

NICKEL INGOTS



The East End Smelter created 9,700 tons of nickel ingots, which are volumetrically contaminated with radionuclides. At one time the estimated value of the nickel exceeded \$130 million, but can vary widely based on fluctuating market prices for scrap metals.

Each of the ~24-inch by 18-inch ingots weighs about a ton. The ingots are 99.9 percent pure nickel. The ingots were made from metal reclaimed during Cascade Upgrade Program in Paducah and Portsmouth in the 1970's and 1980's.

Separation of Nickel from Technetium-Contaminated Scrap

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Report

Introduction

The recovery of nickel (Ni) from Department of Energy (DOE) gaseous diffusion plant barriers contaminated with radionuclides and specifically, the separation of Ni from technetium-99 (^{99}Tc), has proven to be difficult. Manufacturing Sciences Corporation (MSC) could not remove ^{99}Tc from volumetrically contaminated Ni utilizing electro-refining approaches to levels that would allow the free release of Ni for commercial and industrial uses. The various methods applied by Manufacturing Sciences Corporation (MSC) are reported in the attached Appendices. The electro-refining methods employed by MSC resulted in Ni containing residual ^{99}Tc . Residual ^{99}Tc in Ni purified by MSC's electro-refining methods resulted in a moratorium being issued by the Secretary of DOE and congressional opposition to the release of Ni from the K-25 plant at Oak Ridge. The present proposal employs an approach that does not rely on electro-refining for the separation of ^{99}Tc from volumetrically contaminated gaseous diffusion plant Ni barriers.

A major obstacle to the free release of surface or volumetrically radioactive contaminated scrap metal originating in radiation impacted areas within the DOE Complex is the scrap metal industry's position of "zero tolerance." The scrap metal industry produces metals that are used in industrial and consumer products. The scrap metal industry position is that the release of scrap metal from radiation impacted areas at DOE facilities into the industry's recycled scrap metal flow path would have both short and long-term negative impacts. It fears the rejection of its products by customers and the contamination of its processing systems. The scrap metal industry supported the decision by the Secretary of DOE to impose a moratorium on the release of volumetrically contaminated metal. This moratorium halted the release of MSC electro-refined Ni, volumetrically contaminated with residual ^{99}Tc , from the K-25 Oak Ridge, TN facility.

What happens
to Ni ...
is used
battery?

The Nuclear Regulatory Commission (NRC) continues to evaluate national standards for the potential release of materials from radiation impacted facilities. The release of DOE scrap metal from radiation-impacted areas, including radiation impact areas at DOE facilities, would be required to meet standards established by NRC and Agreement States. DOE's moratorium on the release of volumetrically contaminated metals remains in place until the NRC and Agreement States establish national standards for the release of radiation-impacted materials. Furthermore, DOE suspended the unrestricted release for recycling of scrap metal from radiological areas within DOE facilities and the suspension will remain in effect until DOE develops release criteria and established release criteria through DOE Order 5400.5.

DOE indicated in its April 2002 draft Guide "DOE G 441.1-XX, CONTROL AND RELEASE OF PROPERTY WITH RESIDUAL RADIOACTIVE MATERIAL for use with DOE 5400.5, *Radiation Protection of the Public and the Environment*", that the DOE's principle requirements for the release of scrap metal from radiation impacted areas are intended to meet the following goals:

- Property is evaluated, radiologically characterized, and, where appropriate, decontaminated before release.
- The level of residual radioactive material in property to be released is as near background levels as is reasonably practicable, as determined through DOE ALARA process requirements, and meets DOE authorized limits.
- All property releases are appropriately certified, verified, documented, and reported; public involvement and notification needs are addressed; and processes are in place to appropriately maintain records.

Thus prior to release of both surface and volumetrically radiation contaminated scrap metal from DOE and other licensed radiation impact facilities, a number of important concerns must be overcome to ensure the released material will not have a negative impact on both public health and the public perception of the scrap metal industry's use of the material in commercial and industrial products.

Problem Statement

The decontamination and radiation decommissioning of the gaseous diffusion process at the Paducah Gaseous Diffusion Plant, in Paducah Kentucky, has generated and will generate vast quantities of nickel and other metals volumetrically contaminated with radioactive materials. The estimated amount of contaminated nickel could reach 44,794 tons (1). The most frequently identified contaminant in the nickel is technetium-99 (99Tc). However, traces of neptunium (Np), plutonium (Pu), protactinium (Pa), thorium (Th), and uranium (U) have also been identified in the nickel.

There is interest in recovering the nickel and recycling it to the industrial sector. However, as indicated above, there are many regulatory issues associated with any use of such material outside of the nuclear industry. The main problem in decontaminating this nickel is an ultrahigh efficiency separation method necessary to separate technetium and nickel. The other radioactive materials can be separated via electrolysis processes. However, the best available electrolysis process still leaves ~ 1 Becquerel of technetium activity per gram for starting materials of 320 Becquerels: this separation does not meet the required release criteria for radioactive materials. This project will explore a new alternative separation method based on the large differences between the vapor pressures of nickel and technetium.

Prior Art

Prior and present processes

The prior and present processes for removal of Tc from volumetrically contaminated Ni have not been effective for removal of all the residual Tc. The presence of residual Tc in processed Ni metal from K-25 gaseous diffusion plant Ni barriers in Oak Ridge prevented the release of the reprocessed Ni. Such processes include ion exchange, solvent extraction, melt refining, inductoslag refining, and electrolysis. There have been different processes for the recovery of Ni.

Ni is selectively stripped by an organic oxime from an acidic solution of Ni & Cu and then Ni electrowinning (2). Another process is the removal of Ni by liquid-liquid extraction (3,4). Muller, et al. extracted Ni from aqueous solutions that contained large amounts of alkali metal ions by contacting the solution with an organic solution of di-2-ethylhexyl phosphoric acid and naphthenic acid (3). Fujimoto et al. used organic mixtures of 2-ethylhexyl phosphoric acid mono-2-ethylhexyl ester and/or 3,5,5-timethylhexyl phosphoric acid mono-3,5,5-timethylhexyl ester and/or isodecyl phosphoric acid monoisodecyl ester to separate cobalt (Co) from Ni (4). It is known that metallic Ni, contaminated with fission products, could be decontaminated to remove any actinides present by direct electrorefining based on the differences in reduction potential along the electromotive force (emf) series. Actinide removal is favored by two phenomena during electrorefining. Actinides have a significantly higher reduction potential relative to nickel and they are normally won from molten salt electrolyte rather than from aqueous electrolyte (see U.S. Pat. Nos. 3,928,153 and 3,891,741) (5).

Electrorefining and electrowinning of Ni decontaminated streams have been utilized but these processes encountered problems with the co-deposition of the Tc and Ni on the cathodic cell. This problem was solved by converting Tc(VII) to Tc(IV) which prevents it from co-depositing with Ni (6-10). Other problems arise with the electrowinning and electrorefining processes because of the generation of large volumes of radioactively contaminated acid wastes especially when large amounts of Ni have to be recovered.

Need for an efficient process

There is a need for an economical and efficient method to decontaminate radioactive scrap metals from nuclear facilities. Prior studies suggest that direct recycle of decontaminated metals to consumer products is not likely in the US or Europe. The presence of only residual parts per million concentrations of fission daughter products such as technetium in nickel and other like recycled products will make the products unacceptable to the public. The release of material containing residual radioactivity to unregulated non-nuclear markets has been prevented both politically and by regulation. It is likely that residually contaminated products would need to be recycled back to regulated nuclear markets, which would pay a lower price. Many of the references in the review for the Kentucky Consortium by Silverstein address the issues associated with public concerns about such recycled metals (see Table 1 (Silverstein 2007)). Since the 1990's, there have been many studies of nickel purification technologies.

Easier to
with
Suth...

Process constraints for remediation processes are: 1) the efficiency of each processing step, 2) the volume of waste streams, and 3) the contamination in these byproduct streams. The radioactive components will concentrate in some byproduct stream, which probably would need to be stored or placed in a hazardous landfill. The level of remaining contamination in the waste stream(s) is an important parameter for process design, and this issue seems not to have been addressed in prior studies on recycling processes.

Table 1. List of technologies and processes reviewed by commercial and government contractors.
Data from Silverstein, 2007. (Silverstein 2007)

Contractor	Year	Comments	Reference
Westinghouse	1993	Electrorefining processing of radioactive scrap metals. ⁹⁹ Tc not included	(Kessinger 1993)
Argonne National Lab	1993	Consumer reuse issues with contaminated metals. ⁹⁹ Tc in metal slag.	(Murphie and Lilly 1993)
Oak Ridge National Lab	1993	Review of smelt purification, electrorefining, leach/electrowinning, Mond process	(Fellows 1993)
Martin Marietta Energy Systems	1993	Electrorefining to recover Ni from porous barriers	(Bundy and Kennerly 1993)
Department of Energy	1993	Program Summary. MSC/CVMR was to use decontaminated steel in nuclear waste storage	(Motl and Burns 1994)
Department of Energy	1994	Electrolysis might be best process for nickel recovery	(Compere and Griffith 1994)
Argonne National Lab	1994	Radioactive scrap recycling in the context of human health risk, environmental impact, sociopolitical concerns.	(Nieves and Chen 1994)
Manufacturing Sciences Corporation/ Colorado School of Mines	1995	Viability of radioactive scrap metal	(Muth and Shasteen 1995)
Manufacturing Sciences Corporation/ Science Applications International Corp.	1997	MSC, SAIC were to have ownership of metals they cleaned as part of ORNL building remediation. Clean Ni to NiMH battery use	(Neal 1997)
Scientific Ecology Group, Inc.	1997	ORNL environmental assessment found that sale of Paducah nickel ingots would not affect the human health environment. Recycled nickel would be shipped to Spain, which allowed metal with up to 74 Bq/g. SAIC.	(SAIC 1955; Hall 1996)
Argonne National Lab	1997	RSM (iron, steel, SS, copper) disposition alternatives	(Nieves, Chen et al. 1998)
ELR Consultants	2000	ELR bid on contract for ways to profit from Ni reuse	(Walker 2000)
DOE	2002	Call for proposals to purify Ni	(Kuck 2002)
CVMR-USA	2004	Metal vapor processing, 8 employees, 2000 ton/yr nickel	(Walker 2004)
DOE	2007	Expressions of Interest. Disposition of PGDP and ORNL nickel	(Energy 2007)

There remains a need for an economical and efficient method to refine and decontaminate metals from nuclear facilities. Process constraints could include:

- Environmentally friendly, green chemistry if reactions or solvents are needed,
- Simple operation and equipment,
- Suitable for radioactive monitoring of purified and waste products,
- Suitable for small high tech business to operate over an extended period,
- Economical recovery, and
- Significant improvement in nickel purity.

The advantages of the physical vapor separation of Ni-Tc are as follows:

- highly pure nickel can be produced,
- Tc is concentrated in the liquid bottoms by-product, which can be solidified to a high density,
- the process cost is low,
- the process is fundamentally simple, and can be carefully monitored for performance, and
- fugitive gas streams can be scrubbed and/or filtered to remove metals dust particles.

The simplest physical vapor separation method is distillation due to the wealth of information, equipment and know-how surrounding this technology. Both high-boiling and low-boiling components can be separated from the base mixture, although it is less costly to remove low boiling components. In the case of nickel contaminated with ⁹⁹Tc, the nickel would be taken off as a vapor, leaving a liquid phase enrich in technetium. Successful use of distillation for this application could lead to other needed separations in the metal scrap industry.

Other markets for metal distillation technology

The scrap metal industry is a secondary metal industry. The dismantling and the decommissioning of nuclear facilities along with scrap metal inventories at Department of Energy (DOE) sites would supply the scrap metal industry with millions of tons of metals. This requires the decontamination of the scrap metals before release to public use. The principal administrative authorities responsible for controlling the release of scrap metal from nuclear facilities are the DOE, the Nuclear Commission Regulatory (NCR), Department of Defense (DOD), and Agreement States (1). These authorities have a jurisdictional authority over 30,000 structures, of which 8,000 are contaminated (1). The DOE has an estimate of 149,665 tons of contaminated metals in its existing inventory (1). This inventory consists of carbon steel (119,232 t), Ni (10,699 t), stainless steel (7,462 t), aluminum (Al) (2,353 t), copper (Cu) /brass (1,975 t), and 7,943 tons of other metals (1).

The decommissioning of DOE gaseous diffusion plant facilities commenced with the K-25 plant (1998-2006). The proposed schedule for decommissioning of the following DOE facilities are the Portsmouth plant (2007-2015), then the Paducah plant (2015-2023), and finally the rest of the plants (2023-2058) (1). The existing and future contaminated scrap metal at DOE facilities will total to 1,068,022 tons (1). This inventory consists of carbon steel (903,897 t), Cu/brass (53,990

t), Ni (44,818 t), stainless steel (26,960 t), aluminum (36,070 t), lead (Pb) (291 t), monel (83 t), and 1,913 tons of other metals (1).

Our Solution

This project will develop and demonstrate a technically effective and cost-efficient process using distillation to recover pure nickel with no detectable traces of technetium. The slag left behind will be composed of technetium with small levels of nickel. The physical vapor deposition consists of two steps: 1) nickel is preferentially evaporated from solid or liquid solutions of Ni/Tc, 2) the nickel-rich vapor is condensed either as a powder or deposited on cold surfaces to produce Tc-“free” nickel plates, and 3) the ^{99}Tc -rich waste product is solidified and sent to a hazardous landfill.

Thermodynamic data

The project originally was designed to take thermodynamic data on the phase equilibria of ^{99}Tc -Ni mixtures as an important input to an engineering design effort. The phase equilibria can be studied using a Knudsen cell coupled to a GC/MS designed to separate metals in the vapor phase in equilibrium with a nickel mixture. This data would permit the estimate of the phase equilibrium activity coefficients as a function of temperature. Similar systems were constructed at Lawrence Livermore Lab (1969), Los Alamos (1983), and NASA Glenn. Bert Lynn, University of Kentucky chemist, is a design expert on GC/MS instruments and is collaborating on design, construction and commissioning of the new GC/MS. A system designed and assembled by a commercial vendor was estimated to cost well over \$300,000, so the team decided to design and build its own system.

The major components have been designed but are not yet installed as the project funds have been expended. We need to build a vacuum system (~\$20k), assemble and commission the instruments, and are slowly assembling funds outside of the project to do this.

New data obtained with the proposed GC/MS will define the vapor/liquid phase equilibria for Ni and very dilute ^{99}Tc . Data to be obtained includes vapor pressures (sufficient for process design), heats of vaporization, heats of sublimation, and activity coefficients, for the Ni- ^{99}Tc pair at different temperatures. The data will be incorporated into models of the phase diagrams that can be used to refine the design. The method could be applied to the purification of other metal scrap mixtures, revolutionizing the use of distillation for purification of other systems.

Process design

Although it is preferable to base a process design on vapor liquid equilibrium data, a preliminary process design based on data estimates would have significant value in guiding experiments and process concepts. Due to the delays in constructing the instrument, a preliminary design has been completed, and is the main topic of this final report.

Design constraints. Criteria release of the US and other countries

^{99}Tc is a β -emitting radionuclide with maximum beta energy of 297 keV and a half-life of 2.1×10^5 years. At Paducah, nickel processing equipment, contaminated mostly with ^{99}Tc , was smelted and cast into ingots in the late 1970's and early 1980's. This scrap is said to be volumetrically contaminated as the radioactive material became uniformly distributed through the melt. Volumetrically contaminated Ni may have a Tc activity of up to about 5000 Bq/g or more, which is at least an order of magnitude above the maximum international release criteria of 74 Bq/g metal total activities. Certain countries have specified lower criteria of 1.0 Bq/g or less total activity. As indicated in the introduction, the U.S. regulatory agencies and federal government would not release scrap metals with any radioactivity detection to the public. Recent test results showing the actual contamination of these ingots is reported in a later section.

Technical review of current Tc-Ni separation processes

Conventional metal purification processes include zone refining, electrochemical refining/electrowinning, chemical vapor deposition and vaporization/distillation.

Zone refining (zone melting) separates metal contaminants by moving a molten zone through a long ingot. The melting and recrystallization process leaves purer crystals in its wake, pushing contaminants to the front of the molten zone. The contaminated material from this end is cut off, and the ingot can then be used. The process can be slow, and is best applied to systems in which there is a significant concentration difference between the contaminant concentrations between the solid and liquid phases at equilibrium.

Other processes include ion exchange, solvent extraction, inductoslag refining, and electrolysis. There have been different processes for the recovery of Ni. Ni is selectively stripped by an organic oxime from an acidic solution of Ni & Cu and then Ni electrowinning (Skarbo 1974). Another process is the removal of Ni by liquid-liquid extraction (Muller, Witzke et al. 1979; Fujimoto, Miura et al. 1980). Muller, et al. (Muller, Witzke et al. 1979) extracted Ni from aqueous solutions that contained large amounts of alkali metal ions by contacting the solution with an organic solution of di-2-ethylhexyl phosphoric acid and naphthenic acid. Fujimoto et al. (Fujimoto, Miura et al. 1980) used organic mixtures of 2-ethylhexyl phosphoric acid mono-2-ethylhexyl ester and/or 3,5,5-trimethylhexyl phosphoric acid mono-3,5,5-trimethylhexyl ester and/or isodecyl phosphoric acid monoisodecyl ester to separate cobalt (Co) from Ni. It is known that metallic Ni, contaminated with fission products, could be decontaminated to remove any actinides present by direct electrorefining based on the differences in reduction potential along the electromotive force (emf) series. Actinide removal is favored by two phenomena during electrorefining. Actinides have a significantly higher reduction potential relative to nickel and they are normally won from molten salt electrolyte rather than from aqueous electrolyte (Carlin, Darlington et al. 1975; Gendron, Tilak et al. 1975).

Electrorefining and electrowinning of Ni decontaminated streams have been utilized but these processes encountered problems with the co-deposition of the Tc and Ni on the cathodic cell. This problem was solved by converting Tc(VII) to Tc(IV) which prevents it from co-depositing with Ni (Hradil 1995) (Snyder, Ayers et al. 1993; Snyder, Gass et al. 1993; Snyder, Gass et al. 1993; Snyder and Goad 1995). Other problems arise with the electrowinning and electrorefining

processes because of the generation of large volumes of radioactively contaminated acid wastes, especially when large amounts of Ni have to be recovered.

Chemical vapor deposition includes the Mond process, in which carbon monoxide is used to make nickel carbonyl vapor, move the vapor to a different vessel, and reform the nickel. This process has not been considered in this report for the following reasons: metal carbonyls are very hazardous and require great care, and technetium will also form metal carbonyls via similar chemical reactions. Thus, it is unclear what separation would be accomplished, as it would depend on the differences in vapor pressure under the reaction conditions.

Metal distillation is not usually used for purification, but has been studied for metal with high vapor pressure, such as magnesium and zinc. Temperature and pressure affect the vaporization process, but there are design methods for sizing distillation equipment that should be directly applicable to these problems. Since distillation of binary liquid mixtures has been extensively studied, there are a variety of operation methods and equipment configurations that can be used to tailor such processes to specific problems and operation constraints.

A metal distillation strategy for removing ^{99}Tc from nickel scrap has the following potential benefits:

- Distillation could be done continuously if rapid purification of all the scrap metal was needed,
- Distillation could be done batchwise with verification of metal purity to ensure compliance with scrap metal standards,
- It is an environmentally-friendly approach, requiring electricity, cooling water, inert gas (such as nitrogen) and, possibly, vacuum,
- The product could be nickel powder, which could be further transformed to foils and foams with added values, and
- Using a small batch configuration, it is possible to design a process to work away the scrap over a longer time period, and could be performed by a spin-off company.

Silverstein has reviewed both nickel prices long-term trends and nickel market sizes. The following figures were excerpted from data of the London Metal Exchange.

