

*Manufacturing Sciences Corporation*

**Sampling and Analysis Plan  
For Nickel Recycle**

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FINAL DRAFT

## TABLE OF CONTENTS

<b>1</b>	<b>OBJECTIVES.....</b>	<b>3</b>
1.1	PHASE I SAMPLING AND ANALYSES OBJECTIVES .....	3
1.2	PHASE II SAMPLING AND ANALYSES OBJECTIVES.....	3
<b>2</b>	<b>SAMPLING PLAN, PHASE I.....</b>	<b>3</b>
2.1	ANODE SAMPLING .....	3
2.2	ANODE SOLUTION ( <i>ANOLYTE</i> ) SAMPLING .....	4
2.3	CATHODE SOLUTION ( <i>CATHOLYTE</i> ) SAMPLING .....	4
2.4	CATHODE SAMPLING .....	4
<b>3</b>	<b>SAMPLING PLAN, PHASE II.....</b>	<b>5</b>
3.1	ANODE SAMPLING .....	5
3.2	ANOLYTE SAMPLING.....	5
3.3	CATHOLYTE SAMPLING.....	5
3.4	CATHODE SAMPLING .....	5
<b>4</b>	<b>RADIOLOGICAL ANALYSES, PHASES I AND II.....</b>	<b>6</b>
4.1	ANODE AND CATHODE ANALYSES.....	6
4.2	ANOLYTE AND CATHOLYTE ANALYSIS .....	6
<b>5</b>	<b>QUALITY CONTROL.....</b>	<b>6</b>
<b>6</b>	<b>RECORDS .....</b>	<b>7</b>

## 1 Objectives

Recycling of nickel will be conducted in two phases, Phase I and Phase II. Sampling and analyses will follow these phases.

### 1.1 Phase I Sampling and Analyses Objectives

The sampling and analysis objectives of Phase I are to provide confirmatory proof of the process and methods of contaminated nickel recycle.

During Phase I, production anodes and cationic membranes will be used to produce production cathodes. The *current density*, *anolyte recirculation* and *filtration* will be approximately the same as in the planned Phase II production scale processes.

The same type of sampling planned for the Phase II production processes will be undertaken for Phase I, but with a much greater number of samples collected and analyzed during Phase I. The sampling and analyses during Phase I will demonstrate the capabilities of the system to consistently remove contaminants to the desired level thus allowing the Phase II sampling to focus on only the critical parameters to control the process and to demonstrate the releasability of the finished ingot.

Phase I sampling and analysis is also expected to prove the uniformity of individual cathode ingots and the uniformity of ingots within a process bath.

### 1.2 Phase II Sampling and Analyses Objectives

Sampling and analyses during Phase II will focus on the critical parameters identified during Phase I. Focusing on critical parameters will assure the process remains stable and produces releasable metal.

The objectives of Phase II are to demonstrate the releasability of the finished ingot. Samples will be collected and analyzed to verify that production cells are operating correctly and producing virtually contaminant free nickel cathodes suitable for unrestricted release.

## 2 Sampling Plan, Phase I

### 2.1 Anode Sampling

As the anode ingots are prepared, metallic samples will be collected. The samples may be collected as a liquid when the ingot is poured or as

shavings taken either by drilling into the ingot or shaving a small amount from the surface of the ingot.

## 2.2 Anode Solution (*anolyte*) Sampling

Sampling of the anolyte is important to process control as comparison can be made between the anolyte and the catholyte allowing for determination of the membrane efficiencies.

Samples will be collected in any convenient manner as the anolyte is in constant circulation and will be homogenous. Samples will be identified with appropriate information and submitted to the analytical laboratory following appropriate instructions and/or procedures.

The process manager will determine the scheduling of these samples and the analytical requirements.

## 2.3 Cathode Solution (*catholyte*) Sampling

The catholyte will be sampled daily as a minimum throughout cathode generation during Phase I. The sample will consist of a volumetric composite of several random samples taken from various points around the cathode, and is to include subsurface samples. The composite sample(s) will be identified with appropriate information and submitted to the analytical laboratory following appropriate instructions and/or procedures.

