

Direct Reduction of Waste through Refining of DOE Metal Assets - 13632

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Chemical Vapour Metal Refining (CVMR®)

ABSTRACT

CVMR® presents a technology for refining nickel from the enrichment barrier materials of the DOE that is proven through 100 years of use by the metals industry. CVMR® applies modern controls, instrumentation for process and monitoring of the system, and innovative production methods to produce a wide spectrum of products that generate new technology applications and improvements to our society and economy. CVMR® will receive barrier materials as a secure operation and size reduce the metal to a shred that is fed to a carbonylation reactor where nickel is reacted with carbon monoxide and generate nickel carbonyl. The carbonyl will be filtered and decomposed with heat to form a variety of products that include high value nano powders, coated substrates, net shapes and pure nickel. The residue from the reactor will retain radionuclides from enrichment activities. The carbon monoxide will only react and extract nickel under the operating conditions to leave volumetric contamination in the unreacted residue. A demonstration plant was designed and built by CVMR® and operated by BWXT, to demonstrate the systems capabilities to DOE in 2006. A pilot plant operation precedes the detailed design of the nickel refinery and provides essential data for design, safe work practices, waste characterizations and system kinetics and confirms the project feasibility. CVMR® produces nickel products that are cleaner than the nickel in U.S. commerce and used by industry today. The CVMR® process and systems for nickel refining is well suited for DOE materials and will provide value through environmental stewardship, recovery of high value assets, and support of the DOE environmental remediation programs as the refined nickel generates additional long term benefits to local communities.

INTRODUCTION

Gaseous Diffusion Plant (GDP) systems of the DOE contain radiologically contaminated materials including large quantities of nickel. These systems are being dismantled in D&D activities that will lead to environmental remediation of the facilities and disposition of the nickel. The nickel metal is volumetrically contaminated with enrichment materials including the major isotopes of uranium, trace actinides, and technetium. Recovery of the nickel as a scrap resource is restricted by this contamination and controlled as part of a moratorium issued by then DOE Secretary Bill Richardson that blocked the release into commerce of volumetrically contaminated nickel from DOE facilities. The moratorium was intended to allow time for the evaluation of alternatives by DOE and for the Nuclear Regulatory Commission (NRC) to make a decision on national treatment standards.

Currently, disposition of such metal has encountered additional obstacles, primarily because of the lack of a consistent disposition policy, systematic regulatory provisions, and, above all, public understanding.

Chemical Vapour Metal Refining (CVMR®) presents a technology for refining GDP nickel materials that are proven through 100 years of use in the metals industry. CVMR® provides a mature and established, state-of-the-art technology proven to refine nickel to some of the highest levels of metal purity ever measured.

CVMR® has applied modern controls, instrumentation for process and monitoring of the system, and

innovative production methods to provide a safe, reliable system that produces a wide spectrum of product forms for new technology applications and improvements in our society and for our economy.

CVMR[®] proposes to receive DOE materials as a secure operation and size reduce the metal to a particle that is fed to a carbonylation reactor. There nickel is reacted with carbon monoxide and generate nickel carbonyl. The carbonyl is filtered and decomposed to generate forms with unique electrical, wear, and shielding properties and performance characteristics into a variety of products that include high value nano powders, coated substrates, net shapes, and pure nickel ingots and billets.

CVMR[®]

CVMR[®] provides Chemical Vapor Deposition (CVD) processes for refining nickel and other metal assets. CVMR[®] technologies and systems are recognized around the world as innovative, safe, advanced, and successful processes that are proven for this application by a global metals industry. CVMR[®] systems are uniquely qualified to safely refine DOE metals including nickel from the GDPs. CVMR[®] systems provide enhanced system efficiencies, reliability of operations, reduced energy requirements, minimal environmental impact, and very attractive production costs. The CVMR[®] systems also provide radically improved safe operations with state-of-the-art monitoring and environmental controls with no air emissions and no water discharge.

CVMR[®] has extensive experience providing commercial CVD systems to extract and plate nickel using a modified Mond process. This process is an established commercial process developed by Ludwig Mond in 1899. The process is currently used in the nickel industry for refining nickel ores, making nickel powder, and nickel plating of complex shapes such as molds and fixtures. The CVMR[®] CVD process is well known for its patented process control and monitoring technology that creates the safe, consistent, continuous, high-quality processing that has made CVMR[®] a leader in CVD technology applications. CVMR[®] has used the process to provide ultra-pure nickel for the Sudbury Neutrino Observatory (SNO) project, as shown in **Figure 1**.



Figure 1. For the SNO project, CVMR[®] used a precision, ultra-pure nickel tube for SNO project and the final assembly of the detector for detection and characterization of neutrinos.

The purity of CVMR[®]'s products has been tested at the Los Alamos National Laboratory, University of

Washington, Centre d'Etudes Nucléaires de Bordeaux Gradignan, Teledyne Brown Engineering, and other nationally recognized laboratories and all results have indicated the absence of radiological emissions and extremely high nickel purity levels (**Table I**).

Table I. CVMR® produced nickel has been analyzed by laboratories renowned for their ability to detect radionuclides and radiological emissions. In every analysis radioactivity was absent in all refined metals.