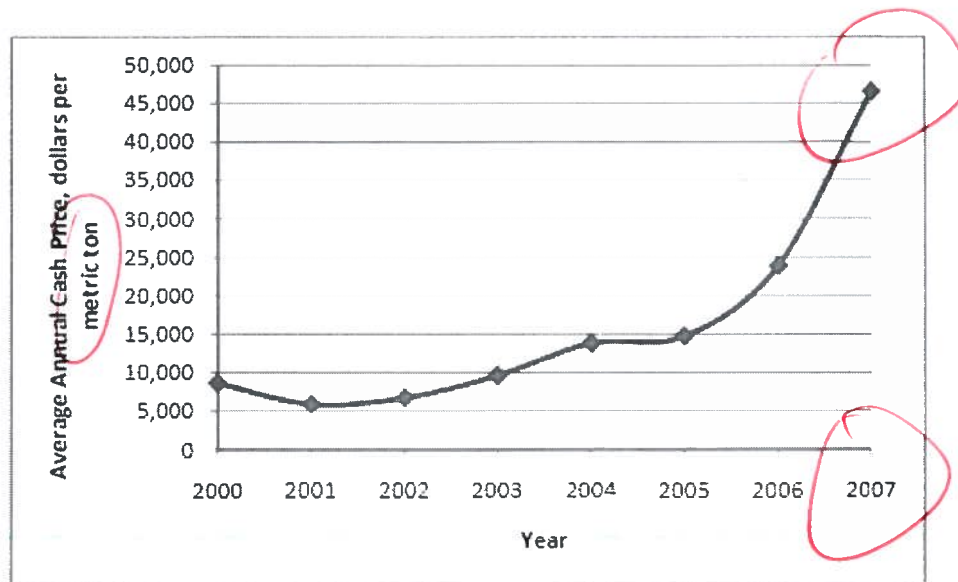


Figure 1. Nickel prices. London Metal Exchange. Silverstein (Silverstein 2007).

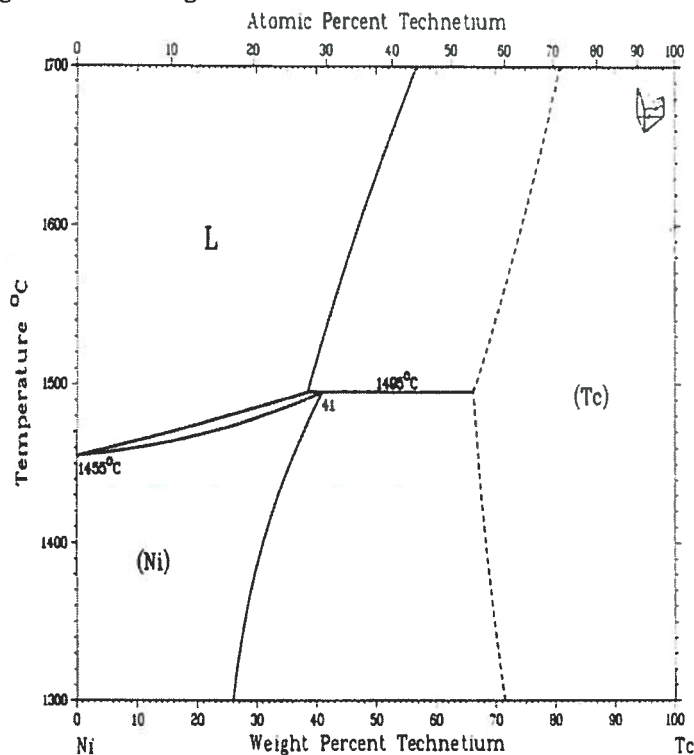
Figure 2. USGS data on nickel volumes and costs (Kuck 2002).

Salient Statistics—United States:	2002	2003	2004	2005	2006*
Production, refinery byproduct	W	W	W	W	W
Shipments of purchased scrap ^a	114,000	119,000	113,000	117,000	107,000
Imports: Primary	121,000	125,000	136,000	143,000	161,000
Secondary	9,110	11,500	18,800	15,500	20,900
Exports: Primary	6,520	6,330	8,000	7,630	15,200
Secondary	39,400	47,300	48,300	55,600	48,600
Consumption: Reported, primary	88,200	87,300	98,900	96,800	102,000
Reported, secondary	63,900	83,500	83,300	77,300	79,300
Apparent, primary	121,000	117,000	128,000	137,000	147,000
Total ^d	205,000	200,000	212,000	214,000	226,000
Price, average annual, London Metal Exchange:					
Cash, dollars per metric ton	6,772	9,629	13,823	14,738	23,871
Cash, dollars per pound	3,072	4,368	6,270	6,685	10,828
Stocks: Consumer, yearend	11,600	11,100	11,000	11,500	10,200
Producer, yearend ^d	6,150	8,040	6,580	4,380	4,100
Net import reliance ^e as a percentage of apparent consumption	52	50	55	56	60

Metal distillation

Metal distillation processes have been developed for several metals with high vapor pressures such as magnesium and zinc. Appendix 4 reviews some of the key patents relevant to this technology. Distillation separates materials based on their vapor pressures over the temperature range of the process. The binary phase diagram of nickel and technetium is shown in the following figure.

Figure 3. Phase diagram of nickel and technetium



P. Nash, 1985

The composition of the nickel scrap at Paducah is located far to the left on the x axis (ppm Tc). Nickel melts at 1455 C, and there is a modest difference between the composition of the solid and liquid phases with respect to technetium. A constant temperature line going through the two phase liquid solid region between 1455 C and 1495 shows that the melt, L, would be deficient in technetium relative to the solid (Ni). Therefore, zone refining might not develop significant changes in purity.

Metal-metal phase diagrams, similar to Figure 1, show solid and liquid phase compositions, but do not contain information on the metal in the vapor phase in equilibrium with either the solid or the liquids. This data is what is needed to design distillation processes. ?

The solid and liquid phases are usually considered to be metal solutions. Metal solutions are often modeled by regular solution theory, which can be used in this case to estimate the possible non-ideality of the liquid phase. It is likely that the vapor phases are ideal thermodynamic fluids at conditions up to the boiling point of the liquid. Up to this point, the vapor pressure of key components would be near atmospheric. That is, associations, complexes, and reactions are not expected in the vapor phase, particularly in the absence of oxygen. Oxygen could combine with either metal to form oxides that might precipitate as slags. This reaction can be prevented by using inert gas over the molten metal, by operating the system under partial vacuum, or by using both techniques.

The thermal transitions of nickel and technetium are shown in the following table.

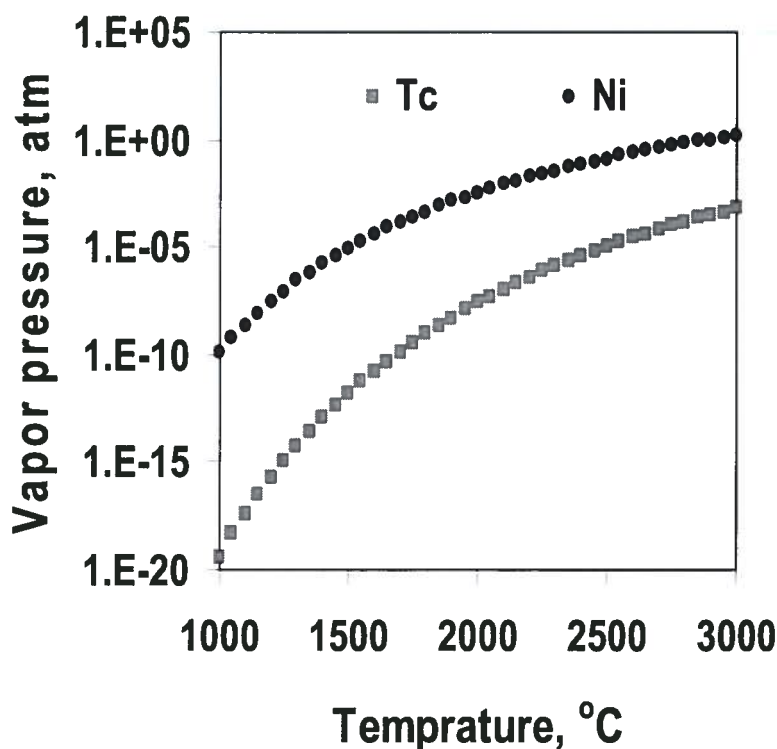
Table 2. Thermal transitions of nickel and technetium.

Component	T _m , K	T _b , K
Nickel	1728	2415
Technetium	2430	5000

Figure 4. Nickel and technetium vapor pressures

The vapor pressures of pure nickel and technetium are shown in the next figure. The data are from the DIPPR data base. The vapor pressure curves are thought to be accurate between the melting and boiling points of the pure metals, and have been extrapolated for comparison purposes. Notice that there are orders of magnitude differences in the pure component vapor pressures that would lead to high purity separations. For example, at the boiling point of nickel (2415 K), its vapor pressure is one atmosphere while the vapor pressure of technetium is estimated to be 3×10^{-7} atm. Based on the extrapolation, lower

operating temperatures could lead to larger vapor pressure differences. However, the distillation system would have to operate at less than atmospheric pressure, which would add to the process equipment and operating costs.



Design basis

1. ⁹⁹Tc level in scrap nickel ingots

There are a number of conventional metal purification methods that can be applied to the separation of technetium from nickel scrap. Table 2 shows the amount of radioactivity found in various samples of the scrap metal at the Paducah site. Processing equipment was smelted and formed into ingots, which have different levels of contamination. The most significant contamination is that of Technetium 99, which ranges from 9.77 picoCuries ⁹⁹Tc/gram metal to 23,500 pCi/gram.

Table 3. Samples of 72 Paducah nickel ingots. 1999-2000. (Energy 2007)

Nuclide	sample results, pCi/g		average	standard deviation
	minimum	maximum		
alpha	4.6	4.6	4.6	N/A
beta	3970	11400	6980	2650
²³⁷ Np	0.163	0.47	0.268	0.126
²³⁹ Pu	6.06	7.53	6.73	0.743
⁹⁹ Tc	8.77	23500	13800	5990
²³⁰ Th	1.13	1.13	1.13	N/A
²³² Th	0.0000264	0.0118	0.0005	0.00196
²³⁵ U	0.00021	0.0184	0.0056	0.0077
²³⁸ U	0.00214	0.912	0.12	0.197

The radiation levels are converted to weight fractions for the purpose of process design calculations. A Curie (Ci) is a unit of radioactivity with the equivalence:

$$1 \text{ Ci} = 3.7 \times 10^{10} \text{ decays per second.}$$

This unit has now been replaced by the SI unit, Becquerel (Bq), defined as one decay per second. The equivalences are:

$$1 \text{ Ci} = 3.7 \times 10^{10} \text{ Bq;}$$

$$1 \text{ Bq} = 2.70 \times 10^{-11} \text{ Ci} = 27.0 \text{ pCi (where pico} = 10^{-12}\text{)}$$

The specific activity of ⁹⁹Tc is $1.69 \times 10^{-2} \text{ Ci/g } ^{99}\text{Tc}$. The specific activity times the equivalence factor gives the number of Becquerels of radiation per gram of ⁹⁹Tc: $6.24 \times \text{Bq/g } ^{99}\text{Tc}$. In Europe, radioactive levels are reported in Bq/g. For example, Germany has a requirement that scrap metal contain less than 0.1 Bq/g. Several of these standards are listed in Table y2, along with their conversion to weight fraction, parts per million (weight) and mole fraction.

Table 4. Conversion of regulatory standards to concentration measures.

	regulatory agency					
	IAEA	EU	NRC (not adopted)	DE scrap metal	DE remelted	BE b emitter
clearance standard		300	100	50	0.1	1
g Tc ⁹⁹ /g solid	4.81E-07	1.60E-07	8.02E-08	1.60E-10	1.60E-09	1.60E-09
ppm Tc ⁹⁹	4.81E-01	1.60E-01	8.02E-02	1.60E-04	1.60E-03	1.60E-03
mol Tc ⁹⁹ /mol solid	2.88E-07	9.60E-08	4.80E-08	9.60E-11	9.60E-10	9.60E-10

The ingot sample data in Table 2 has been converted to concentration measures in Table 4.

Table 5. Concentration measures of the ingot sample tests.

	sample results, pCi/g		
	min	max	average
⁹⁹ Tc, pCi/g		8.77	23500
⁹⁹ Tc, Bq/g	0.324814815	870.3704	511.1111
g Tc ⁹⁹ /g solid	5.21E-10	1.40E-06	8.19E-07
ppm Tc ⁹⁹	5.21E-04	1.40E+00	8.19E-01
mol Tc ⁹⁹ /mol solid	3.12E-10	8.36E-07	4.91E-07

These data suggest that the ⁹⁹Tc contamination may only be as high as ~2 ppm.

We understand that some material may be as high as 100 ppm, and have used 10 ppm ⁹⁹Tc as our design point for this study.

2. Regular solution theory. Vapor-liquid equilibria of metals.

The design of distillation equipment is relatively straightforward when there are models for the equilibrium concentrations between vapor and liquid phases. Regular solution theory has been found to model most metal liquid mixtures well. At a specific temperature for a given liquid phase composition, the vapor phase composition in equilibrium with the liquid phase is:

$$y_i \cdot P = p_i = \gamma_i \cdot x_i \cdot P_i^{\text{sat}} \quad (1)$$

Where y_i and x_i are the vapor and liquid phase mole fraction of component I, P is the total pressure, P_i^{sat} is the saturated vapor pressure of component I, and γ_i is the activity coefficient that accounts for non-ideality in the liquid phase. In an ideal liquid mixture, the activity coefficient value is one, and Eq. 1 collapses to Raoult's law. The saturated vapor pressure of each metal is available from DIPPR as a function of temperature. The starting liquid phase compositions would be known by test. The activity coefficient can be either greater than or less than one. When the activity coefficient is greater than one, the vapor phase is enhanced in that component relative to the case for an ideal liquid phase. In general, the activity coefficients reported for metal liquid mixtures are on the order of 5 or less. In the absence of measured phase equilibria, this is a reasonable estimate as the calculated vapor phase concentration would be a maximum.

Since the nickel is contaminated with ^{99}Tc at the ppm level, it is relatively pure and its activity coefficient is expected to be 1. On the other hand, the activity coefficient for technetium should approach its limit at infinite dilution, $\gamma_{\text{Tc}}^\infty$. Therefore, setting the activity coefficient of technetium completely establishes the phase equilibria for this mixture. [Activity coefficients are usually only modest functions of temperature.]

We have used a technetium activity coefficient of 5, which should give an upper bound to its vapor phase mole fraction.

The total system pressure is:

$$P = p_{\text{Ni}} + p_{\text{Tc}} \quad (2)$$

With the vapor pressure correlations, the assumptions about the activity coefficients, we can model the vapor phase concentrations over a range of temperatures and total pressures.

Process selection

Distillation technology. Batch vs. continuous operation.

Economic design of distillation separations for liquids near room temperature usually results in continuous processes with online monitors to ensure quality control. Materials of construction would be metal or glass with conventional polymer seals for moving parts. The use of continuous equipment drives the processing cost down.

Materials of construction become an issue as the process temperature is raised. If the distillation were to be done above the boiling point of nickel, ceramics would be needed for the process equipment. While it is possible to make such equipment, it would be special order, and would add significantly to the capital costs, and the operating and maintenance costs.

The purification of the nickel to remove a radioactive component complicates the instrumentation needed for continuous process. It is difficult to separate product with continuous processing, so there would have to be a reliable, online monitoring system to verify that no off-spec material was made. And, there probably would have to be additional equipment to separate such material if it were identified.

Finally, continuous processing equipment would work through the nickel stockpile quickly. With this unusual product, it might be preferable to recover it over time using a system that could be modified to accommodate different needs.

For the reasons of materials of construction, equipment and operating complexity, and flexibility in product quality verification, we select batch distillation as the preferred approach.

Batch distillation designs.

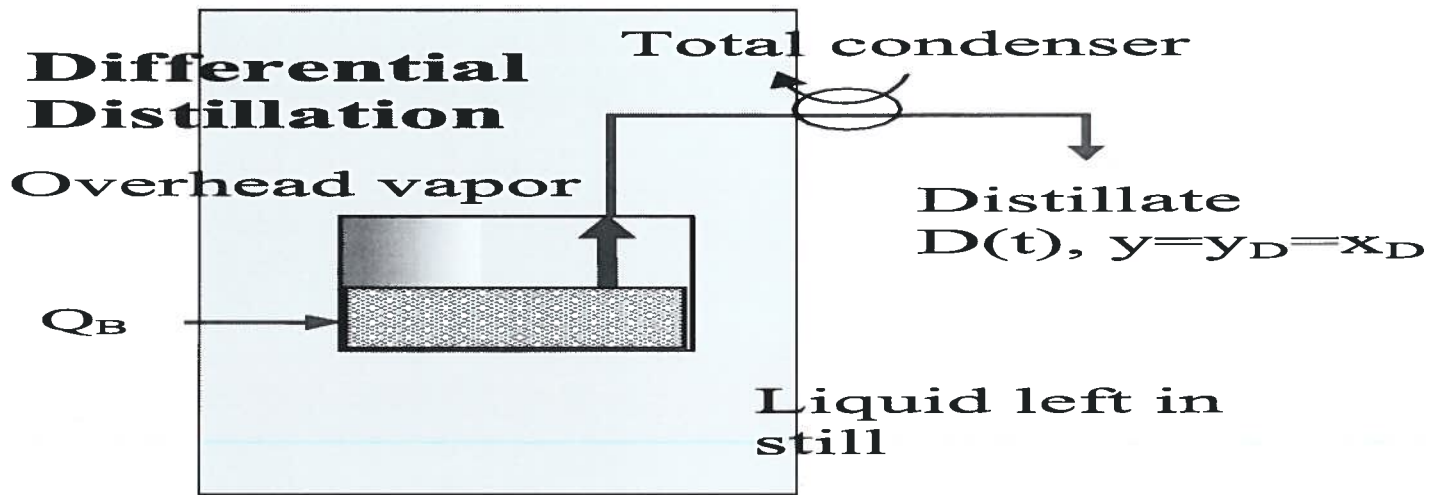


Figure 5. Batch (differential) distillation system. mole units.

In batch distillation, a stream is continuously taken. The differential component balance for this process is

$$-\frac{d(W \cdot x_w)}{dt} = -W \cdot \frac{dx_w}{dt} - x_w \cdot \frac{dW}{dt}$$

The left hand side shows the total change of component w on the right hand side show the change in the total amount of liquid. The distillate composition at any time is in equilibrium with the liquid in the still. If we assume an equilibrium equation of the type, $y=kx$. Eq. 3 can be written as

$$\ln \frac{W_0}{W} = \frac{1}{\alpha - 1} \cdot \left[\ln \frac{x_0}{x} + \alpha \cdot \ln \frac{(1 - x_0)}{(1 - x)} \right]$$

Where α is the relative volatility of the mixture. W is the material left in the still, and the concentration of the liquid in the still. The relative volatility is defined as:

$$\alpha_{Ni,Tc} = \frac{y_{Ni} / x_{Ni}}{y_{Tc} / x_{Tc}} = \frac{\gamma_{Ni} \cdot P_{Ni}^{sat}}{\gamma_{Tc} \cdot P_{Tc}^{sat}}$$

The relative volatility, $\alpha_{Ni,Tc}$, goes from 5.4×10^8 at the boiling point of nickel. These numbers suggest very high temperatures. However, at lower temperatures, the process will need a sweep gas or vacuum to operate the process.

In a batch distillation, the concentration of the volatile component in the distillate changes as more material leaves the bottoms.