The data from this sampling will establish the relationship between the technetium and uranium concentrations within the solution and the technetium and uranium concentrations within the refined metal. Catholyte technetium concentration will also provide information on the condition of the cationic membrane. Increases in the concentration of technetium would indicate a possible failure of the membrane. This parameter will be important in process control.

## 2.4 Cathode Sampling

After the cathode has been removed from the electro-refining bath, removable contamination surveys will be performed following MSC's Work Instruction *Unrestricted Release Survey of Materials*.

Volumetric samples will then be taken from the cathode in a minimum of three different locations. These samples will be collected by drilling through the ingot (*see MSC Work Instruction Volumetric Sampling of Refined Nickel Ingots*).

The samples will be identified uniquely with the cathode ingot. The metal will be packaged in a suitable container and submitted to the analytical laboratory following appropriate instructions and/or procedures.

### **3 Sampling Plan, Phase II**

#### **3.1 Anode Sampling**

As the anode ingots are prepared, a metallic sample will be collected from each melt batch. The samples may be collected as a liquid when the ingot is poured or as shavings taken either by drilling into the ingot or shaving a small amount from the surface of the ingot.

#### **3.2 Anolyte Sampling**

Samples will be collected in any convenient manner as the anolyte is in constant circulation and will be homogenous. Samples will be identified with appropriate information and submitted to the analytical laboratory following appropriate instructions and/or procedures. The process manager will determine the scheduling of these samples and the analytical requirements.

#### **3.3 Catholyte Sampling**

The catholyte will be sampled at the start of a new cathode and at the completion of a cathode as a minimum. Additional samples may be collected during cathode generation but will not be required. The catholyte sampling will be the primary focus of the process control and any additional samples that may be collected will be for process control purposes.

Samples may either be grabs or composites. The sample type and collection will be defined from Phase I information to best target critical parameters. The sample(s) will be identified with appropriate information and submitted to the analytical laboratory following appropriate instructions and/or procedures.

#### **3.4 Cathode Sampling**

Volumetric sampling of each finished ingot will be performed prior to any unrestricted release. Samples will be taken after all removable contamination surveys have been completed.

The sample(s) will consist of the shavings from a drilling completely through the ingot. The metallic sample will be packaged in a suitable container and submitted to the analytical laboratory following appropriate

instructions and/or procedures (see Work Instruction for *Volumetric Sampling of Refined Nickel Ingots*).

#### 4 Radiological Analyses, Phases I and II

##### 4.1 Anolyte and Catholyte Analysis

The analytical methods utilized for the anolyte and catholyte process solutions will be very similar to the analysis for the metallic samples (Section 4.2). The primary exception will be in sample count times as count times for the process solutions will likely be shorter. These solutions are process control solutions only and therefore detection levels need not be as stringent as those for unrestricted release. Liquid scintillation and gamma spectroscopy will be utilized.

##### 4.2 Anode and Cathode Analyses

Analysis for  $^{99}\text{Tc}$  and for uranium will be conducted using liquid scintillation alpha/beta pulse discrimination techniques. Count time is expected to be approximately 100 minutes per sample.

In both Phases, procedures for analyzing metal samples from anodes and cathodes will be much the same, with the only significant exception being count times. Shorter count times should be adequate for quantifying anode base-line data. However, longer count times will likely be required to achieve the levels of detection necessary for unrestricted release.

Sample size will be approximately 15 grams of material. The sample will be digested in a concentrated acid solution; the anticipated solvent for the material is nitric acid. The dissolved sample will then go through several evaporation and dilution steps prior to a final dilution which will be between 100 mL and 500 mL.

Additionally, a percentage of cathode samples will be analyzed to verify no other radionuclides are present in the refined nickel. Process knowledge indicates that this additional analysis is not necessary, however as a conservative measure this verification will be performed.

#### 5 Quality Control

All laboratory instrumentation will be calibrated, operated, and maintained in accordance with appropriate MSC Instructions, Instructions Guides, and/or Procedures.

