for metal production are AOI content, percent volatiles, UO<sub>2</sub>F<sub>2</sub> content, impurity content, and apparent bulk density (ABD). AOI or ammonium oxalate insolubles is essentially a measure of uranium oxide content. Of these parameters, the precipitation conditions have the greatest effect on ABD, and precipitation is generally optimized to achieve high ABD. For ease of processing and achieving high yield reductions, a minimum dried ABD of 1200 kg/m<sup>3</sup> is desired.

To test the effects of various precipitation process parameters on precipitate ABD and filterability, 0.002m<sup>3</sup> portions of a stock solution containing 103.7 kg/m<sup>3</sup> uranium were precipitated under conditions of varying addition rate, temperature at the time of the HF addition, and amount of HF addition. These results are summarized in Table II. Additional tests were also conducted on 22.5 kg size batches to confirm the trends observed in the small scale tests. The results of these tests clearly indicate that careful control of HF addition rate, temperature at time of the HF addition, and effectiveness of stirring during the addition are all required.

The slow addition of HF resulted in a fluffy precipitate that had low ABD. When the rate was between 20-30 minutes, the ABD was usually less than 1000 kg/m<sup>3</sup>. At the highest addition rates the ABD approached 2000 kg/m<sup>3</sup> although this dense precipitate was more difficult to filter. At extremely long times (> 30 minutes) the precipitate became gelatinous, could not be stirred or filtered easily, and contained more than 10 percent by weight hydrated water.

Precipitation and filtration were also affected by stirring and temperature at the time of HF addition. Temperatures above 327K consistently produced UF<sub>4</sub> with higher ABD and improved filterability. Poor mixing of the U<sup>+4</sup> solution with HF caused by ineffective stirring resulted in ABD below 1000 kg/m<sup>3</sup>.

TABLE I

Comparison of the Effects of HBF<sub>4</sub> and H<sub>2</sub>SiF<sub>6</sub> on Dissolution in 2.4 N HCl

Reagent	Concentration (M)	Fluoride Conc F (N)	Dissolution Time (min)	Residue (%)	Comments
H <sub>2</sub> SiF <sub>6</sub>	0.15	0.90	100	29.07	UF <sub>4</sub>
"	0.11	0.65	120	16.08	UF <sub>4</sub>
"	0.09	0.52	150	3.84	UF <sub>4</sub>
"	0.06	0.39	240	2.70	black residue
"	0.04	0.26	220	2.72	black residue
HBF <sub>4</sub>	0.39	1.55	60	20.20	UF <sub>4</sub>
"	0.31	1.24	70	16.40	UF <sub>4</sub>
"	0.24	0.93	90	1.52	trivial
"	0.16	0.64	150	0.09	UF <sub>4</sub>
"	0.08	0.32	240	0.42	nothing visible black residue

#### DRYING AND HEAT TREATMENT

After filtering the green salt is dried to remove excess water and volatiles which may contain some tramp acids carried over with the filter cake. The most important considerations in drying are to limit formation of UO<sub>2</sub>F<sub>2</sub> and AOI. Both of these components add oxygen to the reduction retort causing problems during reduction. Oxygen can be freed during the reduction process and react exothermically with the magnesium used for the reductant. This can have the effect of increasing the reaction rate and temperature and under certain conditions can cause a violent reaction (8). Oxygen will also consume Mg producing MgO and making less Mg available for reducing UF<sub>4</sub>. This usually produces low yield derby. Additionally MgO will raise the viscosity of the MgF<sub>2</sub> slag causing poor separation of the slag and uranium metal resulting in poorly formed low yield derby. Water and HF in the retort will react with magnesium causing difficulties, including safety problems, during the reduction of the green salt to metal. Excess moisture must be removed by drying without increasing UO<sub>2</sub>F<sub>2</sub> or AOI to unacceptable levels.

The drying process involves heating to a temperature less than 450K and time sufficient to drive off the excess water and tramp acids such as HCl, HF and HBF<sub>4</sub>. Above 450K, UF<sub>4</sub> reacts with water and oxygen to form uranyl fluoride (UO<sub>2</sub>F<sub>2</sub>). Drying at 491K for eight hours resulted in the generation of more than 2 percent by weight UO<sub>2</sub>F<sub>2</sub>. Drying at temperatures over 450K necessitates using vacuum or a protective atmosphere to prevent UO<sub>2</sub>F<sub>2</sub> formation and an increase in AOI.