There are two alternative batch designs for generating higher purity products:

- Batch rectification with constant reflux (the distillate compositions has a higher purity but still varies with time), and
- Batch rectification with variable reflux (the reflux conditions are varied continuously to achieve constant composition distillate)

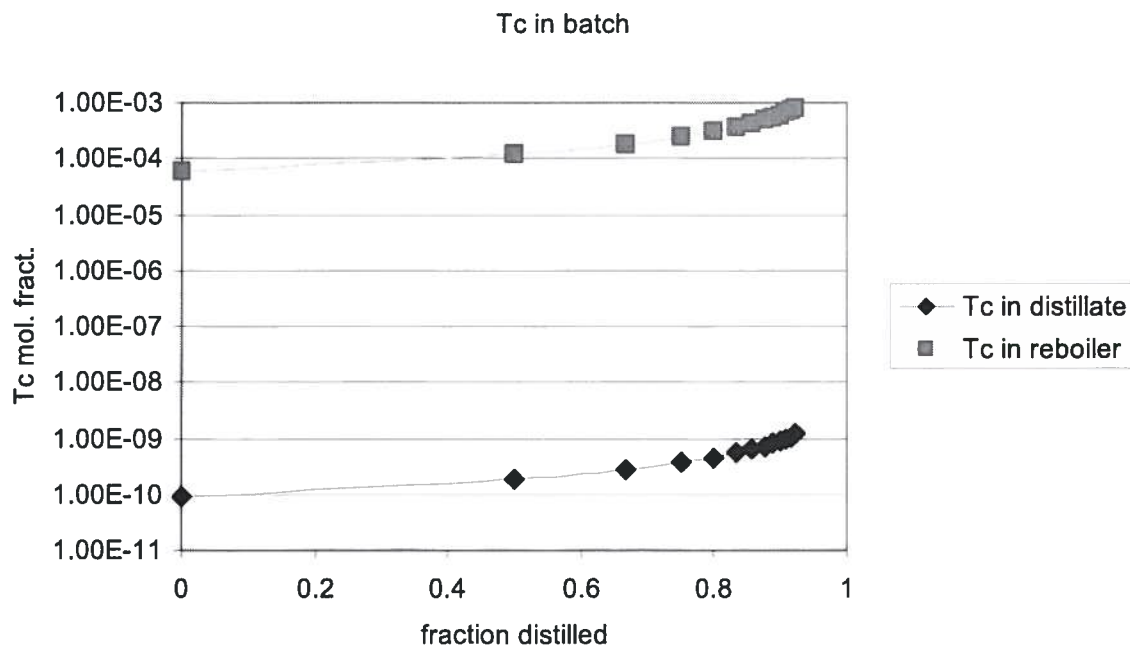
Using the second technique would generate a purified nickel product with constant ^{99}Tc levels. However, the need for liquid metal condensation and recycle imposes complexity and equipment costs that probably are not needed for the intended market.

Computational examples.

Computational examples of the batch distillation equations can be used to demonstrate how the process should function. The three examples shown below include the technetium levels in the distillate (nickel-rich product) and the reboiler (technetium-rich product), the technetium levels in the distillate product for various starting levels of technetium contamination, and the two product streams for the highest concentration of technetium contamination anticipated in the recycling effort (100 ppm). These help demonstrate the utility of the process. All of these examples assume an operating temperature of 2415 K, the boiling point of nickel. Improved separations can be achieved by lowering the operating temperature.

Assuming a 10 ppm concentration of ^{99}Tc in the bottoms at the start of batch distillation, Figure 4 shows the increase of technetium in the reboiler and distillate streams as Ni-rich metal is taken overhead.

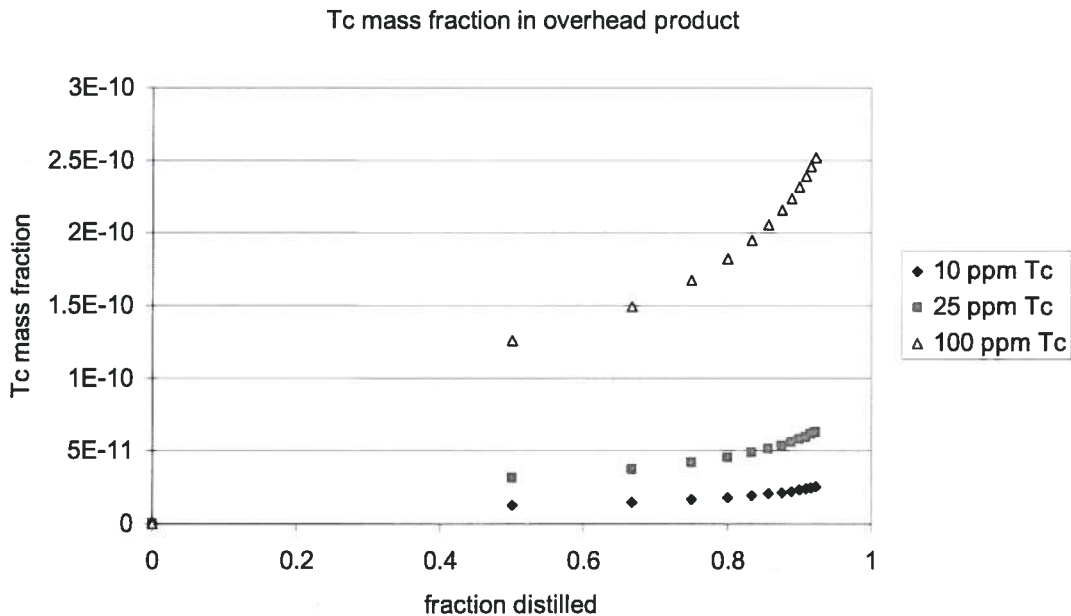
Figure 6. Technetium levels, distillate stream and reboiler. 2415 K.



The technetium concentration in the reboiler increase by an order of magnitude as the metal is removed from the still. The endpoint for distillation is also a design choice. Depending on what the starting material contains, the process would have the flexibility to make a consistent composition in the overhead product. However, this would change the operating schedule and conditions, which might be difficult to monitor.

We have selected distilling 90% of the reboiler contents as the endpoint for this process. This reduces the amount of waste material by about an order of magnitude. Material that could be further purified could simply be recycled through the process.

Figure 7. Effect of feed concentration on technetium in distillate. 2415 K.



A comparison of the technetium mass fraction to data in Table 4 shows that the 100 ppm technetium feed material in result in overhead products that exceed the limits permitted for scrap metal recycle in Germany.

Figure 8. Distillate and reboiler concentrations of technetium. 100 ppm feed. 2415 K.

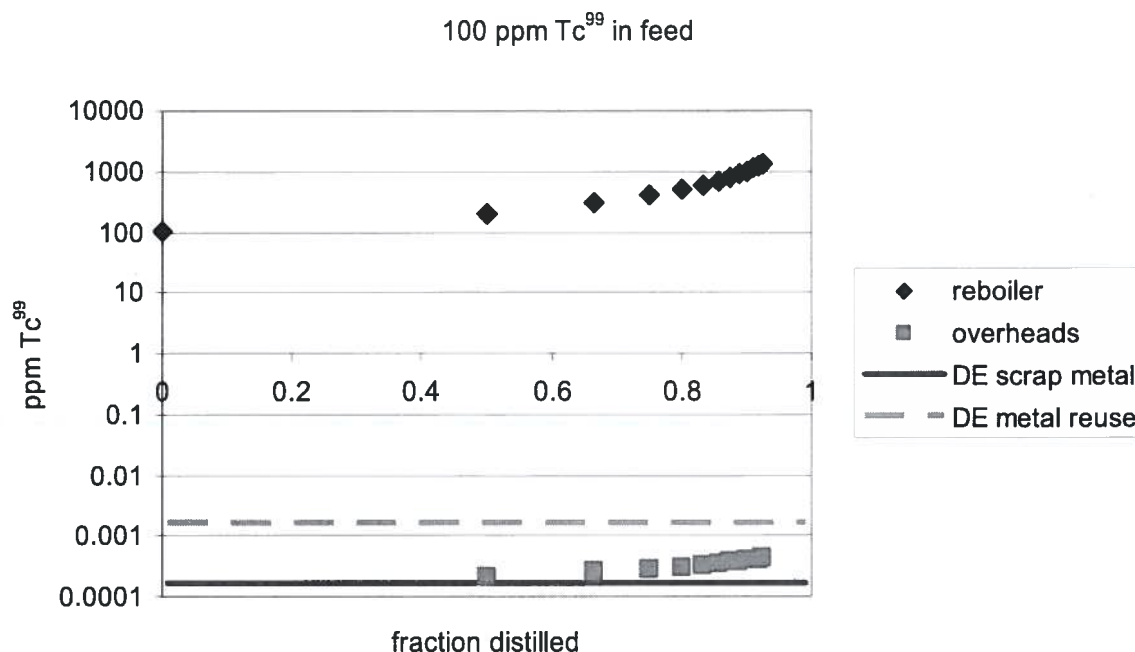


Figure 6 shows both the distillate and bottoms concentrations for the highest expected feed concentration of technetium, 100 ppm.

These calculations show that the separation should be excellent, even near the boiling point of nickel. Lower operating temperatures will improve the separation further, but require vacuum or sweep gas systems that will increase the capital and/or operating costs. As shown by Figure 6, even high technetium concentrations should be manageable in the batch system. A key requirement is that the fluid flow in the device be set up to minimize and eliminate droplet formation and carryover. Any droplets generated from the bottoms would severely contaminate the overhead stream, and greatly decrease the process efficiency.

Process options

Operating temperature. The sensitivity of the relative volatility to temperature provides options for the distillation process. Operating the distillation process at the boiling point of nickel generates vapor flow due to the vaporization of the metal, reducing the need for mechanical equipment. However, the materials of construction might need to be ceramics that could take the high temperatures. Some choices for materials of construction include:

- Commercial alumina – temperatures to 1900 K,
- Pure alumina – temperatures to 2200 K, and
- Zirconia – temperatures to 2700 K.

If the process is operated at temperatures less than the boiling point of nickel, vapor flow must be induced by other means, for example, by using an inert sweep gas or pulling a vacuum on the system. A sweep gas might have to be inert to reduce the oxidation of the metal, which would result in slag that could contaminate the product powder. Oxidation rates for nickel and technetium vapor are probably available in the literature, but have not been included in this report.

Table 6 shows the partial pressures of each component as well as the relative volatility for operating temperatures from the melting point of nickel to its boiling point. Based on the available vapor pressure data, the relative volatility increases as temperature decreases, meaning that the nickel purity will increase at lower operating temperatures. However, the total system pressure is very near that of the nickel partial pressure. Near its melting point, nickel vapor would only have a partial pressure of ~ 0.002 atmospheres.

Process vacuum. There are a number of commercial processes that need to operate at less than atmospheric pressure. While nearly any vacuum may be achievable, the costs may be prohibitive. In general, industrial compressors/vacuum pumps are most efficient and cost effective if they step the pressure up or down by factors of 2 to 5. Higher compressions or lower vacuums are achieved by using equipment in series. Using a guideline of one vacuum system per pressure changes of a factor of three, we can guess at the practical number of pumps in series needed to attain a specific vacuum. Operating the system at 2200 K might require 1-2 vacuum systems in series, while operating the system at 1900 K might require 4 systems in series. Conceptually, the nickel could be sublimed away from a high temperature solid with very high purity, but the vacuum system would be huge. A critical issue for a vacuum system in the process is protecting it from metal condensation in its working parts.

Gas sweeps through or over the molten metal could be used to convey metal vapor to a condensation system for making powder or plating the metal for recovery. The comments to the right of Table 6 suggest options that might be economical for different operating temperatures (pressures).

We will use a temperature of 2200 K as a reasonable pressure condition at which the process can be operated, either with one vacuum system or an inert gas sweep.

Table 6. Metal partial pressures and relative volatility vs. T.

T, K	P_{Ni} , bar	P_{Tc} , bar	P_{Ni}/P_{Tc}
1700	0.0017	2.08E-12	8.17E+08
1800	0.0057	2.60E-11	2.19E+08
1900	0.017	2.50E-10	6.80E+07
2000	0.044	1.94E-09	2.27E+07
2100	0.105	1.23E-08	8.54E+06
2200	0.23	6.60E-08	3.48E+06
2300	0.476	3.03E-07	1.57E+06
2400	0.893	1.21E-06	7.38E+05
2415	0.985	1.47E-06	6.70E+05

Air sweep

High vacuum

Inert gas sweep

Low vacuum

Atmospheric operation

Process economics.

Table 7 lists five different process operating options. Of these, method 2 seems to be a reasonable choice for estimating the process economics. This process cost estimate is based on methods used for estimating chemical plant costs (Peters and Timmerhaus). These methods are often within 25% of the capital and operating costs. More accurate cost estimates would be based on equipment and construction quotations.

Process economics will be done on method 2, a system operating near ~ 0.23 atmospheres total pressure and a temperature of 2200 K.

Table 7. Summary of process operating options.

Method	T, K	Vapor Flow	Comments
1. Atmospheric distillation	2415	Pressure-driven flow from reboiler	Pure Tc melts at 2430 K; no vacuum or inert flow is needed; ZrO ₂ as material of construction?
2. Low vacuum distillation	2200	$P_{Ni} = 0.23$ bar	Inductor-type system used to generate low vacuum
3. High vacuum distillation	1900	$P_{Ni} = 0.017$ bar	Probably needs primary and secondary vacuum systems
4. Sweep gas	1730	$P_{Ni} = 0.0025$ bar	Nitrogen (recycled?), or possibly, air. Lowest possible temperature; assume that metal reaches 1/3 of its saturation pressure in the gas; lance or sweep over liquid surface; alumina materials of construction to 1750 C.
5. Continuous flash system with sweep gas	1730		Low liquid feed rate and removal; high gas sweep rate with condensation of nickel on nickel screens or plates in vapor effluent

Selection of process equipment.

While it is possible to design the entire process equipment system for this application, it is usually desirable to select process equipment that is already manufactured and adapt it to the specific process requirements. In this case, we believe that currently manufactured vacuum induction furnaces would be appropriate for melting the nickel ingots. These systems generate heat through induction heating of a conductive medium, and hold the melt in a crucible surrounded by water-cooled magnetic coils. They provide clean, energy-efficient (> 95%), well-controlled melting and are linked with vacuum systems to reduce oxygen reactions that would lead to slags. Typical commercial sizes range from 1 kg to 100 ton capacities. Typical metal melting applications range from iron, steel, copper, aluminum and nickel alloys.

The induction coil operating frequencies range from 60 Hz to 10 kHz, with the higher frequencies resulting in faster heating. A preheated 1 metric ton furnace can heat an iron cold charge in one hour. The operating furnace emits a hum due to magnetostriction that can be used to identify proper operation. Charged materials must be clean of oxidation products and of known composition. There are several industrial scale induction furnace suppliers in the U.S. that can provide special designs with lances, ladles, and automated casting systems (the ⁹⁹Tc-rich

bottoms material could be recast into ingots for future rework or disposal). There are mini induction vacuum systems that could be used to verify scale-up. Using commercial furnace designs will lower the risk associated with this process.

Recovering the nickel from the vapor phase could be done via cold trapping prior to a vacuum system, by physical vapor deposition from a sweep gas, or by powder formation from the gas phase. There is some prior art for generating powders from condensing solids in compression/expansion flows that could be quite useful.

Batch process cycle time.

The major processing steps are: 1) load nickel ingots, 2) heat the furnace to the processing temperature, 3) distill nickel vapor, and 4) discharge the residual metal to ingot molds for retention or rework. Steps 1 and 4 should be accomplished in minutes. Step 2 should take an hour for a one metric ton induction furnace. Step 3 depends on the size of the charge, the size of the vapor handling system, and the partial pressure of nickel in the vapor phase (which is affected by the processing temperature).

The typical vacuum system on an miniscale induction furnace has a capacity of 10 cfm (standard temperature and pressure). This capacity can be evaluated for higher temperatures by estimating the moles of gas occupying cfm at the temperature of interest, and knowing the partial pressure of the metal vapor at those conditions. The time needed to distill 90% of the 19 kg nickel charge in this furnace can be estimated directly.

Table 8. Time to distill a 1 metric ton batch vs. T.

T, K	P _{ni} , bar	n/V, gmol/cc	gmol/min	kg/min	Time, hr	At low temperatures, the metal vapor pressure is low, so large volumes of gas need to be processed. At the highest temperature, the 10 cfm system would require about 14 hours to process all
1725	0.0023	1.62503E-08	0.004602	0.00027	1073.8	
1800	0.0057	3.85944E-08	0.010929	0.000641	452.1	
1850	0.00997	6.56818E-08	0.018599	0.001092	265.7	
1900	0.017	1.09048E-07	0.030879	0.001812	160.0	
1925	0.0217	1.37389E-07	0.038904	0.002283	127.0	
2200	0.23	1.27417E-06	0.360804	0.021176	13.7	

the vapor. We will assume that the gas processing system can be expanded by a factors of ~ 4 - 6 (adding gas handling systems in parallel for example). **This could allow a total cycle time of 4 hours for a batch.**

Base case design (case I).

Case I.

- Process equipment costs as estimated
- Utilities as estimated
- Land and building available
- No cost for the contaminated nickel
- 90 % of the nickel is recovered to a purity meeting German scrap recycle specifications
- Disposal of the residual nickel is not in these budgets

Table 9. Plant capacity assumptions. Case I.

Ni (smelted at Paducah)			
non-radioactive	7718000	kg	17000000 lb
contaminated	9080000	kg	20000000 lb
	16798000	kg	
plant life	10		
operating days/yr	250		
kg/day	6719.2		
operating hours	24	hr	
batch time	4	hr	
batches/day	6		

Table 10. Utilities. Case I.

utilities	mini furnace	1 metric ton charge
electricity	20 kVA	213 kVA
water, 45 psig	15 gpm	160 gpm
air, 80 psig	-	
gas supply: Ar, N2	1 cubic foot	11 ft ³

Table 11. Purchase equipment costs. Case I.

Purchased equipment cost	
1000 kg charge capacity	
Item	cost
vacuum induction furnace	\$850,000
N ₂ storage + lance	\$50,000
cool down tube	\$150,000
cyclone	\$40,000
baghouse	\$50,000
	\$1,140,000

Table 12. Capital investment costs. Case I.

Capital Investment Analysis							
Ni vacuum furnace							
			Factors				
Direct costs		k \$'s	% FCI	% Eqt.\$	% DC	%TCI	range
equipment + installation							40-60% FCI
purchased equipment cost (PEC)		\$1,140					15-40% FCI
installation		\$285		25			25-55% PEC
instrumentation & controls		\$114		10			6-30% PEC
pipng		\$114		10			10-80% PEC
electrical, installed		\$228		20			10-40% PEC
buildings		\$114		10			10-70% PEC
service facilities	{retrofit}	\$0		0			40-100% PEC
land		\$0		0			4-8% PEC
	DC	\$1,995					
Indirect costs							15-30% FCI
engineering/supervision		\$299			15		5-30% DC
construction expense		\$299			15		6-30% DC
contingency		\$288	10				5-15% FCI
Fixed capital investment (FCI)		\$2,882					
Working capital		\$509					15 10-20% TCI
Total capital investment (TCI)		\$3,390					
this table provides a basis of cost factors for determining DC, FCI and TCI from the PEC							

Table 13. Utilities per batch

Nitrogen			
T, K	P _{ni} , bar	Time, hr	SCF required
1725	0.0023	1074	101960
1800	0.0057	452	41142
1850	0.00997	266	23521
1900	0.017	160	13795
1925	0.0217	127	10807
2100	0.23	13	1020
Cost			\$0.20
			\$203.92
Cooling water			
coil cooling		38418	gal/batch
metal cooling		6136	gal/batch
		44553	gal/batch
Cost		\$1.00	1000 GAL
		\$44.55	per batch
Electricity			
electricity		2.16	kwh/kg
		2161	kwh/batch
cost		\$0.04	kwh
		\$86.43	per batch

Table 14. Total product cost per year. Case I.