**6 Records**

Records of all measurements and evaluations will be properly identified and maintained for any regulatory or customer review. All records will be maintained in accordance with appropriate MSC Instructions, Instruction Guides, and/or Procedures.

## TITLE: UNRESTRICTED RELEASE SURVEY OF MATERIALS

### 1.0 PURPOSE

The purpose of this Instruction is to provide requirements for the release of material for unrestricted use.

### 2.0 SCOPE

This procedure details the steps necessary for survey, tracking and documentation of material released for unrestricted use from Manufacturing Sciences Corporation, Kerr Hollow Road facilities.

### 3.0 REFERENCES

- 3.1 U. S. Atomic Energy Commission. *Regulatory Guide 1.86, Termination of Operating Licenses for Nuclear Reactors*, Retyped by U.S. EPA, August 1997.
- 3.2 Manufacturing Sciences Corporation. *Radioactive Material License R-01078-L00 submittal*.

### 4.0 DEFINITIONS

- 4.1 *Surface Contamination Release Limit* - the allowable surface activity per unit area. The limit is taken from Regulatory Guide 1.86, Table 1 (Attachment 1.) and actual isotopic considerations relative to the material in question.
- 4.2 *Batch* - materials, items, components, etc. from the same customer with the same MSC Job Number and Customer ID Number. This can consist of a single SMC to several large components to many SMC containers.
- 4.3 *Unrestricted Release* - an item or items that have met the criteria relative to *surface*, *removable*, and *maximum* activity established by Regulatory Guide 1.86. The item(s) is/are considered to be releasable to non-radiologically controlled areas for unrestricted use.
- 4.4 *Inaccessible Area* - an area on an item considered for release that cannot be surveyed for unrestricted release with standard survey meters or techniques. The Survey Manager or Designee shall determine unrestricted release status for those items identified with inaccessible areas.

### 5.0 PRECAUTIONS

None.

## 6.0 PREREQUISITIES

- 6.1 Performance of radiological surveys for unrestricted release is limited to personnel who are qualified for the task involved. Qualification is determined in accordance with each individual's Role Proficiency Record.
- 6.2 Personnel considered to be in training may perform surveys under the guidance of a qualified individual. *Initial* of the completed unrestricted release survey record information by the trainee must be accompanied by the initials or signature of the qualified individual.
- 6.3 Review and adherence to the Radiation and Industrial Safety Work Permit is required prior to beginning as well as during work activities.
- 6.4 Calibration of all radiological instrumentation shall be current.
- 6.5 Response check (source check) of instruments is required prior to beginning work.
- 6.6 Minimum Detectable Activity (MDA) for hand-held instruments shall be established prior to survey activities. MDA shall be less than the release limits specified for removable radioactivity.
- 6.7 Background radiation levels must be maintained low enough so that the survey instrument MDA is less than the release limit for the material (*see* Attachment 2. for MDA calculations table for various instruments).
- 6.8 Minimum detectable activity (MDA) for the Laboratory low level counting instrumentation shall be established prior to survey activities for unrestricted release by laboratory qualified personnel. MDA shall be less than the release limits specified for removable radioactivity.
- 6.9 The Laboratory low level counting instrumentation shall be the basis for determination of removable contamination for unrestricted release. Utilization of the Laboratory low level counting instrumentation shall be in accordance with the Instruction or Instructional Guide for that instrument.
- 6.10 Release Limits based on Regulatory Guide 1.86 will be determined for the material being surveyed by the RSO or Designee.

## 7.0 RESPONSIBILITIES

### 7.1 Radiation Safety Officer (RSO)

- 7.1.1 The RSO is responsible for all unrestricted release of materials from MSC.

- 7.1.2 The RSO may designate responsibilities and/or certain tasks of the unrestricted release program.
- 7.1.3 The RSO or designee will establish appropriate unrestricted release limits. These established limits will take into account isotopic contamination considerations, material geometry considerations if necessary, material composition considerations if necessary, and any other applicable considerations which may be determined as necessary.