Laboratory	Analytical Results for CVMR® Produced Nickel	Comments
University of Washington	< 60 disintegrations/kg/Day	..no detectable foreign material. ..other materials 1 million fold higher
Los Alamos National Laboratory	< 60 disintegrations/kg/Day	..no detectable foreign material. ..other materials 1 million fold higher
Teledyne Brown Engineering	<0.05 ppm Total U	..commercial grade nickel compared with 5.8 ppm Total U
B&W, LTC	<0.01 ppm Total U	All other isotopes below Minimum Detectable Analysis (MDA)
General Engineering, Labs	<0.01 ppm Total U	All other isotopes below Minimum Detectable Analysis (MDA)
Environmental Inc-Midwest	<0.01 ppm Total U	All other isotopes below Minimum Detectable Analysis (MDA)
Centre d'Etudes Nucléaires de Bordeaux Gradignan	Th-232+228<0.4 mBq/Kg Ra-226+228 <0.5 mBq/Kg Total U <5.0 mBq/Kg	..purest materials ever observed. ..no detection of Thorium or Uranium chain radioactivity ..total cosmological radiation 5.35 mBq/Kg [Co-56+27+28 & Mn-54 (for 70 day)]

The CVD Refining Process

The CVD process used by CVMR® is referred to as the carbonyl process, because the compound selectively generated during the process is a carbonyl compound of the metal, such as nickel carbonyl (Ni(CO)₄).

In 1995, CVMR® was invited to evaluate the application of their proven, innovative technologies for separation of radionuclides. Pure nickel products with the lowest possible radiation signature were needed for the Sudbury Neutrino Observatory (SNO) project in Sudbury, Ontario, Canada. Additional specialty applications of the CVD process demonstrated a complete partitioning of naturally occurring isotopes and low-level background radiation. Analysis by DOE laboratories, universities, and international institutions,

with the most sensitive techniques for detecting radiation, found no evidence of radioactive emissions in the refined products. This characteristic was critical for the SNO project in establishing high sensitivity to the passage and presence of neutrinos and the optimal application of the scientific foundations of the SNO facility.

Bench Scale Demonstration

In 2003, CVMR[®], through CVMR[®]-USA, worked with B&W (Babcock & Wilcox Technical Services Group, Inc. was formerly known as BWX Technologies, Inc. and will be referred to in this document as B&W) in Lynchburg, Virginia to provide a pilot facility to demonstrate the performance of the CVMR[®] CVD process in separation of nickel from volumetrically contaminated metals. Independent laboratory analyses of the nickel products detected no radiological contaminants using commercially available monitoring equipment and advanced nuclear industry techniques and approved procedures. The complete success of the technology in refining nickel samples from Oak Ridge and Paducah is described later.

Through the use of CVMR[®] patented CVD processes, the elemental nickel from GDP barrier is extracted directly from the contaminated nickel mass and collected as a pure nickel product. The separation occurs at low pressure and at low temperatures. This was validated in a DOE barrier materials pilot program co-funded and completed with B&W using CVMR[®]'s process technology.

DOE Nickel Materials

Nickel material used in the GDPs was purchased as Western Grade commercial product with 99.4% nickel and sintered in classified forms, configurations and applications that were essential for the enrichment process. The process is now antiquated and materials are to be surplused. From an original procurement of 94,000 tons, the current nickel inventory exceeds 35,000 tons of which the majority is volumetrically contaminated. A wide spectrum of radioactive isotopes constitutes ~0.1% of the metal with Technetium, Uranium, Neptunium, Plutonium, and Thorium present.

Criteria for Refined Product

U.S. government and industry requirements for refining of DOE metal assets have established stringent criteria that in many parameters exceed the characteristics of commercial supplies.

First, safe operations through systems engineered to contain, monitor and control hazardous conditions are essential. The radiation hazards from GDP equipment are compounded by the toxicity of reagents and process streams, flammability, and explosive potential of some process components. The use of modern instrumentation and control technologies provides superior management of these risks. These design innovations are directly responsible for improvements to the safety performance of CVD and refining processes in the metallurgical industries.

Second, refined products must meet human health and safety standards for release of radioactive materials. Currently, a proposal modifying the Secretarial suspension on the unrestricted release for recycle of uncontaminated scrap metals originating in DOE "radiological areas" (as defined by 10 Code of Federal Regulations (CFR) Part 835.2, *Occupational Radiation Protection*), with the potential for surface, not volume, radioactivity is under development by the DOE. The volumetric contamination characteristics of most GDP materials and their release are not addressed by the new DOE proposal. Under the proposal for recycle of uncontaminated scrap metals, the candidate scrap metals would only be released following a clearance process to document that they meet requirements for unrestricted release contained in DOE Order 458.1, *Radiation Protection of the Public and the Environment* (DOE 2011). Scrap metals that do not meet these human health and safety standards would be identified as contaminated and would continue to be managed as radioactive material until disposed of as waste. Certainly, all metals to be released for recycle and reuse, including volumetrically contaminated metals,

must satisfy these health and safety criteria.