Ni vacuum furnace		Total product costs			1500 batches/year
		direct costs	\$1,995,000		1995
Manufacturing costs		fixed capital invest.	\$2,882,000		2882
		TCI	\$3,390,000		3390
Basis:	1 year	land + buildings =	\$115,000		
		utilities	\$502,500		
I. Manufacturing costs		factors			range
		\$/batch	% TPC	% OL	% FCI
direct production costs					60% TPC
equipment/parts		\$0			10-50% TPC
operating labor		\$633,182	20		10-20% TPC
direct supervisory and clerical		\$158,295	5	25	10-25% OL
utilities		\$502,500			2-10% FCI
maintenance and repairs		\$339,000			10-20% FCI
operating supplies		\$33,900			0.5-1% FCI
lab charges		\$63,318	2	10	10-20% OL
patents/royalties		\$0	0		0-6% TPC
fixed charges					10-20% TPC
depreciation		\$288,200			10% FCI
local taxes		\$39,900			2 1-4% DCI
insurance		\$33,900			0.4-1% FCI
rent		\$11,500			10 8-12% land + buildings
plant overhead		\$316,591	10		5-15% TPC
		\$2,420,286			
II. General expenses					
administrative		\$126,636	4		2-6% TPC
distribution and selling		\$316,591	10		2-20% TPC
R&D		\$158,295	5		5% TPC
financing/interest		\$144,100			5 0-10% TCI
	\$1,393,000	\$745,623	56		
III. Total product costs		\$3,165,909			T

Table 15. Net profit per year. Case I.

IV. Gross earnings cost	\$1,044,750		
material processed	1,500,000	kg	
contaminant-free product	1,350,000	kg	
Ni price	\$14	per kg	
gross sales	\$18,900,000		
Product cost, each	\$3,165,909		
net profit	\$15,734,091		

Summary. Case I.

This case shows that, if the nickel purified by this process could be sold near the current market price, the plant would make a profit. Also, there is no charge assessed for the final disposition of the residual nickel as well. Costs from this estimating technique can easily vary +/- 25%. The

3,300 pounds - 20 million pounds
 3,300 pounds - 20 million pounds
 Price of Ni:
 21,789,100
 16.14
 Now 16.14?

plant payroll is about \$1,000,000 per year, including operating labor, direct supervisory and clerical, administrative, and distribution & sales.

Case II.

The assumptions for Case II are:

- The equipment costs twice the original estimate
- The costs factors for other elements are at the top of their normal ranges
- The land and building is still available, and
- The utility costs are twice as high as the original estimate.

Table 16 . Capital investment costs. Case II.

Capital Investment Analysis							
Ni vacuum furnace							
			Factors				
Direct costs		k \$'s	% FCI	% Eqt.\$	% DC	%TCI	range
equipment + installation							40-60% FCI
purchased equipment cost (PEC)		\$2,280					15-40% FCI
installation		\$1,254		55			25-55% PEC
instrumentation & controls		\$684		30			6-30% PEC
pipng		\$1,824		80			10-80% PEC
electrical, installed		\$912		40			10-40% PEC
buildings		\$228		10			10-70% PEC
service facilities {retrofit}		\$0		0			40-100% PEC
land		\$0		0			4-8% PEC
	DC	\$7,182					
Indirect costs							15-30% FCI
engineering/supervision		\$1,077			15		5-30% DC
construction expense		\$1,077			15		6-30% DC
contingency		\$1,037	10				5-15% FCI
Fixed capital investment (FCI)		\$10,374					
Working capital		\$1,831					15 10-20% TCI
Total capital investment (TCI)		\$12,205					

Table 17. Total product costs. Case II.

Ni vacuum furnace		Total product costs			1500 batches/year
		direct costs	\$7,182,000		7182
Manufacturing costs		fixed capital invest.	\$10,374,000		10374
		TCI	\$12,205,000		12205
Basis:	1 year	land + buildings =	\$250,000		
		utilities	\$1,000,000		
I. Manufacturing costs		factors			range
		\$/batch	% TPC	% OL	% FCI
direct production costs					60% TPC
equipment/parts		\$0			10-50% TPC
operating labor		\$1,904,245	20		10-20% TPC
direct supervisory and clerical		\$476,061	5	25	10-25% OL
utilities		\$1,000,000			2-10% FCI
maintenance and repairs		\$1,220,500			10-20% FCI
operating supplies		\$122,050			0.5-1% FCI
lab charges		\$190,425	2	10	10-20% OL
patents/royalties		\$0	0		0-6% TPC
fixed charges					10-20% TPC
depreciation		\$1,037,400			10% FCI
local taxes		\$143,640			2 1-4% DCI
insurance		\$122,050			0.4-1% FCI
rent		\$25,000			10 8-12% land + buildings
plant overhead		\$952,123	10		5-15% TPC
		\$7,193,494			
II. General expenses					
administrative		\$380,849	4		2-6% TPC
distribution and selling		\$952,123	10		2-20% TPC
R&D		\$476,061	5		5% TPC
financing/interest		\$518,700			5 0-10% TCI
	\$4,189,340	\$2,327,733	56		
III. Total product costs		\$9,521,227			T

Table 18. Net profit per year. Case II.

IV. Gross earnings cost	\$3,142,005		
material processed	1,500,000	kg	
contaminant-free product	1,350,000	kg	
Ni price	\$14	per kg	
gross sales	\$18,900,000		
Product cost, each	\$9,521,227		
net profit	\$9,378,773		

Summary. Case II.

This plant would still generate a profit. While the payroll appears to be significantly larger than for Case I, this is due to the fact that equipment costs have increased, and the labor costs scale with these. The manpower needs of the plant are probably similar between Case I and Case II. The following issues need to be addressed during process design and/or scale-up.

- What might be the selling price of the purified nickel?

- What might be the price for burial of 3.7 million kg's of nickel contaminated at a level ten times that of the current stock?
- Will the powder particle formation process work?
- What is the true furnace cost with the needed vapor handling systems?
- What purity of nitrogen is needed to prevent oxide formation?
- What are the energy efficiencies for heating, inert gas use, cooling the product, and cooling the magnetic coils?
- Can the graphite liner of the current induction furnace design be used at a temperature 200 C greater than that specified?

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Appendix 1. 902 KAR 100:021. Disposal of radioactive material.

902 KAR 100:021. Disposal of radioactive material.

RELATES TO: KRS 211.842-211.852, 211.990(4), 10 C.F.R. 20.2001-2007, Appendix G-20.2001-2401, 61.80(1)

STATUTORY AUTHORITY: KRS 13B.170, 194A.050(1), 211.090(3), 211.844

NECESSITY, FUNCTION, AND CONFORMITY: KRS 211.844 requires the Cabinet for Health Services to provide by administrative regulation for the registration and licensing of the possession or use of a source of ionizing or electronic product radiation and the handling and disposal of radioactive waste. This administrative regulation provides waste disposal limitations for radioactive material, and shall apply to a person disposing of radioactive material or waste. This administrative regulation shall not establish standards governing naturally occurring radioactive material (NORM) and waste.

Section 1. General Requirements. (1) A person or licensee shall dispose of radioactive material or waste only:

- (a) By transfer to an authorized recipient as provided in 902 KAR 100:040, Section 13, or 902 KAR 100:022;
 - (b) By decay in storage;
 - (c) By release in an effluent within the limits in 902 KAR 100:019, Section 10; or
 - (d) As authorized by Sections 2, 3, 4, or 5 of this administrative regulation.
- (2) A person shall be specifically licensed to receive waste containing radioactive material or waste from other persons for:
- (a) Treatment prior to disposal;
 - (b) Treatment or disposal by incineration;
 - (c) Decay in storage; or
 - (d) Disposal at a land disposal facility licensed under 902 KAR 100:022.

Section 2. Method for Obtaining Approval of Proposed Disposal Procedures. A person, licensee, or applicant for a license may apply to the cabinet for approval of a proposed procedure, not authorized in 902 KAR 100:020, 100:021, 100:022, 100:050, and 100:073, to dispose of radioactive material or waste generated by their activity. An application shall include:

- (1) A description of the waste containing radioactive material to be disposed of, including the:
 - (a) Physical and chemical properties important to risk evaluation; and
 - (b) Proposed manner and conditions of waste disposal;
- (2) An analysis and evaluation of pertinent information on the nature of the environment;
- (3) The nature and location of other potentially affected licensed and unlicensed facilities; and
- (4) An analysis and a procedure to ensure doses are maintained ALARA and within the dose limits in 902 KAR 100:019, Sections 3, 8, 9, and 10.

Section 3. Disposal by Release into Sanitary Sewerage. (1) A person or licensee may discharge licensed material into sanitary sewerage under the following conditions:

- (a) The material shall be readily soluble, or shall be readily dispersible biological material, in water;
 - (b) The quantity of licensed or other radioactive material that the licensee released into the sewer in one (1) month, divided by the average monthly volume of water released into the sewer by the licensee, shall not exceed the concentration in 902 KAR 100:019, Section 44, Table III;
 - (c) For the release of more than one (1) radionuclide, the following conditions shall be satisfied:
 - 1. The licensee shall determine the fraction of the limit in 902 KAR 100:019, Section 44, Table III, represented by discharges into the sanitary sewerage by dividing the actual monthly average concentration of each radionuclide released by the licensee into the sewer by the concentration of that radionuclide in 902 KAR 100:019, Section 44, Table III; and
 - 2. The sum of the fractions for each radionuclide required by subsection (1)(c)1 of this section shall not exceed unity; and
 - (d) The total quantity of licensed and other radioactive material that the licensee releases into the sewerage system in a year shall not exceed five (5) curies (185 GBq) of hydrogen-3, one (1) curie (37 GBq) of carbon-14, and one (1) curie of other radioactive materials combined.
- (2) Excreta from an individual undergoing medical diagnosis or therapy with radioactive material shall not be subject to the limitations contained in subsection (1) of this section.

Section 4. Treatment or Disposal by Incineration. A licensee may treat or dispose of licensed material by incineration only:

- (1) In the amounts and forms specified in Section 5 of this administrative regulation; or
- (2) As specifically approved by the cabinet and authorized by Section 2 of this administrative regulation.

Section 5. Disposal of Specific Wastes. (1) A person or licensee may dispose of the following radioactive material without regard to its radioactivity:

- (a) 0.05 microcurie or less of hydrogen-3, or tritium, carbon-14, or iodine-125 per gram of medium used for liquid scintillation counting or in vitro clinical or in vivo laboratory testing; and
 - (b) 0.05 microcurie (1.85 kBq) or less of hydrogen-3, carbon-14, or iodine-125 per gram of animal tissue averaged over the weight of the entire animal.
- (2) A licensee shall not dispose of tissue pursuant to subsection (1)(b) of this section in a manner that may permit its use as food for a human or as animal feed.
- (3) A licensee shall maintain records required by Section 11 of this administrative regulation.
 - (4) A licensee shall comply with other applicable federal, state, and local regulations governing other toxic or hazardous properties of these materials.

Section 6. Classification of Radioactive Waste for Near-Surface Disposal. (1) Considerations. Determination of the classification of waste shall be given the following considerations:

(a)1. The concentration of long-lived radionuclides, and their shorter-lived precursors, whose potential hazard shall persist long after a precaution such as an institutional control, improved waste form, and deeper disposal have ceased to be effective.

2. The precaution delays the time long-lived radionuclides may cause an exposure.

3. The magnitude of the potential dose is limited by the concentration and availability of the radionuclide at the time of exposure; and

(b) The concentration of a shorter-lived radionuclide for which a requirement on an institutional control, waste form, and disposal methods are effective.

(2) Classes of waste.

(a)1. Class A waste shall be segregated from other waste classes at the disposal site, except for waste described at subparagraph 2 of this paragraph.

2. The physical form and characteristics of Class A waste shall meet the minimum requirements in Section 7 of this administrative regulation.

3. If Class A waste also meets the stability requirements in Section 7(2) of this administrative regulation, it shall not be necessary to segregate Class A waste for disposal.

(b)1. Class B waste shall meet more rigorous requirements on waste form to ensure stability after disposal.

2. The physical form and characteristics of Class B waste shall meet both the minimum and stability requirements in Section 7 of this administrative regulation.

(c)1. Class C waste shall meet more rigorous requirements on waste form to ensure stability and shall require additional measures at the disposal facility to protect against inadvertent intrusion.

2. The physical form and characteristics of Class C waste shall meet both the minimum and stability requirements in Section 7 of this administrative regulation.

(3) Classification determined by long-lived radionuclides. If the waste contains only a radionuclide in Table 1 of this subsection, classification shall be determined as follows:

(a) If the concentration does not exceed one-tenth (0.1) times the value in Table 1, the waste shall be Class A.

(b) If the concentration exceeds one-tenth (0.1) times the value, but does not exceed the value in Table 1, the waste shall be Class

C.

(c) If the concentration exceeds the value in Table 1, the waste shall not generally be acceptable for near-surface disposal.

(d) For waste containing a mixture of radionuclides in Table 1, the total concentration shall be determined by the sum of fractions rule described in subsection (7) of this section.

TABLE 1	
Radionuclide	Concentration curies/cubic meter
C-14	8
C-14 in activated metal	80
Ni-59 in activated metal	220
Nb-94 in activated metal	0.2
Tc-99	3
I-129	0.08
Alpha emitting transuranic radionuclides with half-life greater than five (5) years	100*
Pu-241	3500*
Cm-242	20000*
Ra-226	100*

*Units are nanocuries per gram.

(4) Classification determined by short-lived radionuclides. If the waste contains none of the radionuclides in Table 1 of subsection (3) of this section, classification shall be determined based on the concentrations shown in Table 2 of this subsection. If a radionuclide is not in Table 2, it shall not be considered in determining the waste class.

(a) If the concentration does not exceed the value in Column 1, the waste shall be Class A.

(b) If the concentration exceeds the value in Column 1, but does not exceed the value in Column 2, the waste shall be Class B.

(c) If the concentration exceeds the value in Column 2, but does not exceed the value in Column 3, the waste shall be Class C.

(d) If the concentration exceeds the value in Column 3, the waste shall not generally be acceptable for near-surface disposal.

(e) For waste containing a mixture of the radionuclides in Table 2, the total concentration shall be determined by the sum of fractions rule described in subsection (7) of this section.

TABLE 2			
Radionuclide	Concentration, curies/cubic meter		
	Column 1	Column 2	Column 3
Total of all radionuclides with less than five (5) year half-life	700	*	*
H-3	40	*	*
Co-60	700	*	*
Ni-63	3.5	70	700
Ni-63 in activated metal	35	700	7000
Sr-90	0.04	150	7000

Cs-137	1	44	4600
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*Limits have not been established for a radionuclide in Class B or C waste. Practical considerations, such as the effects of external radiation and internal heat generation on transportation, handling, and disposal, limit the concentrations for these wastes. This waste shall be Class B unless the concentrations of other radionuclides in Table 2 determine the waste to be Class C independent of these radionuclides.

(5) Classification determined by both long-lived and short-lived radionuclides.

(a) If the waste contains a mixture of radionuclides, some in Table 1 of this section, and some in Table 2 of this section, classification shall be determined as follows:

(b) If the concentration of a radionuclide in Table 1 does not exceed one-tenth (0.1) times the value in Table 1, the class shall be determined by the concentration of a radionuclide in Table 2.

(c) If the concentration of a radionuclide in Table 1 exceeds one-tenth (0.1) times the value, but does not exceed the value in Table 1, the waste shall be Class C, if the concentration of a radionuclide in Table 2 does not exceed the value shown in Column 3 of Table 2.

(6) Classification of waste with a radionuclide other than those in Tables 1 and 2. If the waste contains none of the radionuclides in Table 1 or 2 of this section, the waste shall be Class A.

(7) The sum of fractions rule for mixtures of radionuclides. The following shall be considered in determining classification for waste that contains a mixture of radionuclides:

(a) The sum of fractions shall be determined by dividing each radionuclide concentration by the appropriate limit and adding the resulting values.

(b) The appropriate limit shall be taken from the same column of the same table.

(c) The sum of the fractions for the column shall be less than one (1.0) if the waste class is determined by that column.

(d) Example: A waste contains Sr-90 in a concentration of fifty (50) curies/cubic meter and Cs-137 in a concentration of twenty-two (22) curies/cubic meter. Since the concentrations both exceed the values in Column 1, Table 2, they shall be compared to Column 2 values. For Sr-90 fraction, $50/150 = 0.33$; for Cs-137 fraction, $22/44 = 0.5$; the sum of the fractions = 0.83. Since the sum is less than one (1.0), the waste shall be Class B.

(8) Determination of concentrations in waste.

(a) If there is reasonable assurance that an indirect method may be correlated with an actual measurement, the concentration of a radionuclide may be determined by an indirect method, such as use of a scaling factor which relates the inferred concentration of one (1) radionuclide to another that is measured or radionuclide material accountability.

(b) If the units are expressed as nanocuries per gram, the concentration of a radionuclide may be averaged over the volume or weight of the waste.

Section 7. Radioactive Waste Characteristics. (1) The following minimum requirements for each class of waste are intended to facilitate handling and provide protection of health and safety of personnel at the disposal site:

(a) Waste shall be packaged in conformance with the conditions of the license issued to the site operator to which the waste shall be shipped. If the conditions of the site license are more restrictive than the provisions of this administrative regulation, the site license conditions shall govern.

(b) Waste shall not be packaged for disposal in a cardboard or fiberboard box.

(c) Liquid waste shall be solidified or packaged in sufficient absorbent material to absorb twice the volume of the liquid.

(d) Solid waste containing liquid shall contain as little freestanding and noncorrosive liquid as is reasonably achievable. The liquid shall not exceed one (1) percent of the volume.

(e) Waste shall not be readily capable of:

1. Detonation;
2. Explosive decomposition or reaction at normal pressures and temperatures; or
3. Explosive reaction with water.

(f) Waste shall not contain, or be capable of generating, quantities of toxic gases, vapors, or fumes harmful to a person transporting, handling, or disposing of the waste. This shall not apply to radioactive gaseous waste packaged in accordance with paragraph (h) of this subsection.

(g) Waste shall not be pyrophoric. Pyrophoric material contained in waste shall be treated, prepared, and packaged to be nonflammable.

(h) Waste in a gaseous form shall be packaged at a pressure that shall not exceed one and five-tenths (1.5) atmospheres at twenty (20) degrees Centigrade. Total activity shall not exceed 100 curies per container.

(i) Waste containing hazardous, biological, pathogenic, or infectious material shall be treated to reduce to the maximum extent practicable the potential hazard from the nonradiological material.

(2) Stability shall ensure that the waste shall not structurally degrade and affect overall stability of the site through slumping, collapse, or other failure of the disposal unit and lead to water infiltration. Stability shall also be a factor in limiting exposure to an inadvertent intruder, since it provides a recognizable and nondispersible waste. The following requirements shall provide stability of the waste:

(a) Waste shall have structural stability.

1. A structurally-stable waste form shall maintain its physical dimension and its form under expected disposal conditions, such as:

- a. Weight of overburden and compaction equipment;
- b. Presence of moisture and microbial activity; and
- c. Internal factors such as radiation effects and chemical changes.

2. Structural stability may be provided by:

- a. The waste form itself;
- b. Processing the waste to a stable form; or
- c. Placing the waste in a disposal container or structure that provides stability after disposal.

(b) Unless otherwise exempted in subsection (1)(c) and (d) of this section, liquid waste, or waste containing liquid, shall be converted into a form that contains as little free standing and noncorrosive liquid as is reasonably achievable. The liquid shall not exceed one (1) percent of the volume of the waste if the waste is in a disposal container designed to ensure stability, or five-tenths (0.5) percent of the volume of the waste for waste processed to a stable form.

(c) Void spaces within and between the waste and its package shall be eliminated.

Section 8. Labeling. Each package of waste shall be clearly labeled to identify if it is Class A, Class B, or Class C waste, in accordance with Section 6 of this administrative regulation.

Section 9. Transfer for Disposal and Manifests. (1) The requirements of this section and Section 10 of this administrative regulation shall:

(a) Control transfers of low-level radioactive waste intended for disposal at a land disposal facility as established in 902 KAR 100:022;

(b) Establish a manifest tracking system; and

(c) Supplement existing requirements concerning transfers and recordkeeping for the wastes being transferred.

(2) A shipment of radioactive waste intended for disposal at a licensed land disposal facility shall be accompanied by a shipment manifest as specified in Section 10(1) of this administrative regulation.

(3) The shipment manifest shall include a certification by the waste generator as specified in Section 10(12) of this administrative regulation.

(4) A person involved in the transfer for disposal and disposal of waste, including the waste generator, waste collector, waste processor, and disposal facility operator, shall comply with the requirements specified in Section 10(13) of this administrative regulation.