## 7.2 Survey Manager

- 7.2.2 The Survey Manager or Designee will provide the Technicians with job assignments. The job assignments will include information on the customer and the *surface contamination release limits*.
- 7.2.3 The Survey Manager or Designee *may* adjust the limits to be more conservative if this is desired. Any and all adjustments and calculations will be maintained in a log and will be traceable to Customer ID and/or MSC Job Number.
- 7.2.4 It will be left to the Survey Manager's or Designee's discretion as to how the *surface contamination release limits* will be provided.
- 7.2.5 The Survey Manager or Designee will evaluate all items that exhibit elevations greater than the provided *surface contamination release limit* which are not subject to immediate rejection. Evaluation will involve maximum activity considerations allowable by U.S. Atomic Energy Commission Regulatory Guide 1.86 (Attachment 1.).
- 7.2.6 After the surface contamination evaluation is completed, the Survey Manager or Designee will complete the Unrestricted Release Survey Record paperwork by providing the appropriate information after review of the removable contamination survey analyses and officially release the material.
- 7.2.7 The Survey Manager or Designee shall see that prior to materials leaving the MSC Kerr Hollow Road site for unrestricted release, the materials have a dose rate survey completed by authorized personnel with appropriate instrumentation. Any levels greater than 10  $\mu\text{R/hr}$  above background will be investigated further. Further investigation may require segregation and/or unloading of materials.
- 7.2.8 The Survey Manager or Designee shall insure that information within the MIS Systems is maintained as current and accurate as possible with regard to Survey Operations specific information.

### 7.3 Survey Technicians

- 7.3.1 Survey Technicians are responsible for the completion of surface contamination surveys including all associated paperwork.
- 7.3.2 Surveys Technicians are responsible for appropriate instrument response checks, background checks, and/or other operational checks which are prescribed.
- 7.3.3 Survey Technicians are responsible the collection of wipes for evaluation of removable contamination.
- 7.3.4 Survey Technicians are responsible for maintaining good general housekeeping within the areas utilized for survey.
- 7.3.5 The RSO or Survey Manager may give Survey Technicians with demonstrated capabilities additional responsibilities.

7.4 MSC Analytical Laboratory will perform contamination survey wipe analysis. Analysis will consist of individual wipe evaluation with low level  $\alpha/\beta$  counting instrumentation. Utilization of this instrumentation will be in accordance with appropriate instructions and instruction guides.

## 8.0 PROCEDURES FOR UNRESTRICTED RELEASE SURVEYS

### 8.1 *Manual* Surface Contamination Surveys

- 8.1.1 On receipt of survey work assignments, record on the appropriate record the *date*, *time*, and *MSC Job Number* of the materials. *Surface Contamination Release Limits* will be provided with survey assignments.
- 8.1.2 Record the incoming container number and net weight received. Record the appropriate instrumentation information.
- 8.1.3 Conduct a removable contamination survey on the materials with masslinn wiping material.
  - 8.1.3a This survey should include all reasonably accessible pieces and surfaces with the materials still in the transport containers.
  - 8.1.3b Evaluate the masslinn wipe for contamination. If contamination does not exceed 1,000 dpm per wipe, proceed with surface contamination survey. If, however, contamination exceeds 1,000 dpm, perform an additional gross removable contamination survey on the materials as before and evaluate the wipe(s) again. If the materials fail a second time, notify the Survey Manager or Designee for direction on proceeding.

8.1.4 Remove components from the containers to an appropriate survey area. Record the general area background in  $\mu\text{R/hr}$  on the record.

8.1.5 Utilizing appropriate instrumentation, perform a surface scan of the materials. The maximum allowable probe distance while scanning is  $\frac{1}{2}$ " from the surface of the material. The maximum allowable scan rate is 2" per second.

*Note: Inaccessible Areas.* Components with inaccessible areas encountered during surface scans will be disassembled to allow access to all surfaces. Those items that cannot be disassembled will be referred to the Survey Manager or Designee for determination.

8.1.5a If, while scanning, a noticeable increase in count rate occurs, hold the probe stationary for at least 25 seconds at the point of increase to allow the count rate to stabilize.