The most severe standard for release and acceptance is imposed by the industries that consider the metal as a component of their materials' supply chain. Industry requirements are enhanced by potential liabilities and consumer perceptions for contaminated substances. All of the customers for this metal will require that the refined nickel be produced with specific performance parameters and specifications that meet very demanding performance applications. Compliance with these specifications starts with a demand for quality that is superior to commercially available nickel. The presence of NORM and environmental contamination in feedstock has established a well-defined signature that is an inherent *risk de minimus* with a virtually negligible risk to the worker and individual exposed to the commercial supply. Refined metal products from GDP sources must be as clean as the metal resources currently in commercial supply and be radiologically indistinguishable.

As a part of the refining process or an additional step, the classification issues for GDP equipment must be removed. It is intrinsic that a process that “dissolves” the feedstock in an active reagent environment will change the physical and chemical nature of the material. Residues that remain after the target metal extraction may hold classified characteristics and should be controlled and retained by the U.S. Government for appropriate management. All of the products produced by the CVMR[®] processes have no classified characteristics.

Certainly, the refined materials and products must have value that exceeds the cost of refining with added value in the final form. This will generally discourage the production of commodity materials for general use in the metallurgical industry. Special product applications where the unique electrical, mechanical and performance characteristics of the metal are present will produce higher value. Final metal forms that are finished products or that optimize exotic applications will assure the highest value for the GDP materials. The CVMR[®] processes retain the ability to produce multiple product forms that satisfy multiple markets and demands.

The recovery of GDP materials must be compatible with D&D plans and environmental remediation objectives of DOE. Scheduling of material availability, survey of feedstocks for characteristics, and recovery of radioactive residues should be coordinated with the DOE and DOE contractors. Additional benefits for waste minimization and revenues supporting site environmental activities are significant and another direct results from the refining processes.

The life of the metal refinery will not be limited by the availability of GDP materials. Transition to other feedstocks when DOE materials are exhausted can insure operation for the full life of the CVMR[®] equipment, continuity of jobs for highly skilled workers, and supply of high performance supplies for industry.

CVMR[®] Process

The CVD process was developed, in its basic form, at the beginning of the 20th century. At present, 20% of worldwide nickel production is refined using CVD carbonyl technology. Carbonyl technology is a vapor metallurgical refining method based on the ability of some metals to form volatile metal carbonyl compounds. CVMR[®] has taken this hundred-year-old chemical process and modified it to produce unique new products and refine multiple metals for modern technology applications. CVD metals include Ti, Zr, Hf, V, Nb, Ta, Cr, Mo, W, Co, Ni, Fe, Y, Al, Pt, Pd, Ru, Rh, Ni, Fe, and certain other rare-earths.

A fundamental outline of the CVMR[®] process to be applied to DOE nickel assets is presented in **Figure 2**.

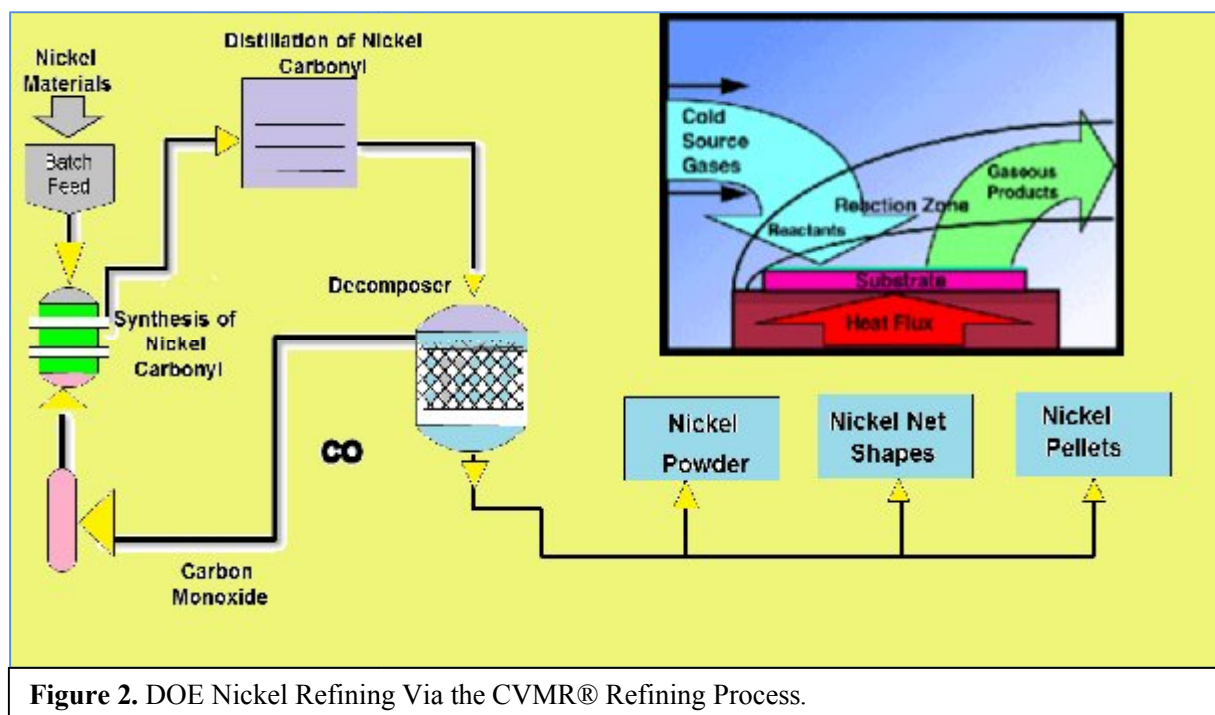


Figure 2. DOE Nickel Refining Via the CVMR® Refining Process.