Section 10. Requirements for Low-level Waste Transfers Intended for Disposal at Land Disposal Facilities and Manifests. (1) A waste generator, collector, or processor who transports, or offers for transportation, low-level radioactive waste intended for ultimate disposal at a licensed low-level radioactive waste land disposal facility shall prepare a manifest reflecting information requested on the following applicable forms, or their equivalent:

(a) NRC Form 540, Uniform Low-Level Radioactive Waste Manifest, Shipping Paper;

(b) NRC Form 541, Uniform Low-Level Radioactive Waste Manifest, Container and Waste Description; and

(c) If necessary, NRC Form 542, Uniform Low-Level Radioactive Waste Manifest, Manifest Index and Regional Compact Tabulation.

(2) NRC Forms 540 and 540A shall be completed and shall physically accompany the pertinent low-level waste shipment.

(3) Upon agreement between shipper and consignee, NRC Forms 541, 541A, 542 and 542A may be completed, transmitted, and stored in electronic media with the capability for producing legible, accurate, and complete records on the respective forms.

(4) A licensee shall not be required by the cabinet to comply with the manifesting requirements of this section, if they ship:

(a) LLW for processing and expect its return for storage as prescribed by their license, prior to disposal at a licensed land disposal facility;

(b) LLW that is being returned to the licensee who is the waste generator or generator, as defined in 902 KAR 100:010; or

(c) Contaminated radioactive material to a waste processor that becomes the processor's residual waste.

(5) For guidance in completing a form, refer to instructions that accompany the form.

(6) A copy of a manifest required by this section may be legible carbon copies, photocopies, or computer printouts that reproduce the data in the format of the uniform manifest.

(7) Information on hazardous, medical, or other waste, required to meet U.S. Environmental Protection Agency regulations, for example, 40 CFR Parts 259 and 261, is not addressed in this section, and shall be provided on the required EPA form. The required EPA form shall accompany the Uniform Low-Level Radioactive Waste Manifest required by this section.

(8) The shipper of the radioactive waste, shall provide the following information on the uniform manifest:

(a) The name, facility address, and telephone number of the licensee shipping the waste;

(b) An explicit declaration indicating whether the shipper shall be acting as a waste generator, collector, processor, or a combination of these identifiers for purposes of the manifested shipment; and

(c) The name, address, and telephone number, or the name and U.S. Environmental Protection Agency hazardous identification number, for the carrier transporting the waste.

(d) The shipper of the radioactive waste shall provide, on the uniform manifest, the following information:

1. The date of the waste shipment;

2. The total number of packages or disposal containers;

3. The total disposal volume and disposal weight in the shipment;

4. The total radionuclide activity in the shipment;

5. The activity of each of the radionuclides, hydrogen-3, carbon-14, technetium-99, and iodine-129 contained in the shipment;

6. The total masses of uranium-233, uranium-235, and plutonium in special nuclear material; and

7. The total mass of uranium and thorium in source material.

(9) The shipper of the radioactive waste shall provide the following information on the uniform manifest regarding the waste and disposal container of waste in the shipment:

(a) An alphabetic or numeric identification that uniquely identifies each disposal container in the shipment;

(b) A physical description of the disposal container, including the manufacturer and model of a high integrity container;

(c) The volume displaced by the disposal container;

(d) The gross weight of the disposal container, including the waste;

(e) For waste consigned to a disposal facility, the maximum radiation level at the surface of each disposal container;

(f) A physical and chemical description of the waste;

(g) The total weight percentage of a chelating agent for waste containing more than one-tenth (0.1) percent of a chelating agent by weight, and the identity of the principal chelating agent;

(h) The approximate volume of waste within a container;

(i) The sorbing or solidification media, if present, and the identity of the solidification media vendor and brand name;

(j) 1. The identity and activity of a radionuclide contained in each container;

2. The masses of uranium-233, uranium-235, and plutonium in special nuclear material;

3. The masses of uranium and thorium in source material; and

4. The identity and activity of each radionuclide associated with, or contained in, discrete waste types within a disposal container, such as:

a. Activated materials;

b. Contaminated equipment;

- c. Mechanical filters, sealed sources or devices; and
 - d. Wastes in solidification or stabilization media;
 - (k) The total radioactivity within each container;
 - (l) The classification of the waste in accordance with Section 6 of this administrative regulation, for wastes consigned to a disposal facility; and
 - (m) Identification of waste not meeting the structural stability requirements of Section 7(2) of this administrative regulation.
- (10) The shipper of the radioactive waste shall provide the following information on the uniform manifest regarding a waste shipment delivered without a disposal container:
- (a) The approximate volume and weight of the waste;
 - (b) A physical and chemical description of the waste;
 - (c) The total weight percentage of a chelating agent if the chelating agent exceeds one-tenth (0.1) percent by weight, and the identity of the principal chelating agent;
 - (d) The classification of the waste in accordance with Section 6 of this administrative regulation for waste consigned to a disposal facility;
 - (e) Identification of waste not meeting the structural stability requirements of Section 7(2) of this administrative regulation;
 - (f) 1. The identity and activity of a radionuclide contained in the waste;
 - 2. The masses of uranium-233, uranium-235, and plutonium in special nuclear material;
 - 3. The masses of uranium and thorium in source material; and
 - (g) For a waste consigned to a disposal facility, the maximum radiation level at the surface of the waste.
- (11)(a) The origin of the LLW resulting from activities of a processor may be attributable to one (1) or more generators, including a waste generator. The requirements in this subsection apply to:
- 1. A disposal container enclosing a mixture of waste originating from different generators; and
 - 2. A mixture of waste shipped in a form without a disposal container, for which portions of the mixture within the shipment originate from different generators.
- (b) For a homogeneous mixture of a waste, such as incinerator ash, provide the:
 - 1. Waste description applicable to the mixture; and
 - 2. Volume of the waste attributed to each generator;
 - (c) For a heterogeneous mixture of a waste such as:
 - 1. The combined products from a large compactor, identify each generator contributing waste to the disposal container; and
 - 2. A discrete waste type, for example, activated materials, contaminated equipment, mechanical filters, sealed sources or devices, and wastes in solidification or stabilization media, the identity and activity of individual radionuclides contained on the waste type within the disposal container;
 - (d) For a generator, the following information shall be provided:
 - 1. The volume of waste within the disposal container;
 - 2. A physical and chemical description of the waste, including, if present, the solidification agent;
 - 3. The total weight percentage of a chelating agent for a disposal container containing more than one-tenth (0.1) percent of a chelating agent by weight, plus the identity of the principal chelating agent;
 - 4. The sorbing or solidification media, if present, and the identity of the solidification media vendor and brand name if the media is claimed to meet stability requirements in Section 7(2) of this administrative regulation; and
 - 5.a. Radionuclide identity and activity contained in the waste;
 - b. The mass of uranium-233, uranium-235, and plutonium in special nuclear material; and
 - c. The mass of uranium and thorium in source material if contained in the waste.
- (12)(a) An authorized representative of the waste generator, processor, or collector shall certify, by signing and dating the shipment manifest, that the transported materials are:
- 1. Properly classified;
 - 2. Described;
 - 3. Packaged;
 - 4. Marked;
 - 5. Labeled; and
 - 6. In proper condition for transportation according to the applicable administrative regulations of the Department of Transportation and 902 KAR 100:070; and
- (b) A collector in signing the certification shall certify that nothing has been done to the collected waste which would invalidate the waste generator's certification.
- (13) A licensee who transfers waste to a licensed waste processor for waste treatment or repackaging shall comply with the requirements of paragraphs (d) through (l) of this subsection. A licensee who transfers waste to a land disposal facility or a licensed waste collector shall:
- (a) Prepare waste to meet a classification in Section 6 of this administrative regulation and the waste characteristics requirements in Section 7 of this administrative regulation;
 - (b) Label each disposal container, or transport container if potential radiation hazards preclude labeling of the individual disposal container, of waste to identify if the waste is Class A, Class B, Class C, or greater than Class C waste, in accordance with Section 6 of this administrative regulation;
 - (c) Conduct a quality assurance program including, management evaluation of audits to assure compliance with Sections 6 and 7 of this administrative regulation.
 - (d) Prepare the NRC Uniform Low-Level Radioactive Waste Manifest as required by this subsection;
 - (e) Forward a copy or electronically transfer the Uniform Low-Level Radioactive Waste Manifest to the intended consignee so that:
 - 1. Receipt of the manifest precedes the LLW shipment;
 - 2. The manifest and the waste are delivered to the consignee at the same time; or
 - 3. Both methods of manifest delivery described in subparagraphs 1 and 2 of this paragraph are used.
 - (f) Include NRC Form 540 and Form 540A, if required, with the shipment, regardless of the option chosen in paragraph (e) of this subsection;
 - (g) Receive acknowledgment of the receipt of the shipment in the form of a signed copy of NRC Form 540;

(h) Retain a copy of or electronically store the Uniform Low-Level Radioactive Waste Manifest and documentation of acknowledgment of receipt as the record of transfer of licensed material as required by 902 KAR 100:040; and

(i) For a shipment, or parts of a shipment, for which acknowledgment of receipt has not been received within the times established in this section, conduct an investigation in accordance with subsection (17) of this section.

(14) A waste collector licensee who handles only prepackaged waste shall:

(a) Acknowledge receipt of the waste from the generator within one (1) week of receipt by returning a signed copy of NRC Form 540;

(b) Prepare a new manifest to reflect consolidated shipments that meet the requirements of this section, including identification of the generator of each container of waste in the shipment;

(c) Forward a copy or electronically transfer the Uniform Low-Level Radioactive Waste Manifest to the intended consignee so that either:

1. Receipt of the manifest precedes the LLW shipment; or

2. The manifest and the waste are delivered to the consignee at the same time; or

3. Both methods of manifest delivery described in subparagraphs 1 and 2 of this paragraph are used.

(d) Include NRC Form 540 and Form 540A, if required, with the shipment regardless of the option chosen in paragraph (c) of this subsection;

(e) Receive acknowledgement of the receipt of the shipment in the form of a signed copy of NRC Form 540;

(f) Retain a copy of or electronically store the Uniform Low-Level Radioactive Waste Manifest and documentation of acknowledgment of receipt as the record of transfer of licensed material as required by 902 KAR 100:040;

(g) For a shipment, or parts of a shipment, for which acknowledgment of receipt is not received within the time established in this section, conduct an investigation in accordance with subsection (17) of this section;

(h) Notify the shipper and the cabinet if a shipment, or part of a shipment, has not arrived within sixty (60) days after receipt of an advance manifest, unless notified by the shipper that the shipment has been cancelled.

(15) A licensed waste processor who treats or repackages waste shall:

(a) Acknowledge receipt of the waste from the shipper within one (1) week of receipt by returning a signed copy of the manifest or equivalent documentation;

(b) Prepare a new manifest that meets the requirements of this subsection:

1. Preparation of the new manifest shall reflect that the processor shall be responsible for meeting these requirements; and

2. For each container of waste in the shipment, the manifest shall identify the waste generators, the preprocessed waste volume, and other information required by subsection (11) of this section;

(c) Prepare waste to meet a classification in Section 6 of this administrative regulation and the waste characteristics requirement in Section 7 of this administrative regulation;

(d) Label each package of waste to identify the waste as Class A, Class B, or Class C, in accordance with Sections 6 and 8 of this administrative regulation;

(e) Conduct a quality control program to assure compliance with Sections 6 and 7 of this administrative regulation, including management evaluation of audits;

(f) Forward a copy or electronically transfer the Uniform Low-Level Radioactive Waste Manifest to the intended consignee so that:

1. Receipt of the manifest precedes the LLW shipment;

2. The manifest and the waste are delivered to the consignee at the same time; or

3. Both methods of manifest delivery described in subparagraphs 1 and 2 of this paragraph are used.

(g) Include NRC Form 540 and 540A, if required with the shipment regardless of the option chosen in subsection (15)(f) of this section;

(h) Retain a copy of or electronically store the Uniform Low-Level Radioactive Waste Manifest and documentation of acknowledgment of receipt as the record of transfer of licensed material required by 902 KAR 100:040;

(i) Receive acknowledgment of the receipt of the shipment in the form of a signed copy of NRC Form 540;

(j) For a shipment or part of a shipment for which acknowledgment of receipt is not received within the time established in this section, conduct an investigation in accordance with subsection (17) of this section; and

(k) Notify the shipper and the cabinet when a shipment, or part of a shipment, has not arrived within sixty (60) days after receipt of an advance manifest, unless notified by the shipper that the shipment has been cancelled.

(16) The land disposal facility operator shall:

(a) Acknowledge receipt of the waste within one (1) week of receipt by returning a signed copy of the manifest or equivalent documentation to the licensee that last possessed the waste and transferred the waste to the operator. If the returned copy of the manifest or equivalent documentation indicates discrepancies between materials on the manifest and materials received, copies or electronic transfer of the affected forms shall be returned indicating the discrepancy;

(b) Maintain copies of completed manifests, or equivalent documentation, and electronically store the information required by 10 CFR 61.80(l) until the cabinet terminates the license; and

(c) Notify the shipper, generator, collector, or processor and the cabinet if a shipment, or part of a shipment, has not arrived within sixty (60) days after the advance manifest was received, unless notified by the shipper that the shipment has been cancelled.

(17)(a) The shipper shall investigate a shipment or part of a shipment for which acknowledgment is not received within the time established in this section, if the shipper has not received notification of receipt within twenty (20) days after transfer.

(b) The investigation shall include tracing the shipment and filing a report with the cabinet.

(c) A licensee who conducts a trace investigation shall file a written report with the cabinet within two (2) weeks of completion of the investigation.

Section 11. Records. (1) A licensee shall maintain a record in the same units used in this administrative regulation.

(2) A record of disposal of licensed material required by this administrative regulation shall be maintained until the cabinet authorizes disposition, or in accordance with 902 KAR 100:073, Section 28.

(3) A licensee shall maintain a record of the disposal of licensed materials required by 902 KAR 100:022 and Sections 2, 3, 4, and 5 of this administrative regulation, and disposal by burial in soil, including burials authorized before January 28, 1981.

(4) A licensee shall retain the records required in subsection (3) of this section until the cabinet terminates each pertinent license requiring the record.

Section 12. Annual Report of Waste Generated. (1) A licensee issued a specific license, pursuant to 902 KAR 100:040, shall file an annual report with the cabinet containing information regarding low-level radioactive waste associated with activities authorized by the license. The report shall be filed if the licensee was, or was not, a waste generator during the reporting period.

(2) The report shall contain information regarding the waste for a period of one (1) calendar year and shall be filed no later than January 15 of the following year.

(3) The report shall be filed on a Low-Level Radioactive Waste (LLW) Report Form provided by the cabinet and shall contain types and amounts of generated waste and estimates of future wastes to be generated.

Section 13. Incorporation by Reference. (1) The following material is incorporated by reference:

(a) "NRC Form 540, Uniform Low-Level Radioactive Waste Manifest, Shipping Paper, 7/2001";

(b) "NRC Form 540A, Uniform Low-Level Radioactive Waste Manifest, 3/1995";

(c) "NRC Form 541, Uniform Low-Level Radioactive Waste Manifest, Container and Waste Description, 7/2001";

(d) "NRC Form 541A, Uniform Low-Level Radioactive Waste Manifest, 3/1995";

(e) "NRC Form 542, Uniform Low-Level Radioactive Waste Manifest, Manifest Index and Regional Compact Tabulation, 7/2001";

(f) "NRC Form 542A, Uniform Low-Level Radioactive Waste Manifest, Manifest Index and Regional Compact Tabulation, 3/1995"; and

(g) "Low-Level Radioactive Waste (LLW) Report, 2001".

(2) This material may be inspected, copied, or obtained, subject to applicable copyright law, at the Department for Public Health, Office of the Commissioner, 275 East Main Street, Frankfort, Kentucky 40621, Monday through Friday, 8 a.m. to 4:30 p.m. (12 Ky.R. 1123; eff. 1-3-86; Am. 16 Ky.R. 2538; eff. 6-27-90; 20 Ky.R. 2380; 2867; eff. 5-18-94; 28 Ky.R. 1940; 2210; eff. 3-28-2002.)

SPECS original quote and drawing



Appendix 3. Invention disclosure

UNIVERSITY OF KENTUCKY
INTELLECTUAL PROPERTY DISCLOSURE FORM

A. Descriptive title of the intellectual property. (Must Be Typed)

Purification of metals contaminated with radioactive materials by vacuum distillation.

B. Describe the intellectual property. If needed, attach additional sheets. Please include examples, drawings or other data supporting your intellectual property. If the intellectual property is described in a manuscript that is being prepared for publication, attach a copy, and indicate when it will appear in print.

The Department of Energy has a number of facilities with metal equipment that was used to process radioactive materials. The metal equipment is stored as is or, in some cases, has been smelted into scrap ingots. These materials might be recycled to make materials for construction of new nuclear power plants or defense applications if their radioactive components could be removed economically. Common approaches include electrorefining and electrowinning, which are used for purification and separation of spent reactor fuels. In the case of some metal/contaminant pairs, these methods achieve the very low contamination levels needed to meet U.S. or international standards for use as scrap or recycle materials.

The invention proposed here uses the differences between the metal and radioactive contaminant vapor pressures to separate the mixtures. For this method to work, the radioactive contaminant should have either a higher or a lower vapor pressure than the bulk metal. In the first case, the radioactive contaminant would be removed via the vapor stream, leaving a purified metal. In the second case, the purified metal would be removed via the vapor stream and the radioactive contaminant would concentrate in the remaining bulk metal.

In embodiments of this art, any processing concepts useful for separating materials based on their vapor pressure can be used, such as continuous flash systems, flash systems with sweep gases, continuous distillation systems (operating above atmospheric, at atmospheric, and below atmospheric pressure), reactive distillation systems, and batch distillation systems (operating above atmospheric, at atmospheric, and below atmospheric pressure, or with sweep gases). The processing equipment can include metal process equipment modified for this purpose, including induction vacuum furnaces, reverberatory furnaces, and smelting furnaces.

The scrap metals that can be purified in this manner include steel, stainless steel, nickel, aluminum, copper, Monel (a copper-nickel alloy), cobalt, gold and silver. The radioactive contaminants that can be removed include cerium 137, neptunium 237, plutonium 239 and 240, potassium 40, strontium 90, technetium 99, and uranium 234, 235 and 238.

C. Describe the *prospective commercial use* of the intellectual property and your best assessment of what companies or firms might be interested in the technology.

Between 1952 and 1986, the Paducah Gaseous Diffusion Plant operated smelters to produce scrap metal from unneeded processing equipment. About 17 million pounds of non-radiologically contaminated nickel were smelted, and an additional 20 million pounds of radioactively contaminated nickel was smelted. One of the objectives of the DOE award listed in Section D is to identify methods for removing Technetium 99, the primary radioactive contaminant, from the nickel.

The research supporting this intellectual property was focused on the separation of technetium 99 from nickel. The general method is expected to be applicable to many other contaminated metals.

Nickel has a market value near \$14/lb. The Paducah community would support industries to recycle or reprocess scrap materials from the plant as a way to generate jobs in the community.

- D. If this intellectual property was *extramurally supported*, provide the name and type of agency and *contract number(s)*.

This intellectual property was developed under DOE award DE-FG05-030R23032, "Kentucky Research Consortium for Energy and the Environment", led by Dr. Lindell Ormsbee. This research project addresses energy and environmental issues associated with the Paducah Gaseous Diffusion Plant, Paducah, KY.

Identify and attach any *contract-related progress reports*.

A number of quarterly reports on the progress of this research have been provided to the Kentucky Research Consortium.

If the intellectual property was made with industrial sponsorship, please identify the *sponsoring company* and attach copies of all agreements executed with the company.

While there was no industrial sponsorship of this research, the PI will be discussing furnace modifications and designs with commercial furnace suppliers. These discussions could lead to equipment especially tailored for this process.

- E. If information or samples (e.g., cell lines) relating to this intellectual property have been provided to anyone *outside* the University, please provide all details and dates.