8.1.5b If the count rate exceeds the provided *surface contamination release limits* specified, note the area of the elevated count rate by marking with an appropriate marker.

8.1.5c The piece is *rejected* if (1) greater than 25% of the piece's total surface area exhibits elevations greater than the provided release limit, or if (2) any elevation greater than three times the provided *surface contamination release limit* is detected. All reject pieces will immediately be placed in the appropriate waste container.

8.1.5d If the piece exhibits elevations greater than the release limit but less than *three times* the release limit and covers an area less than 25% of the total surface area, the Survey Manager or Designee will be informed and will then make a determination. The Survey Manager or Designee may make maximum allowable exceptions on a case by case basis.

8.1.5e Point source considerations may be applied if the materials exhibit point source characteristics and the technician performing the survey is qualified with regard to point source considerations. The RSO or Designee will make qualification determinations.

8.1.5f If the piece exhibits *no elevations*, proceed with wipe collection.

8.1.6 After the piece has passed the surface scan evaluation, the technician will collect removable contamination wipes.

8.1.6a The Survey Technician will take enough 100  $\text{cm}^2$  wipes (with appropriate material, i.e. Mohawk Wipes, other disk wipes) to ensure a representative sampling of the batch surveyed.

- 8.1.6b The technician shall field check all the wipes associated with the piece just scanned in order to allow segregation of pieces which exhibit removable contamination greater than 1000 dpm/100 cm<sup>2</sup>. Field checks are also used to assure that no excessively elevated wipes proceed to the laboratory.
- 8.1.7 Each Survey Technician will initial or uniquely mark each piece after evaluation and wipe collection.
- 8.1.8 After wipe collection, wipe field check, and marking of the piece, place the piece into a "clean unrestricted release holding container" and proceed with the next piece.
- 8.1.9 Collect and assemble the wipes in a manner as to minimize cross contamination and label them appropriately for future laboratory analysis.
- 8.1.10 At the completion of a "batch" of material, complete the required blocks on the record (net weight released and rejected, container numbers, etc.) and submit the wipes from that batch to the laboratory for evaluation following appropriate laboratory procedures for sample submission.
- 8.1.11 Turn all related paperwork over to the Survey Manager or Designee, obtain new assignments from the Survey Manager or Designee, and continue as before with the new assignment.

## 8.2 *Conveyorized* Surface Contamination Surveys

- 8.2.1 On receipt of survey work assignments, document on the appropriate record the *date*, *time*, and *MSC Job Number* of the materials. *Surface Contamination Release Limits* will be provided with survey assignments.
- 8.2.2 Record the incoming container number and net weight received. Record the appropriate instrumentation information.
- 8.2.3 Conduct a removable contamination survey on the materials with masslinn wiping material.
- 8.2.3a This survey should include all reasonably accessible pieces and surfaces with the materials still in the transport containers.
- 8.2.3b Evaluate the masslinn wipe for contamination. If there is no contamination > 1,000 dpm per wipe, proceed with surface contamination survey. If, however, contamination exceeds 1,000 dpm per wipe, perform an additional removable contamination survey on the materials as before and evaluate the wipe(s) as before. If the materials fail a second time, notify the Survey Manager or Designee for direction on proceeding.