The reaction of CO at atmospheric pressure with metallic nickel at 40-80°C to form gaseous nickel tetracarbonyl (Ni(CO)₄) was discovered by Lange and Mond in 1889. The reaction is readily reversible, with nickel tetracarbonyl decomposing to metallic nickel and CO at 150-300 °C.

Nickel tetracarbonyl is a volatile liquid that melts at -17.3 °C and boils at 42.5 °C. Under the mild conditions employed for reaction at atmospheric pressure, the carbonyl forming impurities in crude nickel metal do not volatilize. Iron forms a volatile carbonyl, iron-pentacarbonyl, Fe(CO)₅, with a freezing point of -20.5 °C and a boiling point of 103°C, but the rate of formation is slow. Cobalt forms Co₂(CO)₈, which melts at 51°C and decomposes at 52 °C to form Co₄(CO)₁₂ but both are solids with low volatility. Copper, like most elements, does not form carbonyls directly with CO. Thus the extraction of nickel as a carbonyl from a crude metal feed is a highly selective process.

The formation of metal carbonyls by the interaction of CO with reduced metals can be catalyzed to enhance production and efficiency for safe, reliable refining of nickel. A substantial increase in reaction rates is possible from the addition of a controlled amount of catalyst to provide an increase in tonnage quantities of extracted nickel and iron as carbonyls of nickel, cobalt, and iron either selectively for pure metals or together for alloy production.

The carbonyl extraction process is notable not only for its selectivity in the volatilization of the metals as carbonyls but also for the relative ease with which the carbonyls can be separated and recovered under mild conditions to produce high-purity metals. Combinations of nickel and iron carbonyls can be separated by simple fractional distillation because of the large difference in their boiling points.

The carbonyl process makes it possible to produce nickel of very high purity with exceptionally low contaminant content through the use of precise temperature and pressure conditions in the reactor. This is a modern development and innovation where CVMR® has exceptional strength and intellectual property.

The thermal decomposition of nickel-tetracarbonyl is carried out by two different methods. Very rapid

heating of the gas phase produces a fine nickel powder in the gas stream. Alternatively, if the carbonyl gas stream contacts a hot metal surface, nickel deposits on the surface in an exceptionally hard and conformationally precise coating. This deposition effect is used to produce nickel granules by the successive deposition of layers of nickel onto a seed particle, until the required size is attained.

Environmental Controls

CVMR[®] designed facilities are environmentally responsible and follow the toughest safety standards and environmental controls. All of CVMR[®]'s plants in Canada, Germany and China are located in commercial/residential areas. Environmental protection authorities of these countries gave CVMR[®] permits to operate its facilities based on our company's safety record and engineered controls. CVMR[®]'s plants are fully automated and are located in a containment building to eliminate a possibility of environmental emissions (double and triple containment). The CVMR[®] process recycles the reagent gases to minimize air emissions and has no water discharge other than cooling for system air compressors.

The CVMR[®] Process reacts solid feed materials with the selected reagent gas and produces purified metals in the form of forms, pellets, powders or foams. The reagent gases are recycled. All residues are solid. All CVMR[®]'s facilities are using state of the art environmental monitoring system with redundant sampling and operate 24 hours per day. All information is recorded and is available to authorities any time for review.

Radionuclide Separation Demonstration

In 2004, CVMR[®] designed and built a bench scale nickel separation system to demonstrate the partitioning of nickel from GDP samples from Paducah and Oak Ridge. The system was installed in a B&W controlled atmosphere glove box and commissioned on commercial nickel in Toronto. B&W personnel were trained in Toronto and the system sent to Lynchburg for testing.

The objectives of this project were to demonstrate the refining process on three sources of nickel; commercially pure nickel chips, volumetrically contaminated nickel ingot chips, and on contaminated, classified shredded material. A series of six runs were performed using the three types of starting material.

This system was used for clean and contaminated samples. Feed material for each run was 100 grams of 125 mesh machined chips and processed under Lynchburg Technical Center (LTC), Area Operating Procedure B-SML-10. A new reactor and decomposer were used for each test. **Figure 3** is a photograph of the installed demonstration unit.



Figure 3. CVMR® Separation unit installed in a B&W glovebox at LTC.

Four process steps were used to provide the CVD conditions for nickel refining:

1. Purge-pure nitrogen gas for one hour
2. Activation-
 - a) Heating of reactor and decomposer
 - b) Hydrogen gas for one hour
 - c) Temperature optimization
3. Carbonylation
 - a) Multiple pressurization with $\text{CO}/\text{H}_2\text{S}$ mixture
 - b) Waiting for the reaction to occur
 - c) Bleeding off the reactant to the decomposer
4. Passivation
 - a) Purge residual carbonyl with flowing nitrogen gas
 - b) Argon/oxygen gas mixture to passivate carbonyl

A heated decomposer cell was used to precipitate nickel as a deposit on the cell walls. Carbon Monoxide was exhausted to simplify the demonstration. **Figure 4** and **Figure 5** show deposition of pure nickel in the decomposer cell.