After drying it is generally desirable to heat treat the green salt to remove the water of hydration, reduce hygroscopicity and increase bulk density. Drying at less than 450K does not remove the water of hydration. This water can be liberated during the reduction process causing excessive pressure within the reduction vessel. Additionally, the bulk density of the dried UF<sub>4</sub> is low and not ideally suited for use in reduction without compromises to the process. This is due primarily to the lower chemical energy per unit volume avail-

able with a lower density charge. High chemical energy is required to achieve efficient separation of the metal and slag in order to produce good quality, high yield derby. Therefore, the green salt should be heat treated to make it suitable for reduction.

The dried UF<sub>4</sub> must be heat treated at higher temperatures than used for drying to remove the hydrated water. Heat treatment will also decrease the hygroscopicity of the green salt and increase its bulk density (9,10,11). It has been found that at temperatures in excess of 970K, the hydrated water in Hydromet green salt is reduced from over 4 percent to less than 0.05 percent. The tendency for the green salt to reabsorb water is also reduced by heat treatment from over 2 percent to less than 0.05 percent. The apparent bulk density is also increased from approximately 1400 kg/m<sup>3</sup> to over 3000 kg/m<sup>3</sup>. Heat treated Hydromet green salt is comparable to pyromet-allurgical green salt and responds similarly in the reduction process.

#### PRODUCTION OF URANIUM METAL DERBY

The initial reduction trials involving Hydromet green salt consisted of nine reductions of 655 Kg. The charges for these reductions were blended in concentrations of 10 to 30 percent Hydromet with pyromet. The Hydromet green salt was not pre-heat treated. While eight reductions were performed successfully, yields in some instances were as low as 74.6 percent. The ABD of the green salt used for these charges was generally low, ranging from 1440 to 1920 kg/m<sup>3</sup>. The low ABD limited charge weight below normal production standards, and contributed in part to the lower yields.

The UO<sub>2</sub>F<sub>2</sub> + H<sub>2</sub>O was fairly high with most of the material over 3 percent. The high UO<sub>2</sub>F<sub>2</sub> + H<sub>2</sub>O limited the amount of Hydromet that could be used in the charge, and caused some problems in reduction. It was only after reduction process steps were modified to incorporate an in-situ heat treatment to compensate for the water in the non-heat treated Hydromet that acceptable reductions were achieved.

A subsequent study to evaluate the effect of heat treatment was performed using small 6 kg derby reductions (12). A total of 5 derbies with Hydromet concentrations up to 100 percent were produced for this study. The composition of the Hydromet used to produce these derbies is given in Table III. This green salt was made from a U-6Nb alloy, hence the

residual Nb content. As can be seen from Table III, the ABD were all over 2000 kg/m<sup>3</sup> and UO<sub>2</sub>F<sub>2</sub> concentrations were low.

The heat treated Hydromet was blended 33, 67 and 100 percent with pyromet to make up charges for these reductions. The derby results are given in Table IV. All the derbies were well formed except No. 12 where a small piece of metal was not attached to the bulk of the derby. The yields for all derbies