- 8.2.4 Only approved personnel shall operate and conduct surface contamination surveys with conveyORIZED monitoring equipment. Approval requires passing a written examination and displayed operational competence as determined by the RSO or Designee.
- 8.2.5 All conveyORIZED monitors will be calibrated annually as a minimum. All instruments will be response checked prior to operations and must meet pre-operational criteria before any materials may be surveyed.
- 8.2.6 The Survey Technician shall perform a  $\mu\text{R/hr}$  background check with an appropriate instrument and record the background on the Unrestricted Release Survey Record. The background should be at or below 50  $\mu\text{R/hr}$  and stable. If it is above 50  $\mu\text{R/hr}$ , the system may not allow operation (the system provides internal checks to ensure alarm levels are obtainable with the current background level).
- 8.2.7 The Survey Technician shall set the instrument alarm level to the appropriate activity for the materials that will be evaluated. This information will be provided by the RSO or Designee.
- 8.2.8 The Survey Technician shall complete the appropriate Response Check Form (Attachment 4.) by verifying operation diagnostics checks, response checks, and false alarm checks.
- 8.2.9 The Survey Technician shall ensure that all materials placed on the belt are positioned correctly. Materials are to be positioned flat on belt and no materials are to be stacked. The belt is laid out with a grid pattern. A minimum of 50% of any grid area *with material* must be covered. Grid areas may be left empty. Materials may cross over into several grids but all grids in question must be covered by at least 50%. Grids may be covered by more than 50%.
- Note: Inaccessible Areas.* Components with inaccessible areas encountered during surface scans will be disassembled to allow access to all surfaces. Those items that cannot be disassembled will be referred to the Survey Manager or Designee for determination.
- 8.2.10 During monitoring operations, a new background will be taken at a minimum every two hours. The system is set to automatically take a background after two hours of operation.
- 8.2.11 Any materials that fail may be re-evaluated. Materials that fail are rejected if they are not re-evaluated. If materials fail again on re-evaluation, they will be rejected. Any rejected materials shall be immediately placed in an appropriate waste container.

- 8.2.12 If the materials pass monitoring, take enough 100 cm<sup>2</sup> wipes (with appropriate material, i.e. Mohawk Wipes, other disk wipes) to ensure a representative sampling of the batch surveyed. Wipes shall be field checked after collection. Place the materials into the appropriate "clean unrestricted release holding container."
- 8.2.13 Collect and assemble the wipes in a manner as to minimize cross contamination and label them appropriately for future laboratory analysis.
- 8.2.14 At the completion of a "batch" of material, complete the required blocks on the record (net weight released and rejected, container numbers, etc.) and submit the wipes from that batch to the laboratory for evaluation following appropriate laboratory procedures for sample submission.
- 8.2.15 Turn all related paperwork over to the Survey Manager or Designee, obtain new assignments from the Survey Manager or Designee, and continue as before with the new assignment.
- 8.2.16 At the end of the shift, remove all materials from the belt and power the system down. Power the printer down after the reports have been removed and filed or logged (*note: never advance or retract the printer paper using the knob on the right side of the printer*). The floor under the belt should be swept and all loose debris should be removed. The lower bank of detectors should be periodically vacuumed to remove any debris that may have fallen onto them.

### 8.3 *Removable Contamination Surveys*

- 8.3.1 The MSC Analytical Laboratory will analyze the contamination wipes submitted for evaluation with appropriate instrumentation following appropriate Instructions, Instruction Guides, and/or Procedures.
- 8.3.2 The MSC Analytical Laboratory contamination wipe analysis results will be reported to the Survey Manager or Designee for review and evaluation.
- 8.3.3 The Survey Manager or Designee will review and evaluate the contamination wipe analysis results and complete the Unrestricted Release Survey Record, supplying the necessary information for the Record.
  - 8.3.3a Review and evaluation of the contamination wipe analysis results will involve comparisons of results against the removable release limits of the relative materials. The removable release limit is taken from Regulatory Guide 1.86.
  - 8.3.3b After removable contamination has been assessed and accepted as passable, the Survey Manager or Designee will complete the Unrestricted Release Survey Record form (Attachment 3.),

assemble the documentation for the complete Unrestricted Release Record, review the complete record, and unrestricted release the materials.

8.3.3c All Unrestricted Release Records are MSC QA Records and will be maintained in accordance with approved instructions.

#### 8.4 Refined Nickel Ingot Contamination Surveys

8.4.1 Ensure the surface of the electro-refined nickel ingot (from now on called the "ingot") is clean and dry. Mark the ingot with a unique identifying number using an indelible pen if not already identified.

8.4.2 Perform removable contamination surveys following the above procedures. Either manual or conveyORIZED means may be utilized for surface contamination surveys.