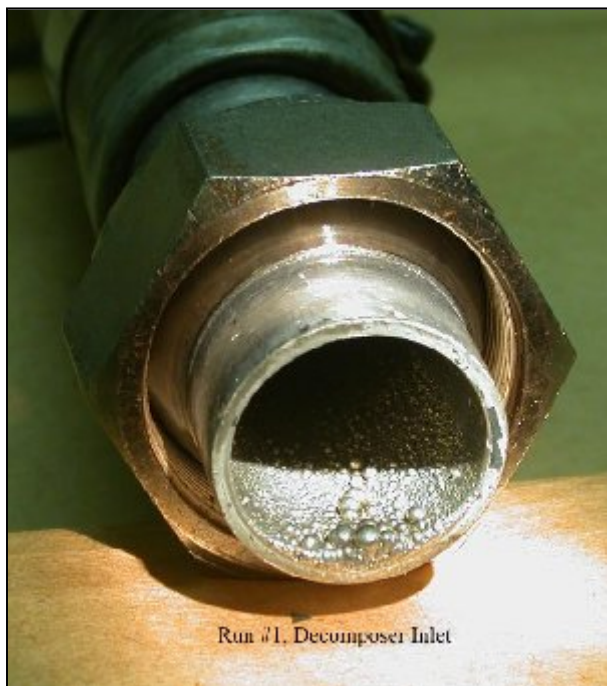


Figure 4. View of decomposer inlet from Run #1 showing nodular product deposits on lower portion of tube.

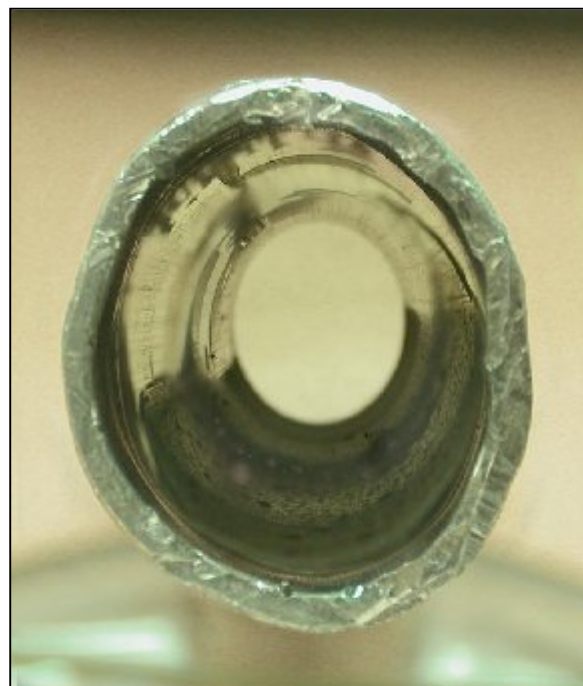


Figure 5. Decomposer from Run #2 showing product deposits on lower portion of tube.

After each run, the amount of purified material was determined by weighing and isotopic analysis was performed. The isotopic analysis was compared to the baseline analysis performed on each of the source materials.

All of the refined samples had activities below detection for all of the isotopes that were analyzed. Scanning electron microscopy was performed on the residue from some of the runs. A photograph of typical refined product material after removal from the decomposer is presented in **Figure 6**.



Figure 6. Brittle segments of pure nickel removed from the decomposer cell.

Isotopic Analysis

Isotopic analysis was performed using two samples from each of the three starting materials and one sample of the product from each of the six runs. Appropriate analytical techniques were used to quantify the activity for each isotope of interest. Each sample was dissolved in acid, split, and analyzed using three separate techniques. **Table II** provides a listing of the methods and isotopes.

Table II. B&W Isotopic Analysis Methods

Method	Isotope
EPA 901.1	Mn-54, Co-60, Cs-134, Cs-137, U-235, Pa-234m
Alpha Spectrometry:	Th-229, Th-228, Th-230, Th-232, U-234, U-235, U-236, U-238, Np-237, Pu-242, Pu-238, Pu-241, Pu-239/Pu-240 ratio
Liquid Scintillation	Pu-241, Tc-99

B&W compiled a detailed isotopic analysis report of the starting material [1] and compiled a detailed isotopic analysis report of the nickel product samples [2]. The specific detection limits are listed in **Table III**.

Table III. Comparison of Minimum Detection Limits

Radionuclide	Isotopic Activity, pCi/g			
	B&W	General Engineering Laboratories, LLC	Environmental Inc.	IAEA Release Limits
Pa-234m	6.07e+1	1.35e+1	1.00e-1	2.0e+1
Th-228	5.00e-2	4.27e-2	8.10e+0	2.0e+1
Th-230	3.50e-2	4.47e-2	9.00e-1	2.0e+1
Th-234	4.18e+0	3.08e+0	1.00e+0	2.0e+1
U-234	1.50e-2	3.77e-2	5.00e-1	2.0e+1
U-235	1.50e-2	1.46e-2	5.00e-1	2.0e+1
U-238	1.00e-2	4.08e-2	5.00e-1	2.0e+1
Np-237	2.50e-2	3.73e-2	3.00e-1	2.0e+1
Pu-239/Pu-240	2.50e-2	3.90e-2	5.00e-1	2.0e+0
Pu-241	2.75e+0	3.11e+0	1.52e+2	2.0e+2
Tc-99	1.05e+1	2.92e+0	9.40e+0	2.0e+1

The commercial nickel feed sample showed very low isotopic content with U-238 showing the highest concentration followed by U-235 along with very low levels of Th-230 and U-234. The total uranium content averaged 0.5 ppm.