A non-disclosure agreement will be completed prior to discussion with furnace equipment companies.

- F. When was the intellectual property conceived? Attach annotated copies of any written records that substantiate the conception date. Such records can include notebook entries, letters, reports, etc.

The intellectual property was conceived as a response to the KY Research Consortium award. The original proposal is attached.

- G. When did any *experimental work* relating to the intellectual property first occur? Attach copies of substantiating notebook entries. Have you retained representative samples or products from the early experiment work? _____ Where are the notebooks and representative samples or products located now?

The key experimental work is yet to be done, as the equipment needed to be designed from scratch (mass spectroscopy attached to a Knudsen cell). The critical experiments are to verify the vapor pressure of the radioactive contaminant (⁹⁹Tc) over nickel liquid near the expected operating temperatures for the separation.

H. Please provide as many details and exact copies as possible for:

Any information about this intellectual property that has been revealed in *grant proposals* or *research agreements*.

Any presentations about this intellectual property, abstracts or manuscripts submitted for publication?

Two KY Research Consortium presentations are provided: one demonstrating the processing concept and the other showing an initial process design with cost estimates.

I. Summarize further experimental work now underway or contemplated in the near future relating to this intellectual property.

The key experimental tests will be initiated soon. Discussions with equipment suppliers will be started in order to better determine costs and capabilities currently available. A detailed process design package will be completed as the equipment and utility costs are better defined.

J. State the nature and extent of any *literature search* made to date, and attach copies of the closest references, and *closest prior art* your search has provided.

A literature search was included in the original proposal.

K. Indicate if *University resources* (i.e.; facilities; equipment; faculty; staff; or student time) were used in the development of this intellectual property.

YES

NO

x

☐

L. Full Name of Primary Investigator , _____

Furnish the following information for any investigator or collaborator contributing to this intellectual property, include information for yourself:

Full Name: Eric A. Grulke	Chemical & Materials Engineering
Position or Title Professor	
Work Address: 359 RGAnderson Bldg College of Engineering University of Kentucky Lexington, KY 40506	2050 Delong Road Lexington, KY 40515-9506

Telephone Numbers:859-257-6097,	Home 859-271-2843
Other (Fax, Pager, Voice Mail, etc.):	
E-mail address: <u>egrulke@engr.uky.edu</u>	
Citizenship:USA	
Signature & Date:	

***Please Note Number of Additional Pages or Attachments and forward this information to: University of Kentucky,
Intellectual Property Development Office, A144 ASTeCC, Lexington, KY 40506-0286.
Telephone No.: (859) 257-2300, ext. 230.***

FOR IPDO OFFICE USE ONLY	
CASE NO: _____	DATE RECEIVED: _____
DATE REVIEWED IPDO COMMITTEE: _____	UK INTEREST: YES _____ NO _____
NUMBER OF INVENTORS: _____	ASSIGNED TO FIRM: _____
RELEASED TO: _____	DATE RELEASED: _____ Date Entered _____

(Duplicate this page if you have additional investigators.)

Page No.:

(Continued:) Additional Investigator(s) or Collaborator(s) on this Intellectual Property

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E-mail address:	
Citizenship:	
Signature & Date:	

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Full Name: Louei El-Azzami	Department
Position or Title: Ph.D.	
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E-mail address:	
Citizenship:	
Signature & Date:	

Please forward this information to:
University of Kentucky, Intellectual Property Development Office
A144 ASTeCC, Lexington, KY 40506-0286.
Telephone No.: (859) 257-2300, ext. 230

IPDOFORM.doc

Appendix 4. NASA Glen Knudsen Cell Design

There were a few Knudsen/mass spectrometer instruments in the United States at the beginning of this project. The instrument at the NASA Glen research center in Cleveland, Ohio was being used for research, and the researchers agreed to show us their work. The research team had been developing Knudsen cells coupled to mass spectrometers for about 10 years. Most of their emphasis has been on the Knudsen cell, and they had less expertise on mass spectroscopy.

Dr. Nathan S. Jacobson of NASA Glen hosted our visit on 13 December, 2004. They use their high temperature Knudsen cell/mass spectrometer (HT KCMS) to study titanium-aluminum alloys and the interactions of boron nitride with water. They do their own cell designs, and have fabricated over three different systems of varying complexity.

There were two companies offering commercial instruments that would be customized for our project, but these systems started at \$300,000. The University of Kentucky had an expert in mass spectrometry, Dr. Bert Lynn, who has designed and constructed a number of systems. We chose to use his expertise for the mass spectrometer portion and buy the Knudsen cell from SPEC, a German instrument firm with an office in the US.

The following photographs show the NASA Glen HT KCMS at the time of the visit.

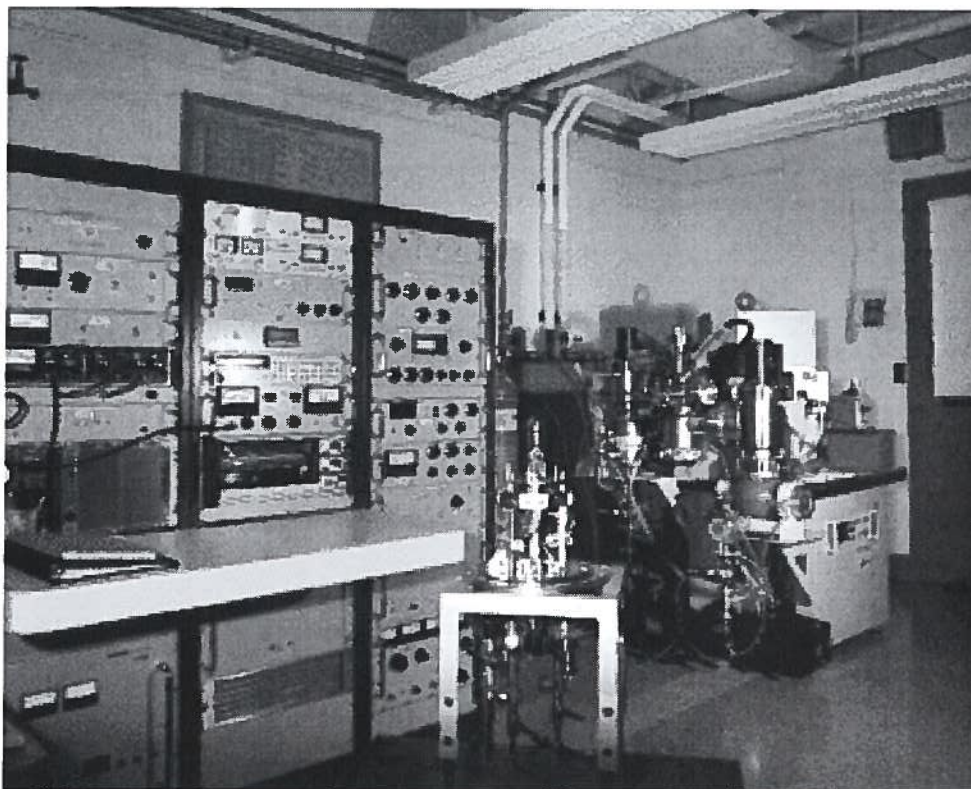


Figure A4-1. NASA Glen HT KCMS.

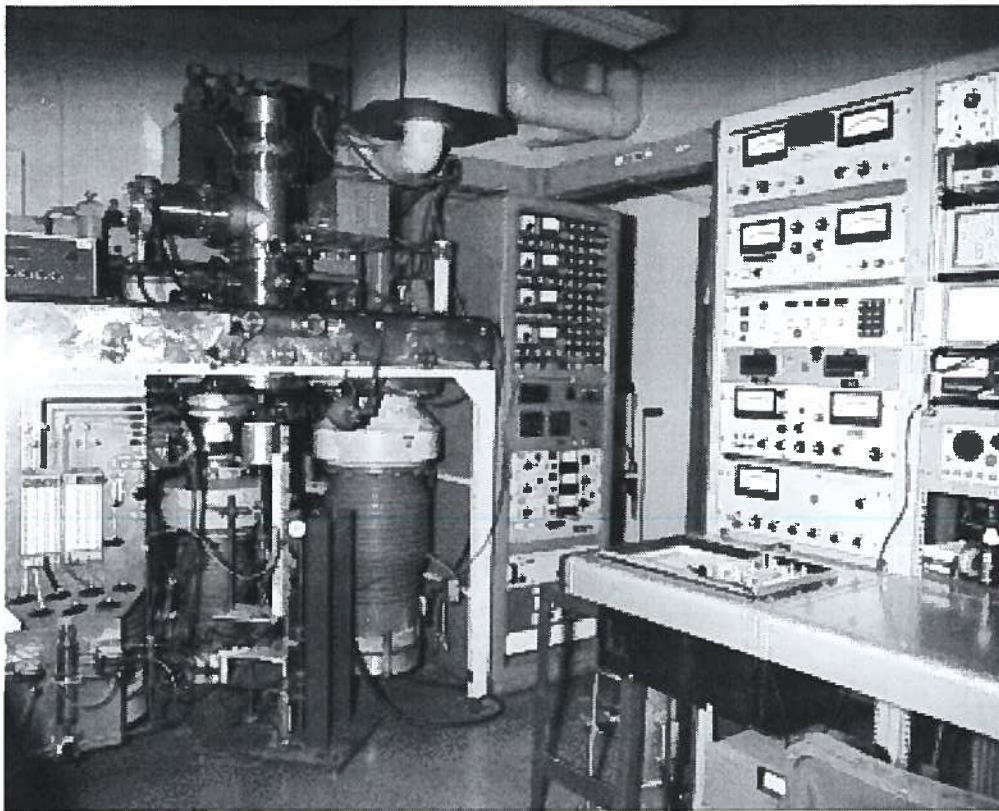
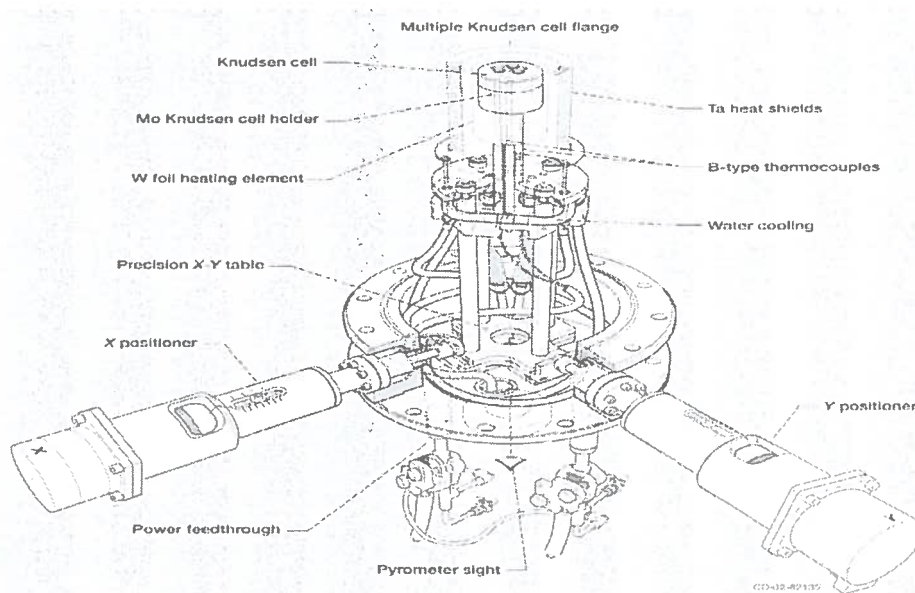


Figure A.4-2. NASA Glenn molecular beam sampling MS.

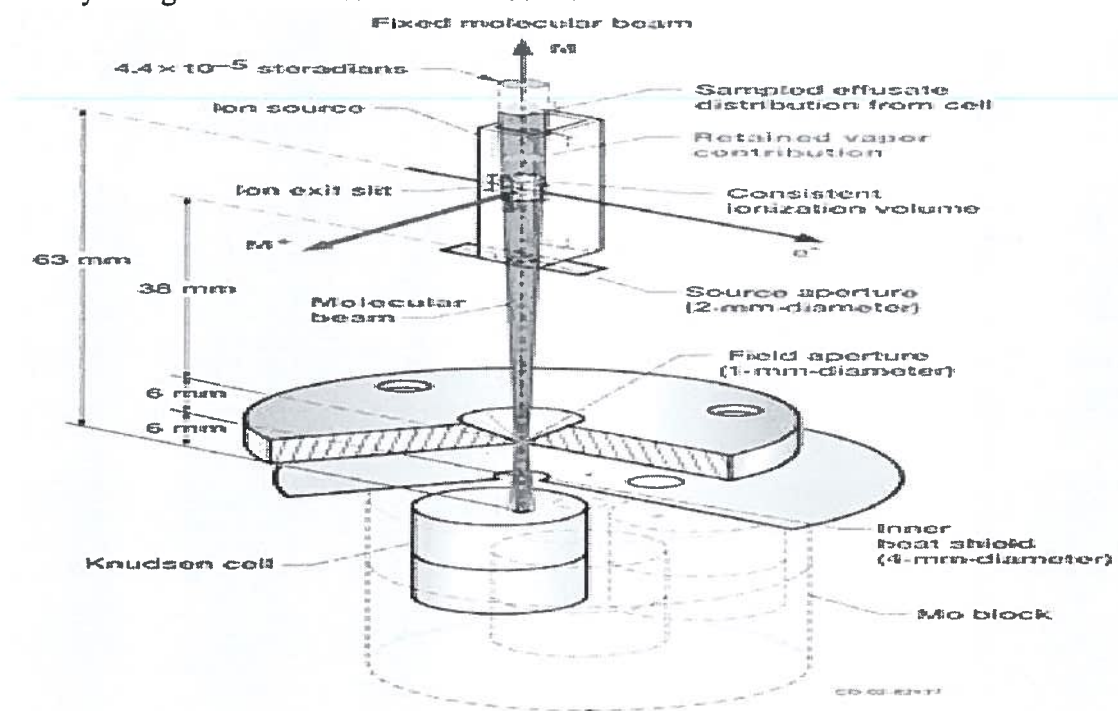
Both systems have extensive instrumentation for control purposes. The multiple Knudsen cell diagram is shown in the next figure. Key design issues include the foil heating elements, the heat shields, the precision x-y positioner to adjust the instrument for thermal expansion, and water cooling systems.



The multiple Knudsen cell has the following attributes:

- The vapor effusate from all cells can be sampled at constant temperature with time and cell position.
- The resistance furnace has the following attributes:
 - Radial symmetry in a molybdenum block at the center
 - Cylindrical tungsten sheet-heating element ($t=25\text{mm}$)
 - Seven-layer tantalum heat-shield pack.
- Temperature stability was achieved by separating the water-cooling circuit from the electrical power circuit.
- Temperature measurement: B-type thermocouples & filament optical pyrometer.

The system generates a fixed molecular beam.



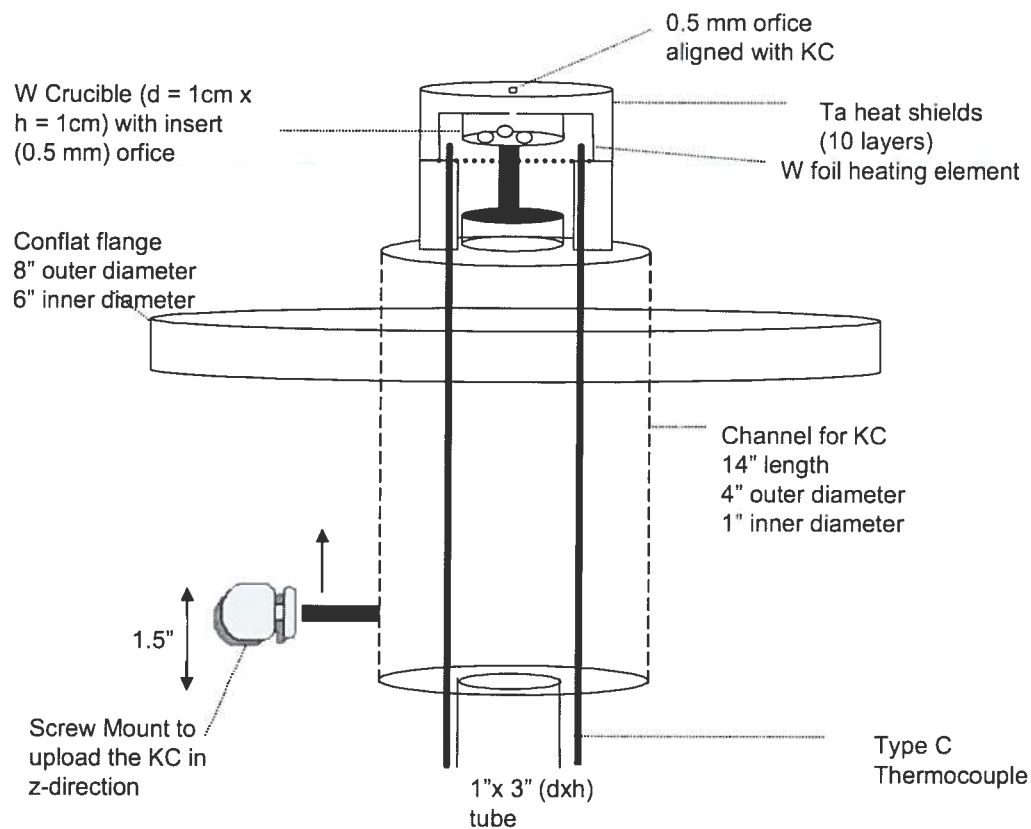
The vapor outlet from the Knudsen cell is filtered through an aperture protected by a heat shield. The vapor stream is ionized by an ion source, and, once ionized, the metal ions will be driven through the mass spectrometer by charge. The challenge in the design is alignment, and the vacuum system.

The molecular beam has the following attributes:

- Field and source apertures were added between the effusion cell and the ion source.
- Fixed field and source apertures → fixed molecular beam.
- Alignment of cells with the fixed apertures → The effusate is sampled.
- Maintains a constant ionization volume, while an accurate positioning mechanism (automated X-Y table) ensures that the same portion of the effusate distribution is sampled from all cells .

- Reference cell- The relative difference in effusate distribution, due to variation in orifice shape (compare ion intensities).
- The factors affecting the measured ion intensity of species effusing from a cell are limited to the composition and temperature of the condensed sample.

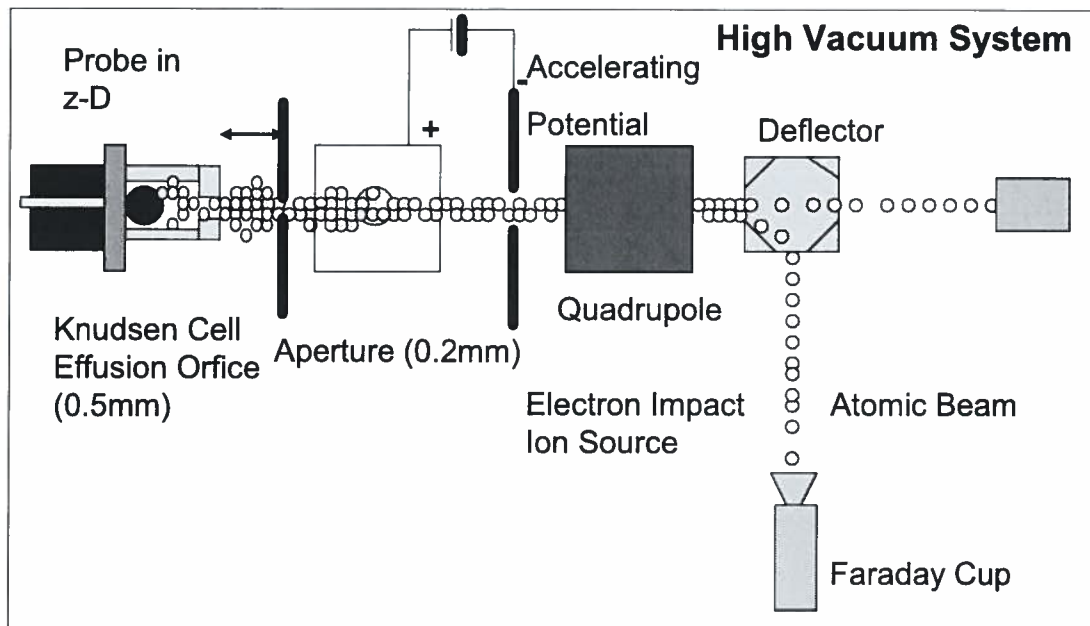
The NASA Glenn team recommended a single Knudsen cell positioned in the z-direction for our application. A conceptual sketch is shown below.