8.4.3 In addition to surface contamination surveys, each nickel ingot will be volumetrically sampled following MSC Work Instruction *Volumetric Sampling of Refined Nickel Ingots* and analyzed by the analytical laboratory. Analysis results must be within established limits before the nickel may be released for unrestricted use.

8.4.4 Information with regard to unrestricted release of ingots will be summarized on the *Nickel Unrestricted Release Shipment Summary* form (Attachment 6.). No shipments will exceed 20 tons of nickel metal.

#### 8.5 Release Limits

8.5.1 Release Limits are derived directly from U.S. Atomic Energy Commission Regulatory Guide 1.86, Table 1.

8.5.2 Non-detectable isotopes are considered when evaluating materials for unrestricted release by the RSO or Designee.

8.5.3 Isotopic decays and their relative decay intensities and energies are considered when evaluating materials for unrestricted release by the RSO or Designee.

8.5.4 Isotopic information is taken from either client-supplied data and/or MSC analytical evaluation.

#### 8.6 Alarms at Outside Facilities

8.6.1 Any alarms of radiation detectors at outside facilities (i.e. scrap yards or procurers of recycled metals) shall initiate a response by MSC personnel.

- 8.6.2 As soon as possible after notification of such alarms MSC shall dispatch qualified personnel to the reporting site to verify the cause of the alarm.
- 8.6.3 MSC personnel will perform appropriate surveys to isolate any suspect materials and take corrective actions, when warranted.
- 8.6.4 When material that has been positively identified as MSC material and does not meet Unrestricted Release criteria, an internal Non-Conformance Report will be generated, MSC will take possession of the material, TDRH will be notified, and an Exemption For Transport will be requested if appropriate.
- 8.6.5 All material returned to MSC from an outside alarm response shall be through approval of TDRH.
- 8.6.6 When material that is not MSC material is identified as exceeding Unrestricted Release criteria, TDRH will be notified and disposition determined in accordance with TDRH direction.

*Note:* Alarms may be caused by cumulative effect and may not indicate material that does not meet Unrestricted Release criteria.

## 9.0 RECORDS

The Unrestricted Release Record shall consist of the Unrestricted Release Survey Record (Attachment 3.), materials isotopic evaluations, removable contamination wipes analysis data, and conveyORIZED survey monitor data if applicable. All Unrestricted Release Records are MSC QA records and shall be maintained in accordance with approved instructions.

## 10. ATTACHMENTS

- 10.1 *Table 1.* from U. S. Atomic Energy Commission Regulatory Guide 1.86
- 10.2 MDA Calculations for Scanning
- 10.3 Unrestricted Release Survey Record
- 10.4 ConveyORIZED Monitor Pre-Operational Response Check Form
- 10.5 Nickel Unrestricted Release Shipment Summary

# **Attachments**

**ATTACHMENT 1. TABLE 1. FROM REGULATORY GUIDE 1.86**

*Table 1. from U. S. Atomic Energy Commission Regulatory Guide 1.86*

TABLE I

ACCEPTABLE SURFACE CONTAMINATION LEVELS

NUCLIDE <sup>a</sup>	AVERAGE <sup>b c</sup>	MAXIMUM <sup>b d</sup>	REMOVABLE <sup>b e</sup>
U-nat, U-235, U-238, and associated decay products	5,000 dpm $\alpha$ /100 cm <sup>2</sup>	15,000 dpm $\alpha$ /100 cm <sup>2</sup>	1,000 dpm $\alpha$ /100 cm <sup>2</sup>
Transuranics, Ra-226, Ra-228, Th-230, Th-228, Pa-231, Ac-227, I-125, I-129	100 dpm /100 cm <sup>2</sup>	300 dpm /100 cm <sup>2</sup>	20 dpm /100 cm <sup>2</sup>
Th-nat, Th-232, Sr-90, Ra-223, Ra-224, U-232, I-126, I-131, I-133	1,000 dpm /100 cm <sup>2</sup>	3,000 dpm /100 cm <sup>2</sup>	200 dpm /100 cm <sup>2</sup>
Beta-gamma emitters (nuclides with decay modes other than alpha emission or spontaneous fission) except Sr-90 and others noted above.	5,000 dpm $\beta$ - $\gamma$ /100 cm <sup>2</sup>	15,000 dpm $\beta$ - $\gamma$ /100 cm <sup>2</sup>	1,000 dpm $\beta$ - $\gamma$ /100 cm <sup>2</sup>

<sup>a</sup> Where surface contamination by both alpha- and beta-gamma-emitting nuclides exists, the limits established for alpha- and beta-gamma-emitting nuclides should be applied independently.