The ingot samples from Paducah showed moderately high activities for U-238 followed by U-235, and U-234. This material also had low levels of Np-237. The total uranium content averaged 6.9 ppm.

The shredded samples from Oak Ridge had relatively high levels of U-238 followed by U-235, with significant levels of U-234, Th-232, and Tc-99. There were also low levels of Th-230 and Th-228 and a trace of Pu-241. The total uranium content averaged 7,000 ppm.

The isotopic analysis of the refined nickel product showed all isotopes below the minimum detection limit for each analyte.

Independent Laboratory Analysis

In order to confirm the isotopic analysis results, two independent laboratories analyzed selected samples of the refined nickel. Analytical reports from General Engineering Laboratories, LLC (GEL) and Environmental, Inc. - Midwest Laboratory are available in an Appendix C that is not published with this paper. GEL was asked to perform the same analysis suite as B&W for independent verification of results. Allegheny Technologies, Inc., a potential customer for the refined nickel, contracted Environmental, Inc. to perform their standard nickel analysis. Except for Thorium-230 and Plutonium 239/240 reported by GEL, all the laboratories reported results below their respective minimum detection limits. The minimum detection limits for all three labs were compared to the IAEA release limits for decontaminated material as shown in the **Table III** and in **Figure 7**.

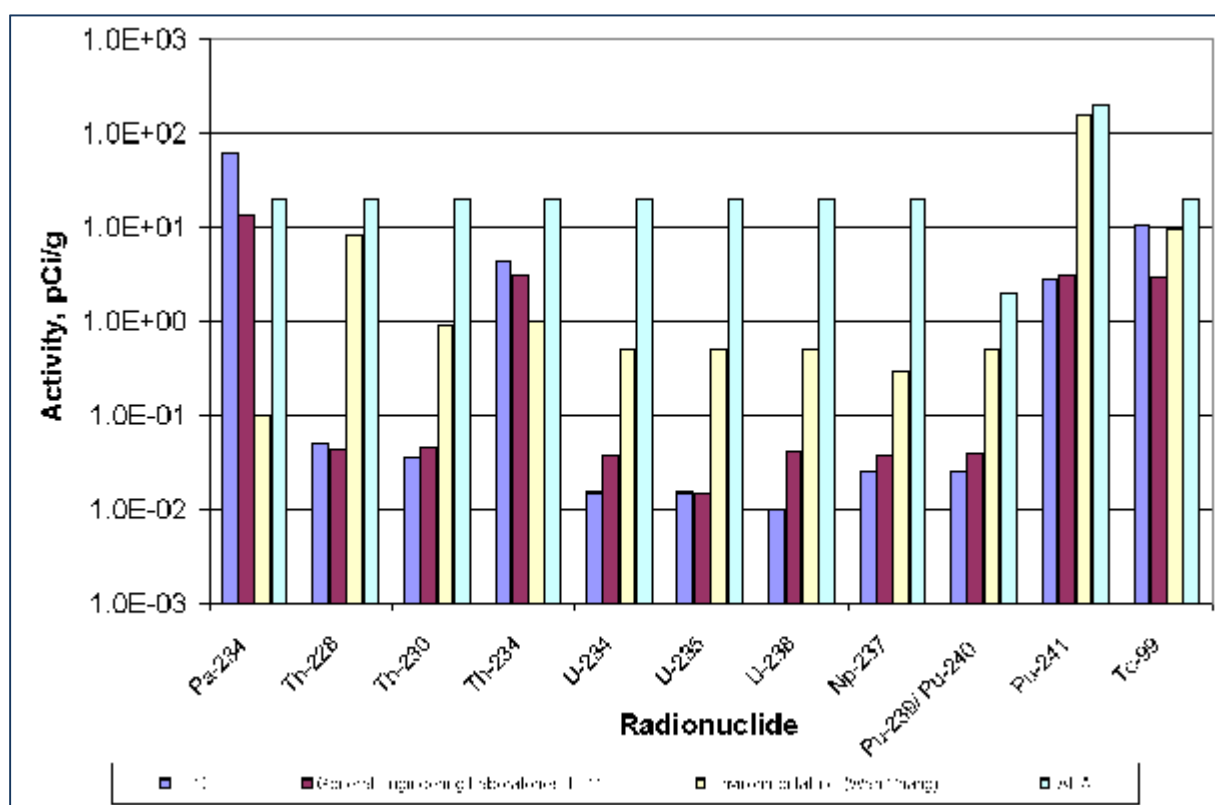


Figure 7. Comparison of Minimum Detection Limits for Three Labs and IAEA.