This design has the following attributes:

- The cell will be sitting on a probe that will enter through the channel.
- Dimensions are fixed except in z-direction.
- The distance and the alignment between the orifice and the MS is most critical- Avoid divergence of beam.
- Water cooling and thermocouples integrated in KC.

A sketch of the HT KCMS integrated system is shown in the following sketch:

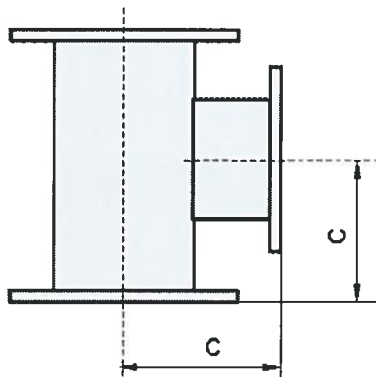
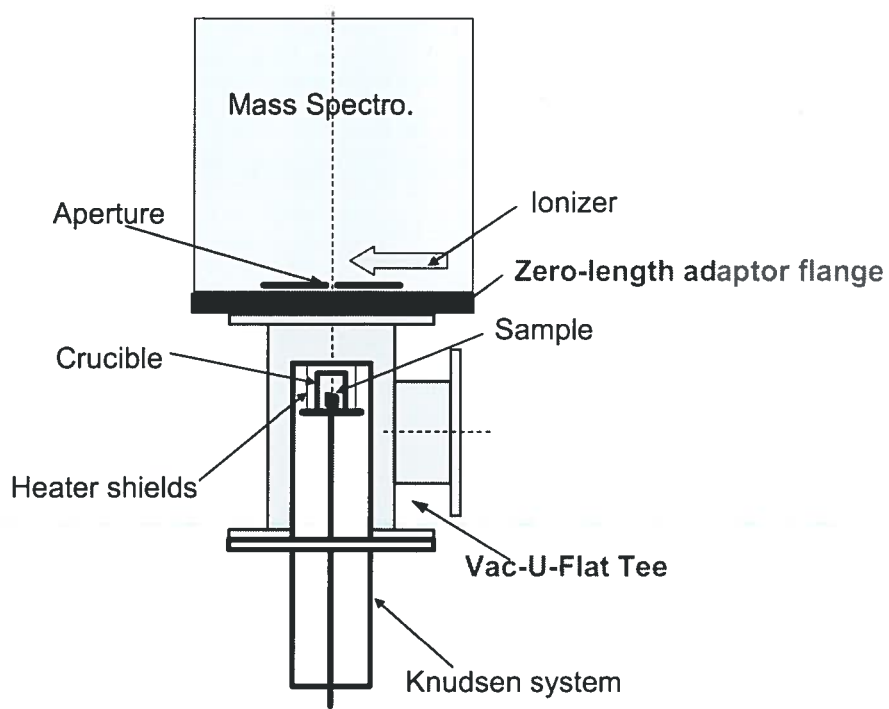


This set up has the following attributes:

- The ability of moving KC in z-direction- optimize the distance between the MS & KC → optimum signal
- MS- measures levels of 1ppb Tc
- The sensitivity of MS- depends on the accurate alignment of beam (KC – orifice - aperture - MS)

Design work with SPEC Instruments began in January, 2006, but was not completed until March, 2006.

The key issue was the flange requirements to join the two instruments (16 Nov. 2005 sketch sent to SPEC Instruments).



- Vac-U-Flat Tee:
 - Tube OD: 2.5"
 - Flange OD: 4.5"
 - C: 4.12"



- Zero-length Adapter Flange:
 - Lg Flg OD: 8.00"
 - Sm Flg OD: 4.5"
 - ID: 2.50"
 - T: 0.87"

Requirement: this design requires that Knudsen cell system has diameter less than 2.5"

Appendix 5. Metal Distillation and Vaporization

Metal distillation and vaporization has been utilized as a means to purify metals from contaminants. The efficiency of metal distillation depends on the difference of the boiling points and the vapor pressures of the metallic components of the molten mixture. Ginder, et. al., purified zinc (Zn) from cadmium (Cd) by metal distillation (13, 14, and 15). The influent Zn-Cd mixture contained 0.47% Cd (B.P = 767°C) and the remaining Zn (B.P = 905°C). The vapor pressure of Cd is higher than that of Zn. Distilling the Zn-Cd mixture at 975°C, Ginder was able to purify Zn and reduce the content of Cd to 0.0013%.

U.S. Patent 5,582,630 demonstrates how to purify magnesium (Mg) from metallic and nonmetallic elements by vacuum distillation (16). The final Mg product contained 5.87 ppm Zn and 0.73 ppm of other metals (25 ppb Al, <10 ppb As, <5 ppb B, <10 ppb Bi, <20 ppb Ca, 18 ppb Cd, <10 ppb Co, <10 ppb Cr, <20 ppb Cu, <10 ppb Fe, <10 ppb Ga, <10 ppb In, <10 ppb K, < 5 ppb Li, 41 ppb Mn, <10 ppb Mo, 36 ppb Na, <10 ppb Ni, 36 ppb Pb, 22 ppb Sb, 226 ppb Si, < 10 ppb Sn, < 1 ppb Th, 10 ppb Ti, <1 ppb U, <30 ppb V, and 23 ppb Zr).

Tayama and Hodozuka used vacuum distillation to purify indium (In) from silicon (Si), iron (Fe), Ni, Cu, gallium (Ga), antimony (Sb), and lead (Pb) (1). The In feed had the following contaminants: 0.14 ppm Si, 0.15 ppm Fe, 2.3 ppm Ni, 0.28 ppm Cu, 0.03 ppm Ga, 0.02 ppm Sb, and 0.2 ppm Pb. The purified In had the following impurities: <0.03 ppm Si, <0.01 ppm Fe, <0.01 ppm Ni, <0.01 ppm Cu, <0.01 ppm Ga, <0.01 ppm Sb, and 0.01 ppm Pb.

Weil distilled a mixture of metals to recover magnesium. The mixture was composed of 85.654% Mg, 10.7% Al, 0.67% Cu, 0.006% Fe, 0.24% Mn, 0.01% Ni, 0.16% Si, and 2.56% Zn. The distillate was composed of >97.805% Mg, <0.01% Al, <0.01% Cu, <0.003% Fe, <0.01% Mn, 0.002% Ni, 0.02% Si, and 2.86% Zn. The residue was composed of 90.16% Al, 0.3% Mg, 5.9% Cu, 0.04% Fe, 2.1% Mn, 0.08% Ni, 1.4% Si, and 0.02% Zn (18).

Historically, metal distillation and vaporization has not been applied to separate radiocontaminated metals from the radioactive contaminants.

Appendix 6. Prior & Present Arts of Electrorefining & Electrowinning

Electro-refining using aqueous acid electrolytes is known to be effective for the removal of actinides from contaminated Ni; in such a technique the Ni is deposited selectively on a cathode, with the actinide ions remaining in solution due to their lower electrochemical reduction potential. Conventional electro-refining is however ineffective for reducing Tc concentrations in Ni; Tc is found to co-deposit with Ni at the cathode in a ratio that is the same as, or higher than, that in which it is found in the electrolyte. To reduce the concentrations of Tc in Ni ingots, new techniques were implemented. These techniques are listed below.

1) A technique in which solvent extraction is combined with electrorefining is described in Snyder et. al. U.S. Pat. No. 5,156,722. Solvent extraction is used to separate heptavalent Tc from the electrolyte in which radio-contaminated Ni is dissolved, followed by electrowinning to recover Ni.

2) The process described in Snyder et. al. U.S. Pat. No. 5,183,541 and U.S. Pat. No. 5,156,722 employs an electro-refining cell that utilizes a semi-permeable membrane. Tc is chemically precipitated in the anodic compartment, using a variety of agents to reduce it to its tetravalent state, and is removed by filtration. A hydrochloric acid-based electrolyte is used because it is more amenable than sulfuric acid to the chemical precipitation of Tc.

3) U.S. Pat. No. 5,217,585, also to Snyder et. al., describes an electrorefining process in which the Tc-containing Ni is again electrolytically dissolved in an acid electrolyte. The electrolyte is contacted with activated carbon to absorb pertechnetate ions, after which the solution is filtered and transferred to an electrowinning cell, where the Ni is recovered at the cathode. The contaminated carbon is subsequently incinerated to produce Tc-containing ash, which can be encapsulated for disposal.

4) In U.S. Pat. No. 5,262,019, Snyder et al address the contaminated ash problem by providing an electro-refining process with separate electrolytic dissolution and electrowinning steps. Contaminated nickel is first electrolytically dissolved in a sulfuric acid electrolyte, followed by treatment of the filtered nickel-laden electrolyte with an ion exchange resin to remove pertechnetate and other ions; the treated electrolyte is then processed in an electrowinning cell to deposit purified Ni at the cathode.

5) In U.S. Pat. No. 5,439,562, Snyder et al developed a novel method for decontaminating radio-contaminated nickel comprising, in an electrorefining cell having a semi-permeable membrane, cathodically depositing Ni from an alkaline solution containing electrolyte, Ni ions, and radioactive ions. Preferably, the electrolyte solution is ammonium sulfate maintained at a pH of least about 10.

6) In U.S. Pat. No. 5,458,745, Hardil et al employs a three-step process to separate Tc from radio-contaminated metal. The contaminated metal is dissolved in an acid solution; the Tc, present in the resultant solution as pertechnetate ions, is quantitatively reduced to its metallic state through a metal displacement (cementation) reaction with a base metal of lower reduction potential; and the desired metal is electrolytically recovered from the solution.

All these techniques were not capable of meeting the release criteria for radioactive materials.



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FACSIMILE: 270-112-4689

Edwin A. Jones
ejones@bsgpad.com

September 2, 2020

VIA MAIL AND EMAIL:

Mr. Robert Edwards
US Department of Energy
5501 Hobbs Road
Kevil, KY 42053

robert.edwards@pppo.gov

Mr. Greg Wiles
Director of Paducah Pacro
64 Bent Creek Drive
Benton, KY 42025

gwiles.pacro@gmail.com

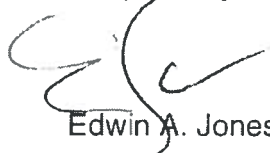
RE: Nickel

Dear Gentlemen:

Enclosed please find a Wall Street Journal article about nickel.

Please contact me with your questions.

Respectfully,



Edwin A. Jones

EAJ/krq

Enclosure: Wall Street Journal Article about nickel

100 W. MARKET STREET, SUITE 2300
LOUISVILLE, KENTUCKY 40202-3351
TELEPHONE: 502-584-9980
FACSIMILE: 502-584-9100

141 WEST SECOND STREET
LEXINGTON, KENTUCKY 40502-1010
TELEPHONE: 859-252-6711
FACSIMILE: 859-253-1115

137 MAIN STREET, SUITE 200
PIKEVILLE, KENTUCKY 41502
TELEPHONE: 606-432-9670
FACSIMILE: 606-432-9680

ELSBY EAST, SUITE 204, 100 PEARL STREET
NEW ALBANY, INDIANA 47150
TELEPHONE: 812-918-5053
FACSIMILE: 812-918-9233

Nickel 000058

BUSINESS

One of the Brains Behind Tesla May Have a New Way to Make Electric Cars Cheaper

What's the secret to building a more affordable electric vehicle? JB Straubel has an answer. It starts with a pile of old cellphones.

By *Tim Higgins* / Photographs by *Max Whittaker* for *The Wall Street Journal*

Updated Aug. 29, 2020 11:39 am ET



Listen to this article
15 Minutes

Almost every day old iPhones and other used personal electronics arrive by the truckload at a warehouse in Carson City, Nev., where workers crack them open, pull out their batteries and strip them for raw materials.

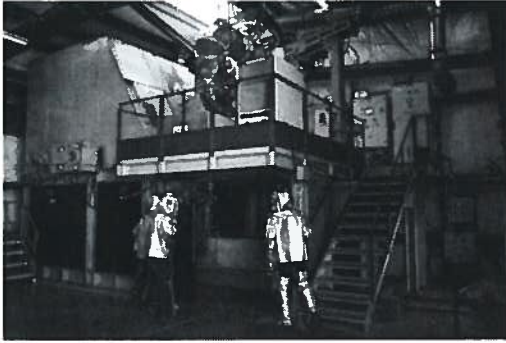
To JB Straubel, one of the brains behind Tesla Inc., [TSLA -1.13%](#) ▼ that refuse holds the key to driving the electric car revolution forward—and making the vehicles affordable enough for everyone to own one.

Mr. Straubel, Tesla's longtime chief technology officer, pioneered the lithium-ion battery powertrain design that helped propel the Silicon Valley company to what is now the highest valuation in the car industry. Since leaving Tesla about a year ago, he has been trying to solve a problem created by that success: Where to find all the nickel, cobalt and lithium needed to make the batteries that power Tesla's cars and their growing list of rivals.

Extracting those materials from nature, through mining and other processes, is costly and difficult, and production is lagging far behind expected demand. Mr. Straubel's company, Redwood Materials, is taking a different tack, quietly aiming to build the biggest car battery-recycling operation in the U.S. The 44-year-old is betting that he can perfect a fast and efficient way of collecting and repurposing those materials to disrupt the centuries-old mining industry.



Boxes of electronics wait to be recycled by Redwood Materials in Carson City, Nev.



Melting down batteries for recycling is difficult and sometimes hazardous work.

"Forever the entire market has been dictated by the commodity price of these metals," Mr. Straubel said in his first in-depth interview about his new venture since it was formed in 2017 while still at Tesla. "This is a chance to change that whole equation and to realize material cost savings in a way that short circuits that industry."

He and Tesla Chief Executive Elon Musk share an obsession with electric vehicles but in other aspects are mirror opposites—Mr. Musk a swaggering showman, Mr. Straubel a behind-the-scenes engineer whose former employees tell stories of him swapping out lightbulbs at hotels that he found inefficient. Mr. Straubel has been interested in chemistry and batteries since childhood in Wisconsin where a lab accident left a scar down his left cheek and a story to tell when he went on to earn degrees at Stanford University. At school, he gained a reputation in the burgeoning electric car crowd, for converting an old Porsche into an electric car and drag racing it for fun.

Now he is engaged in difficult and sometimes hazardous work on a grand scale. The ovens involved in the recycling run at temperatures of 2,700 degrees Fahrenheit to reduce the materials to brightly colored powders. Lithium-ion cells are prone to catching fire if not properly handled, and the packs housing them often weigh thousands of pounds and come in different sizes and configurations. It isn't clear yet what kind of market there will be for the recycled car batteries and who the competition will be as an assortment of longtime recyclers, mining companies and startups are eyeing the market. Few are willing to make huge investments yet required for the machinery and tools needed for such work.



The ovens at Redwood Materials are used to extract lithium oxide.

It is work that is essential, Mr. Straubel says, if the industry is going to continue to increase production of electric cars at the pace companies are planning. Regulatory pressures to lower emissions and falling battery prices have led almost every major car manufacturer to include electric vehicles in their product lineup. That is expected to drive a surge in global demand for lithium-ion batteries in the next five years to almost 800 gigawatt hours from 177 gwh last year, or about 22 times the amount of cells produced at Tesla's giant factory outside of Reno in 2019, according to Simon Moores, managing director of researcher Benchmark Mineral Intelligence.

The cost of batteries has long been the biggest obstacle to making electric cars affordable for the masses. As a result, electric vehicles still carry a hefty price premium compared with gas engine cars. McKinsey & Co. estimates that premium at \$12,000 on average. Hyundai Motor Co., one of the few to offer the same vehicle in an all-electric and gas version, charges \$17,000 more for the plug-in Kona sport-utility vehicle.

Tesla has made great strides in reducing battery costs and is expected to detail further advances during its Battery Day event on Sept. 22. In its early days the biggest cost of the batteries lay in the complex processes to assemble them. As those processes have been perfected, Mr. Straubel says 50% to 75% of the cost of a battery for the industry now lies with its raw materials—where he sees potential for recycling to lower costs.



Chemical engineer Fredy Bridges filters metal salts at the Redwood Materials facility in Carson City.



What lithium looks like after it's been extracted.

At the same time, the supply of used batteries is exploding. Half-a-million electric vehicles are expected to be scrapped in 2025, according to environmental engineer Maria Kelleher, who specializes in recycling and renewable energy. The figure should jump to more than one million vehicles in 2030, she projects.

Mr. Straubel already has won over some big name investors. In his first fundraising round this year, he raised around \$40 million from investors led by Capricorn Investment Group and Breakthrough Energy Ventures, an environmental investment fund that includes Amazon.com Inc. founder Jeff Bezos and Microsoft Corp. co-founder Bill Gates. Dipender Saluja, a managing director at Capricorn, said what Mr. Straubel is proposing represents a shift in thinking. "It's about rebuilding what I just finished using exactly the same material," he said.

Tesla and Mr. Musk aren't part of this venture, though Mr. Straubel remains on friendly terms with his former employer. Instead, Mr. Straubel aims to work with the entire automotive industry, developing recycling processes that work for any battery and car design.

Mr. Straubel first became enamored of lithium-ion batteries for cars around 2003. That year he hung around a Los Angeles area car shop that experimented with the idea of

stringing together cells to power a car dubbed the Tzero. Mr. Straubel, then 27, wanted to create his own car with 10,000 cells that he estimated capable of crossing the U.S. in a single charge and sought money from Mr. Musk, who was sitting on a fortune from his share of PayPal and investing in a rocket startup called Space Exploration Technologies Corp.

SHARE YOUR THOUGHTS

What would it take for you to purchase an electric car? Join the conversation below.

During a 2003 lunch to talk about an unmanned, hydrogen-powered airplane, Mr. Straubel raised his other passion, noting his car project and the work at the shop called AC Propulsion.

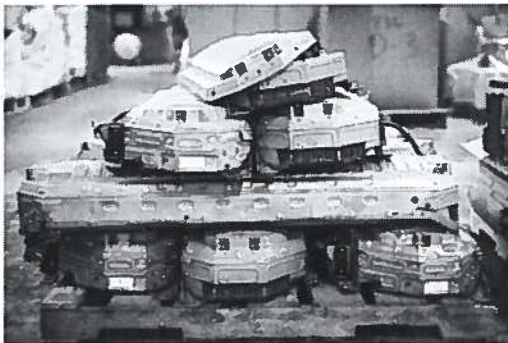
Mr. Musk wanted an electric sports car of his own but the shop wasn't interested in converting one for him. He turned instead to a tiny startup in Menlo Park called Tesla Motors that had just got off the ground in hopes of making its own sports car to be dubbed the Roadster and was looking for investors.

Through a string of events Mr. Musk became Tesla's largest investor and the public face of Tesla, turning the startup into a household name in part through his showmanship and swagger. Mr. Straubel was hired as an early employee where his contribution was so great that Mr. Musk considers him a co-founder of Tesla.

The partnership has made Mr. Straubel a rich man. The small stake that he held in Tesla when he departed last year would be worth more than \$600 million today if he didn't sell any shares, according to FactSet data. His time at Tesla also introduced him to his future wife, Boryana, whom he married in 2013. She's a self-described nerd who shares her husband's affinity for data. They have a home outside of Carson City and in Silicon Valley.

Despite having helped create some of the quickest cars in the world, his true love is batteries and not cars. He is also a pilot who spends most of his time in the air in a German-made Stemme S10 glider, which, he said, is "one of the few airplanes much more efficient than a car."