TABLE II

Test Series on the Effect of Precipitation Conditions on ABD

Addition Rate (m <sup>3</sup> /min)	Temp (K)	HF Addition (m <sup>3</sup> - 50% w/w)	Settling Rate (% hr)	ABD kg/m <sup>3</sup>	Comment
5 x 10 <sup>-6</sup>	298	.000135	-	1500	
5 x 10 <sup>-6</sup>	298	.000135	10	1430	
Dump	298	.000150	12.9	1840	
Dump	298	.000150	10.5	1880	
Dump	298	.000135	-	1777	1.22 N HCl
Dump	298	.000135	-	1860	1.83 N HCl
Dump	298	.000135	-	1430	3.05 N HCl
-	298	.000135	22	1.75	(A)
-	298	.000135	13.3	1.79	(B)
-	298	.000135	13.4	1.80	(C)
5 x 10 <sup>-6</sup>	298-306	.000135	10		
10 x 10 <sup>-6</sup>	298-305	.000135	21	1.42	
15 x 10 <sup>-6</sup>	-	.000135	13		
27 x 10 <sup>-6</sup>	298-305	.000135	-		
27 x 10 <sup>-6</sup>	298-305	.000135	-	(D)	
9 x 10 <sup>-6</sup>	333	.000135	61	1.73	
9 x 10 <sup>-6</sup>	333	.000120	44.8	1.77	
10 x 10 <sup>-6</sup>	333	.000150	84.2	1.80	
Dump	333	.000135	20	1.76	
Dump	335	.000135	-	1.74	
Dump	333	.000270	35	1.88	25% HF (w/w)
Dump	333	.000270	23.3	1.89	25% HF (w/w)
-	333	.000135	24	1.78	(C)
-	333	.000150	25.7	1.93	(C)
Dump	352	.000135	-	1.77	
Dump	354	.000135	-	1.67	

(A) U solution added to HF @ .0001 m<sup>3</sup>/min.

(B) U solution added to HF @ .0002 m<sup>3</sup>/min.

(C) U solution dumped into HF.

(D) Solution solidified.

TABLE III

Analysis of Heat Treated Hydromet Green Salt Used for 6 Kg Derbies

Sample	ABD kg/m <sup>3</sup>	UF <sub>4</sub> %	UO <sub>2</sub> F <sub>2</sub> %	AOI %	Diff %	Nb ppm	Cu ppm	Fe ppm	Ni ppm	Co ppm
1B-ICHT	2250	97.33	1.09	1.48	0.10	25	9	174	17	5
3BHT	2340	95.79	0.72	3.19	0.29	25	4	133	10	6
3CHT	2340	94.96	0.35	4.50	0.19	25	5	74	9	5
1GHT	2510	96.28	0.31	3.14	0.27	515	9	101	11	3
1GHT-2	2430	97.30	0.66	2.28	-0.24	25	13	101	11	6

TABLE IV

## Heat Treated Hydromet Derby Results

Reduction No.	Blend (%)	Fire Time (HR)	Nb (ppm)	Cu (ppm)	Fe (ppm)	Ni (ppm)	C (ppm)	Yield %
11	33	2.3	198	9	92	11	33	92.6
12	33	1.9	217	6	92	10	30	93.1
13	67	2.35	425	5	165	9	37	92.8
14	100	2.35	395	3	230	6	79	96.6
15	100	2.5	759	15	164	5	220	90.6

were high and within the typical range for pyromet derbies. The high carbon content in derbies No. 14 and 15 was probably the result of using uncleaned saw chips in the original dissolution step. The chips were stored under dilute machine coolant and no attempt was made to remove residual oils prior to dissolution. Iron contamination was picked up during processing of the green salt, and most likely due to contact with various steel components in the furnaces and pulverizing equipment. The average iron level after drying was 38 ppm, so most contamination occurred during subsequent processing. The sources of Fe contamination can be eliminated by drying under suitable covers, heat treating in a nickel lined muffle and grinding in a ceramic ball mill. Carbon contamination can be eliminated by cleaning and degreasing chips prior to dissolution.

## SUMMARY

The Hydromet process for producing UF<sub>4</sub> developed by NMI provides a means for recycling uranium and uranium alloy scrap. Using the Hydromet process, it is possible to recycle low grade scrap that would otherwise require disposal by burial. The UF<sub>4</sub> from Hydromet produces high purity, high quality metal. Unlike direct remelting, purification is inherent in the processes of dissolution and precipitation that are the basis for Hydromet.

The Hydromet process is stable, controllable, and with appropriate heat treatment, the product is entirely comparable to standard pyrometallurgical green salt. The essential conditions and requirements for each step of the process have been defined. Hydromet green salt can be used on an equivalent basis to pyromet green salt to produce derby without any modifications or adjustments to the reduction process.

Future work is planned on adapting this technology to the recycle of other types of uranium process scrap. This includes leaching uranium from the magnesium fluoride slag produced during derby reduction, sludges from derby pickling solutions and other types of depleted uranium alloys. Hydromet is also suited to the recycle of enriched uranium as the chemistry of depleted and enriched are identical. However, criticality factors obviously need to be considered when processing enriched material.

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