In comparing the results, GEL achieved similar minimum detection limits for the analysis suite, which verifies the results from B&W. For Th-230 and Pu-239/240, GEL had measured results just above their minimum detection limit with the lower uncertainty for the results being below the minimum detection limit. For Protactinium-234m, both B&W and GEL had minimum detection limits, which were at or above the IAEA release limits for decontaminated material. Therefore, the analysis method used by both labs for Protactinium-234m is not acceptable for comparison to the IAEA release limit.

In comparing the detection limits of the labs to the IAEA release limits for decontaminated material, the following points can be made:

- All of the labs reported Technetium-99 slightly lower than the IAEA release limits.
- For Thorium-230, Plutonium –239/240, Plutonium-241, B&W and GEL achieved minimum detection limits significantly lower than the IAEA release limits, while Environmental Inc.'s minimum detection limits were only slightly lower,
- For Protactinium-234m and Thorium-234, Environmental Inc. achieved a detection limit significantly lower than the IAEA release limits while B&W's and GEL's minimum detection limits were only slightly lower or equivalent to the IAEA release limit.

The results showed the refining process effectively purified the nickel in a predictable and reproducible manner resulting in no detectable activity of any of the isotopes that were characterized. Characterization of the refined material was done at LTC and at two independent laboratories. All of the refined material had isotopic levels far below IAEA release limits. Additionally, the refining of the classified form produced unclassified material.

The contaminated samples from Oak Ridge and Paducah GDPs had higher levels of activity than commercially available nickel. LTC isotopic analysis of the refined nickel product showed no detectable radionuclides. The isotopic analysis was compared to the baseline analysis performed on each of the source materials. Samples of the product from the fifth run that contained nickel with the highest levels of radioactive contamination were sent to two different outside laboratories: General Engineering Laboratories, LLC (GEL) and Environmental, Inc. – Midwest Laboratory.

In comparing the results, GEL achieved similar minimum detection limits for the analysis suite, which verifies the results from B&W. For Thorium-230 and Plutonium –239/240, GEL had measured results just above their minimum detection limit with the lower uncertainty for the results being below the minimum detection limit. For Protactinium-234m, both B&W and GEL had minimum detection limits, which were at or above the IAEA release limits for decontaminated material. Therefore, the analysis method used by both labs for Protactinium-234m is not acceptable for comparison to the IAEA release limit.

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The results showed the refining process effectively purified the nickel in a predictable and reproducible manner resulting in no detectable activity of any of the isotopes that were characterized. Characterization of the refined material was done at LTC and at two independent laboratories. All of the refined material characterized had isotopic levels far below IAEA release limits. Additionally, the refining of the classified form of the material allowed the product to be handled as unclassified material. A summary of the isotopic analysis data for the starting material is provided in **Table V**.

As expected, the contaminated samples from Oak Ridge and Paducah GDPs had higher levels of activity than commercially available nickel. LTC isotopic analysis of the refined nickel product showed no detectable radionuclides present. The isotopic analysis was compared to the baseline analysis performed on each of the source materials.

Isotopic Analysis Summary of Starting Material													
(Activity converted to weight ratios in ug/kg)													
Material Source	²³⁵ U	²³⁸ U	²³² Th	²³⁴ U	²³⁸ U	²³⁷ Np	⁹⁹ Tc	²³⁹ U	²⁴⁰ Pu	²³⁵ Th	²³⁸ Th	²⁴¹ Pu	Total Uranium Content
Commercial	1.1E-01	2.6E-01	1.2E-01	2.3E-01	1.1E-01	MDA	MDA	MDA	MDA	MDA	MDA	MDA	1.5 ppm
Paducah	7.1E+01	4.2E+01	1.0E+01	2.0E+01	MDA	MDA	1.5E-02	2.5E+01	MDA	MDA	MDA	MDA	1.3 ppm
Oak Ridge	7.0E+05	2.0E+05	5.5E+01	1.9E+05	MDA	MDA	3.9E-03	MDA	3.3E+03	8.4E+02	1.8E+04	MDA	7,000 ppm

Highest amounts of each material type is shown
MDA is Minimum Detectable Activity

Table V. Isotopic Analysis Summary of Starting Material.

Samples of the product from the fifth run that contained nickel with the highest levels of radioactive contamination were sent to two different outside laboratories: General Engineering Laboratories, LLC and Environmental, Inc. – Midwest Laboratory. These laboratories also detected no radionuclides for the analysis suite as shown in **Table VI**, verifying the results from LTC.

A supplemental Appendix to this paper is available that presents analytical reports for materials as received and batch results for refined nickel.

Table VI. Analytical results for decontaminated nickel. The highest amounts of each material type are shown.