Mr. Straubel is well versed in the frustrating history of the electric car. Auto makers before had experimented with expensive batteries that were heavy and held comparatively little charge, so electric vehicles were pricey and impractical, and sales failed to take off.



Toyota Prius batteries wait to be recycled inside Redwood Materials's Carson City, Nev., warehouse.

Mr. Straubel and Mr. Musk changed that when they began delivering Tesla's Roadster sports car in 2008. It featured almost 7,000 battery cells tightly packed together in a box in the rear of the vehicle. The typical laptop computer battery has just a handful of such cells. The design delivered a car that could go more than 200 miles on a single charge, and go from zero to 60 miles-per-hour in 3.9 seconds.

The Roadster and the Model S luxury sedan that followed in 2012 still appealed mainly to

a niche audience of wealthy enthusiasts. By 2013, Mr. Musk set his sights on turning Tesla into a mass-market car maker. He and Mr. Straubel plotted the first mega battery factory to produce the many billions of cells they would need in coming years for the Model 3 compact car, the company's biggest bet electric vehicles could go mainstream.

The two planned big. The so-called Gigafactory that Tesla set out to build with Japanese partner Panasonic Corp. was designed to initially produce about 35 gigawatt hours of cells annually at the plant in Sparks, Nev., for 500,000 vehicles, or roughly what the entire battery industry combined was making in 2013.

The electric car industry has exploded since then. Tesla built about 35,000 vehicles in 2014. The company, before the coronavirus pandemic, had planned to sell around half a million this year, and rivals such as General Motors Co., Nissan Motor Co., and South Korea's Hyundai add to the total as the industry goes global.

To meet that demand, a building boom is occurring around the world to copy Tesla's Gigafactory model. China is building a mega-battery factory every week while in the U.S. one is opening every four months, Mr. Moores said.

But production is constrained by the lack of raw materials. Commodity prices for such key ingredients as lithium and cobalt have taken a roller-coaster ride in recent years amid excitement and skepticism for electric cars. Ingredients, such as cobalt, often are sourced in politically fraught places leaving suppliers eager to find dependable sources. Cobalt comes from mines around the world, including the Democratic Republic of the Congo.

Mr. Musk went so far on a recent earnings call as to put out a public plea. "Please mine more nickel," he said. "Tesla will give you a giant contract for a long period of time if you mine nickel efficiently and in an environmentally sensitive way."

Mr. Straubel saw the crunch coming for years. When visiting a nickel mine in Canada a few years ago as Tesla's chief technology officer, he surveyed the massive operations. Its scale and complexity illustrated to him that simply opening more mines wasn't really an option. Why not recycle the cells already out there now or in the near future, he thought.

Mr. Straubel also knew one of the electric-car industry's dirty secrets: For all its aspirations of environmental benefits, the industry is wasteful. He saw first hand how wasteful the process could be when overseeing development of Tesla's Reno Gigafactory. When the car maker in 2018 struggled to increase production of the Model 3 car, one of the pinch points was the battery factory, where a former employee has alleged that the company was wasting as much as \$200 million in scrapped material. Tesla has said the amount was overly stated.

Mr. Straubel won't say how much waste his old factory generated, but said it reinforced his idea that a market for recycling those costly and difficult to dispose of materials would exist.

"We need to really appreciate that we need to build a Gigafactory in reverse," he said.

Like at Tesla, Mr. Straubel has grand ambitions but is ready to start small as he takes on incumbents, in this case centuries old mining companies. Redwood is honing its processes by working on batteries from consumer electronics such as cellphones, which are smaller and easier to handle compared with the large packs that come from cars.



Cell phones are smaller and easier for Redwood Materials to handle than large battery packs from cars.

While the number of electric cars hitting end of life is comparably small, the more lucrative market for Mr. Straubel is recycling the scrapped battery materials from the cell making process for electric cars.

Factories making cells will scrap an average of about 10% of those batteries, according to Benchmark Mineral's Mr. Moores. In 2025, that could mean about 80 gigawatt hours of cells will be trashed, or the equivalent to the size of the entire battery market in 2016, he projects.

Within that scrap, he said, lie 64,000 tons of lithium or the equivalent of what more than two mines might produce in a year with a market value of \$500 million to \$1.5 billion depending upon shifting market prices. The waste also includes other precious components such as cobalt, nickel and other materials, representing billions of dollars of potential value in total.

"Those that crack this technology to turn it into a battery quality material will have a huge business," Mr. Moores said.

Mr. Straubel says that within 10 years, he hopes his recycling will bring the price of raw materials down to about half compared with mines. That, he said, could help make electric vehicles—from trucks to trains—ubiquitous.

Mr. Straubel's vision is already winning converts, including Panasonic, his old partner for the Gigafactory. The company late last year began a trial with Redwood to reclaim more than 400 pounds of the scrap it generates in making battery cells and now has upped that to 2 tons. All of the scrap coming from its side of the Nevada battery facility is now shipped to Redwood.

"His process looks like it's way more sustainable from an environmental perspective," said Celina Mikolajczak, a vice president of Panasonic Energy of North America and a former Tesla battery expert who advised on the original battery issues for the Roadster. "He doesn't have to landfill anything and if you look at the typical process at this point a lot of the less valuable materials get landfilled."

The materials that are recycled are being used to produce new products. Panasonic, she said, is working with Mr. Straubel to see if his recycled materials can be refined well enough to be reused in its batteries, noting that old cellphone batteries could be a good source of cobalt for new ones.

Mr. Straubel envisions a process so efficient that batteries coming from the mountain of electric cars being retired in coming years could be quickly stripped down, recycled for their core materials, and used to rebuild new power cells, creating a closed loop where hardly any materials are lost.



Redwood Materials chemical engineer Tunkie Saunders monitors the furnace used to melt down recycled batteries.

The outside money is intended to accelerate research, build up the operation and grow the workforce to as many as 200 staff by year's end from about 50 this summer. He recently hired one of his former top deputies from his Tesla days, Kevin Kassekert, who helped oversee the construction of Tesla's giant battery factory in Sparks.

Like Tesla, Redwood has global aspirations. Mr. Straubel already has plans to scale up and build new facilities around other battery factories. "I'm looking into the future and seeing this freight train coming at us."

Write to Tim Higgins at Tim.Higgins@WSJ.com

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Hyperdrive

Elon Musk Is Going to Have a Hard Time Finding Clean Nickel

By [Mark Burton](#), [Libby Cherry](#), and [David Stringer](#)

August 21, 2020, 9:00 PM PDT

► Industry accidents show challenges in supplying 'green nickel'

► Demand for nickel to soar as more electric cars hit the road



A polluted river following a diesel spill outside Norilsk, Russia, on June 6. Photographer: Irina Yarinskaya/AFP via Getty Images

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Elon Musk promises a "giant contract" with the miner that can supply nickel for [Tesla Inc.](#) batteries at low cost with minimal environmental impact, yet the industry's messy track record may make that deal difficult to clinch.

Recent accidents such as a [diesel spill](#) in Arctic Russia and a burst waste pipeline in Papua New Guinea suggest the industry will struggle to meet Musk's [request](#) for a large quantity of the metal produced in an "efficient" and "environmentally sensitive" way.

As the world's most-valuable carmaker extends manufacturing arms to [China](#) and [Germany](#), its billionaire owner may have to rely increasingly on the biggest supplier of nickel: Indonesia. Yet miners there are being criticized for plans to pump waste into the open sea, meaning Musk and other carmakers may need to compromise on sourcing

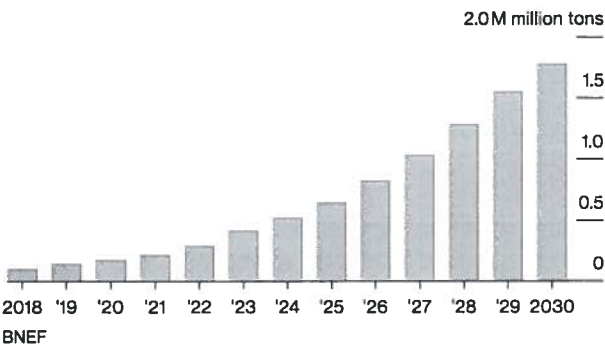
standards while trying to compel the industry to clean up its act.

"Nickel projects being built in Southeast Asia will rely on coal, fuel oil or diesel to run their operations and will leave a very large carbon footprint," said Sam Riggall, chief executive officer of Clean TeQ Holdings Ltd., which is developing an Australian mine to supply nickel for vehicle batteries. "This sort of makes a bit of a mockery of driving a green, sustainable car."

EVs will comprise 58% of global passenger car sales in 2040, compared with 10% by 2025, according to BloombergNEF. Nickel helps cram more energy into cheaper and smaller battery packs, allowing EVs to charge faster and travel farther between plug-ins.

Power Boost

Batteries fuel demand for nickel over the next decade



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Indonesia holds about a quarter of all nickel reserves. To meet demand from carmakers, companies there are investing in projects that will use acid to process low-grade nickel ore and produce high-quality battery chemicals. The miners plan to dilute the byproducts and pipe them out to sea – a process known as deep-sea tailings disposal.

A major spill last year arising from a ruptured pipeline at the Ramu nickel mine in Papua New Guinea highlighted the potential hazards of the process.

"The disposed tailings will have a drastic, non-reversible impact on the ecosystems, marine life and humankind," Alex Mojon, president of the Swiss Association for Quality and Environmental Management, said in an Aug. 11 report on the accident.

Metallurgical Corp. of China, which runs the Ramu nickel mine, declined to comment.

In the same way EV makers sought to reduce their exposure to cobalt from the Democratic Republic of Congo because of human-rights concerns, they also may decide to halt purchases of nickel from Indonesian mines using this combination of high-pressure acid leaching, or HPAL, and deep-sea tailings disposal, the consultancy Benchmark Minerals said.

"For new nickel supply, Elon and the battery industry look to HPAL in Indonesia," Simon Moores, founder and managing director of London-based Benchmark Minerals, said in an email. "Yet deep water disposal methods are increasingly putting these mines on the same black list as illegal artisanal cobalt from the DRC."

Tesla didn't respond to a request for comment. Chinese battery maker GEM Co., which is jointly developing a HPAL project in Indonesia with steelmaker Tsingshan Holding Group Co., declined to comment. Tsingshan didn't immediately respond to a request for comment.

The scrutiny isn't just concentrated on Indonesia. A massive diesel spill at MMC Norilsk Nickel PJSC's operations in Arctic Russia sparked public outrage recently and left the Moscow-based company on the hook for what could be the largest environmental fine in the country's history.

Norilsk Nickel also is one of the world's largest emitters of sulfur dioxide, a cause of acid rain. Its \$3.7 billion project to capture the toxic gas won't start working until at least 2023.

In the meantime, the company expects the spotlight to get brighter.

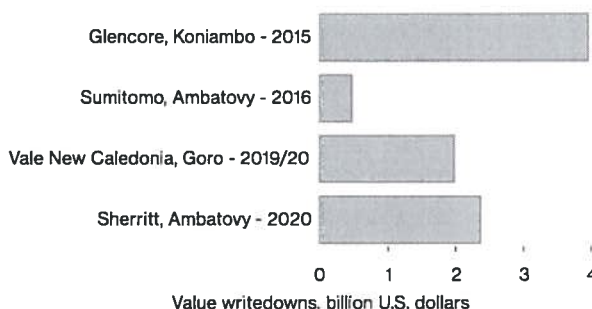
"There is a tendency to trace the origin of the metal, and in the future the pressure will grow," Vladimir Potanin, Norilsk Nickel's CEO and co-owner, said in an interview.

After two deadly waste-dam failures inside four years at Vale SA's Brazilian iron ore mines, the company is using the same infrastructure at its nickel operations in New Caledonia. After heavy writedowns, Vale is in talks to sell the mine to New Century Resources, which pledges to invest in an alternative waste-storage system.

Compounding the challenges, many of the mines brought online in recent years face financial and technical problems. Analysts including Jim Lennon, senior commodities consultant at Macquarie Securities, say it's doubtful major companies will sign off on new nickel projects while prices are low and the threat of further impairments on existing assets looms large. Sumitomo Corp. last month wrote \$500 million off the value of its Ambatovy mine in Madagascar.

Lost Nickels

Mining companies swallow billion-dollar writedowns



Several junior miners such as Giga Metals Corp. and Canada Nickel Co. are talking up their green credentials, and their share prices surged as a result. But it's unlikely they can produce enough clean nickel for EV makers, putting pressure on the mining titans to deliver.

"The industry is focused on these issues because the shareholders, the institutional investors are very much focused on it," Lennon said. "They're starting to rapidly wake up."

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Electric Car Batteries Are Going to Need a Lot of Nickel

There is a real environmental cost to this.

By Lloyd Alter

Published September 1, 2020 01:51PM EDT



Mining siltation in the Philippines.
Jacob Maentz/ Getty Images



Sustainability for All.

component in batteries; Tesla buys nickel-cobalt-manganese (NCM) from LG in South Korea and nickel-cobalt-aluminum (NCA) from Panasonic.

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Only 5% of the world's nickel goes into batteries now; the rest goes into making stainless steel. But this is going to change as more companies start producing electric cars and pickups. According to Zach Shahan of CleanTechnica, Ford's F150 electric pickup will use NCM batteries that are 90% nickel.

Advertisement

twice the nickel and all the
batteries.

the other stuff in



A March 2012 photo of an open cut nickel laterite mine near Kendari, Southeast Sulawesi province, Indonesia. Photography by Mangiwau / Getty Images

The problem is that unfortunately for Elon, nickel is not usually mined in an environmentally sensitive way. Henry Sanderson writes in the Financial Times that demand for nickel could rise sixfold by 2030 and that nickel mining can get messy.

"Analysts predict that Indonesia will account for almost all of the growth in nickel supplies over the next decade, overwhelming output from new mines in Canada and Australia. But a number of Chinese-backed projects in the country plan to dump mine waste containing metals such as iron into the sea, in an area renowned for its unique coral reefs and turtles. 'It could undermine the entire proposition of trying to sell a consumer a product that is environmentally friendly, if you have this back story,' said Steven Brown, a Jakarta-based consultant and former employee at nickel miner Vale."

The rock contains only about one percent nickel, so it generates a lot of waste, and when it is dumped in the ocean, it spreads in suspension over a large area, including the beaches on other islands.

problematic metals like cobalt. Elon Musk is having a "battery day" in September where he will probably announce yet another technological breakthrough. According to Reuters, Tesla is also working on the recovery of all of these elements through recycling, "as well as new 'second life' applications of electric vehicle batteries in grid storage systems"

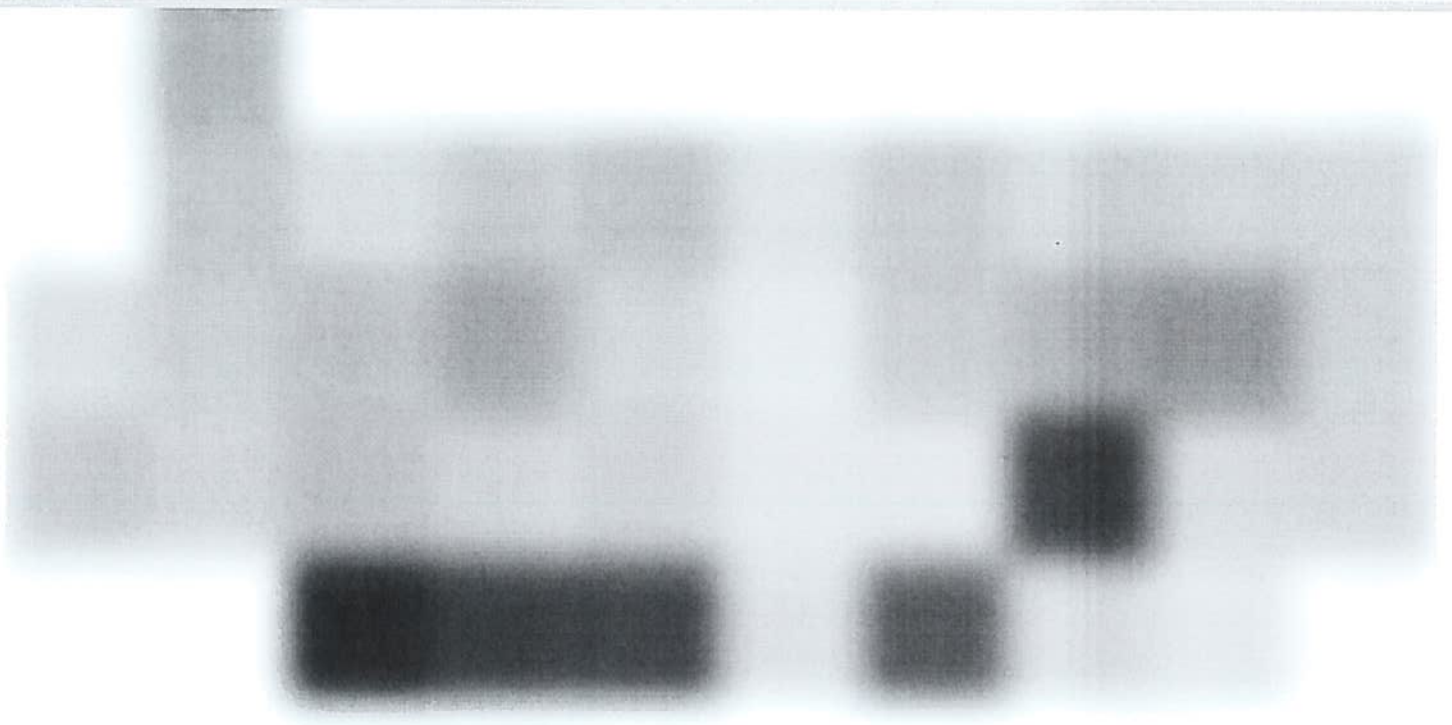
But we keep coming up against the huge numbers being thrown around for electric vehicle production. Zach Shahan asks "How many electric vehicles (EVs) could Ford produce if demand was sky high? It appears that 300,000 by 2023 is the limit according to current plans (across all electric models), but that is not 100% certain."

switch to electric vehicles as quickly as possible. But they pointed out that replacing the world's projected fleet of 2 billion vehicles is going to require an explosive increase in mining: global annual extraction of neodymium and dysprosium will go up by another 70%, annual extraction of copper will more than double, and cobalt will need to increase by a factor of almost four – all for the entire period between now and 2050. We need to switch to electric cars, yes; but ultimately we need to radically reduce the number of cars we use."

It's probably not going to be as bad as Hickel suggests; they are already making cobalt-free batteries, and their energy density will continue to rise. But there is also the need for all the electricity to charge them, more turbines and solar panels and batteries, all requiring more mining. But don't worry, it won't likely be in your backyard. Hickel writes:

"It's important to keep in mind that most of the key materials for the energy transition are located in the global South. Parts of Latin America, Africa and Asia are likely to become the target of a new scramble for resources, and some countries may become victims of new forms of colonisation. It happened in the sixteenth, seventeenth and eighteenth centuries with the hunt for gold and silver from South America."

None of this even mentions the embodied carbon of the plain old steel and aluminum that the car bodies are made of. Much will be recycled from the internal-combustion-engine powered cars that come off the roads, but we are still talking vast numbers.



Big Nickel Postcard. old postcard

Many Canadians will remember what Sudbury was like once. "As mining, stripping, sintering, and smelting operations increased with world demand for metals, Sudbury's landscape began to look like a barren moonscape. The mining and processing of sulfide minerals released sulfur that contaminated and acidified soils." When American astronauts trained there in the early 70s, it was said it was because it resembled the moon. (They were there because it was a mineral-rich meteor crater, but we can't let that get in the way of a good story.) It took 30 years, billions of dollars in abatement technology, and two million trees to restore it.

Now all this mining is happening far away, and I doubt they are being so careful about clean production or restoration. Elon Musk wants his nickel mining to be environmentally sensitive, but he also wants his nickel efficient and cheap.

It becomes very hard to consider electric cars "clean" when you add up what goes into them, especially when we are getting all these monster Rivians and F150s and Hummers that are twice as big as they need to be.