Isotopic Analysis Summary of Product Material													
(Activity converted to weight ratios in ug/kg)													
LTC Laboratory													
Material Source	²³⁵ U	²³⁸ U	²³² Th	²³⁴ U	²³⁸ U	²³⁷ Np	⁹⁹ Tc	²³⁹ U	²⁴⁰ Pu	²³⁵ Th	²³⁸ Th	²⁴¹ Pu	Total Uranium Content
Commercial	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	< 0.01 ppm
Paducah	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	< 0.01 ppm
Oak Ridge	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	< 0.01 ppm
Highest amounts of each material type is shown. MDA is Minimum Detectable Activity of the laboratory.													
General Environmental Laboratories, LLC													
Material Source	²³⁵ U	²³⁸ U	²³² Th	²³⁴ U	²³⁸ U	²³⁷ Np	⁹⁹ Tc	²³⁹ U	²⁴⁰ Pu	²³⁵ Th	²³⁸ Th	²⁴¹ Pu	Total Uranium Content
Paducah	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	< 0.01 ppm
Highest amounts of each material type is shown. MDA is Minimum Detectable Activity of the laboratory.													
Environmental, Inc - Midwest Laboratory													
Material Source	²³⁵ U	²³⁸ U	²³² Th	²³⁴ U	²³⁸ U	²³⁷ Np	⁹⁹ Tc	²³⁹ U	²⁴⁰ Pu	²³⁵ Th	²³⁸ Th	²⁴¹ Pu	Total Uranium Content
Oak Ridge	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	MDA	< 0.01 ppm
Highest amounts of each material type is shown. MDA is Minimum Detectable Activity of the laboratory.													

No Detectable Radionuclides

The results showed the CVD effectively processed the nickel, resulting in no activity detected and well below the IAEA limit of any of the isotopes that were characterized. The final results are compared in Figure 8.

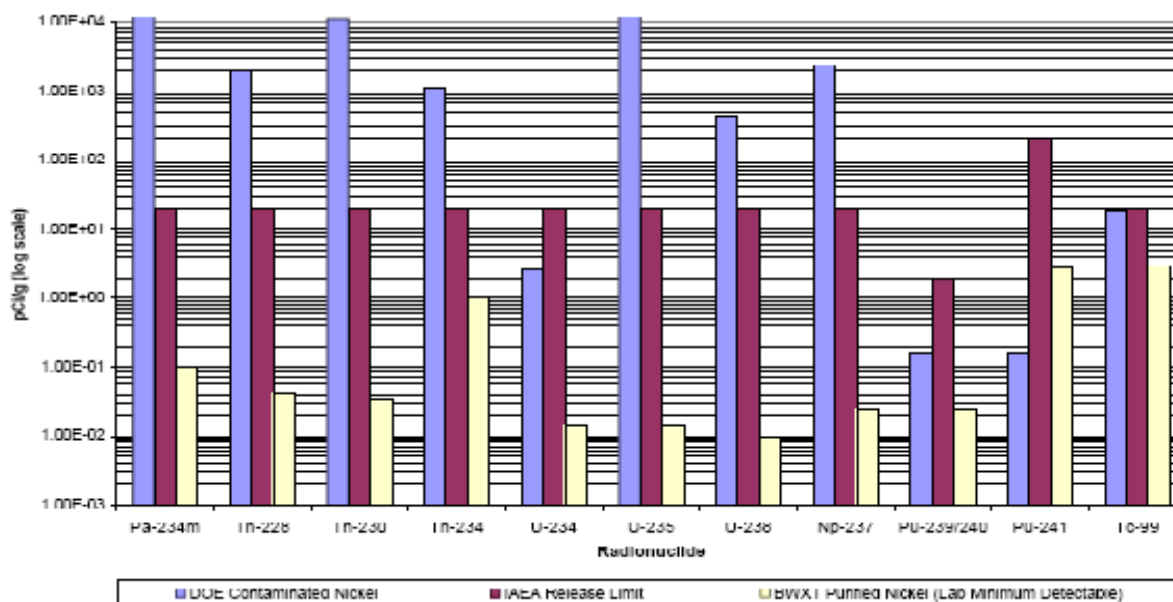


Figure 8. No radionuclides were found in Nickel Refined from GDP samples from Oak Ridge and Paducah.

CONCLUSION

The CVD process provides refined nickel products with no detectable radionuclides from GDP contamination. CVMR® systems using CVD are currently used for the production of a wide spectrum of nickel products where the separation of nickel provides innovative forms and performance of very high purity nickel products. Utilization of these systems for refining of DOE metal assets will require minimal modification to current CVD process systems and size reduction equipment to assure efficient metal refining. Considerations are needed for radiation hazards present in the preparation of GDP materials.

Additional manufacturing facilities will be needed to be located with close proximity to refined metal production. These industrial operations will also require independent, third party verification of product quality and the complete removal of radioactive contamination.

Benefits of CVMR® Refining of DOE Metals

Reduction of the waste material to 1% of the original mass has direct benefits to the environmental management objectives of the DOE. These benefits are increased when on-site disposal is unavailable. Clear accountability for the radioactive components is established with **NO** carryover of radioactivity in finished products.

CVMR® will produce nickel products that are cleaner than the nickel in U.S. commerce and used by

industry and consumers today. This quality expectation will be verified by an independent quality control organization that will affirm the purity of all products.

The CVMR® process and systems for nickel refining is well suited for GDP materials and will provide value through environmental stewardship, recovery of high value assets, and support of the DOE environmental remediation programs as the refined nickel generates additional benefits to Ohio communities.

Production of metal products that have no detectable radionuclides and a purity that is superior to commercially available supplies will produce a near term return to the cleanup process as the legacy wastes are removed. This material will then become a strategic resource for the economic development of DOE sites and a future that includes refining of non-radioactive metals.