<sup>b</sup> As used in this table, dpm (disintegrations per minute) means the rate of emission by radioactive material as determined by correcting the counts per minute observed by an appropriate detector by background, efficiency, and geometric factors associated with the instrumentation.

<sup>c</sup> Measurements of average contaminant should not be averaged over more than 1 square meter. For objects of less surface area, the average should be derived for each object.

<sup>d</sup> The maximum contamination level applied to an area of not more than 100 cm<sup>2</sup>.

<sup>e</sup> The amount of removable radioactive material per 100 cm<sup>2</sup> of surface area should be determined by wiping that area with dry filter or soft absorbent paper, applying moderate pressure, and assessing the amount of radioactive material on the wipe with an appropriate instrument of known efficiency. When removable contamination on objects of less surface area is determined, the pertinent levels should be reduced proportionally and the entire surface should be wiped.

**ATTACHMENT 2. MDA CALCULATIONS FOR SCANNING**

*Table of MDA Calculations for Scanning*

177 Friskers with Pancake GM Probes (44-9)						
Scan Rate (in/sec)	Probe Width (in)	Count Time, T <sub>s</sub> (min)	Efficiency	Bkg, R <sub>b</sub> (cpm)	Bkg Time, T <sub>b</sub> (min)	MDA (dpm)
2	1.9	0.0158	12.5%	50	1	1490.72
2	1.9	0.0158	12.5%	100	1	2108.20
2	1.9	0.0158	12.5%	150	1	2582.00
2	1.9	0.0158	12.5%	200	1	2981.44
2	1.9	0.0158	12.5%	250	1	3333.35
2	1.9	0.0158	12.5%	300	1	3651.50
25 sec Static		0.4200	12.5%	100	1	483.96
		0.4200	12.5%	150	1	592.72
		0.4200	12.5%	200	1	684.42
		0.4200	12.5%	250	1	765.20
		0.4200	12.5%	300	1	838.24
2224 Frisker with 43-89						
Scan Rate (in/sec)	Probe Width (in)	Count Time, T <sub>s</sub> (min)	Efficiency	Bkg, R <sub>b</sub> (cpm)	Bkg Time, T <sub>b</sub> (min)	MDA (dpm)
Probe 43-89 for alpha						
2	2.95	0.0246	20.2%	5	1	235.12
2	2.95	0.0246	20.2%	10	1	332.50
2	2.95	0.0246	20.2%	15	1	407.23
2	2.95	0.0246	20.2%	20	1	470.23
2	2.95	0.0246	20.2%	25	1	525.74
Static Count		0.4200	20.2%	5	1	66.97
		0.4200	20.2%	10	1	94.70
		0.4200	20.2%	15	1	115.99
		0.4200	20.2%	20	1	133.93
		0.4200	20.2%	25	1	149.74
Probe 43-89 for beta						
2	2.95	0.0246	9.2%	50	1	1625.41
2	2.95	0.0246	9.2%	100	1	2298.67
2	2.95	0.0246	9.2%	150	1	2815.29
2	2.95	0.0246	9.2%	200	1	3250.81
2	2.95	0.0246	9.2%	250	1	3634.52
2	2.95	0.0246	9.2%	300	1	3981.42
2	2.95	0.0246	9.2%	400	1	4597.35
2	2.95	0.0246	9.2%	500	1	5139.99
Static Count		0.4200	9.2%	100	1	654.70
		0.4200	9.2%	200	1	925.89
		0.4200	9.2%	300	1	1133.98
		0.4200	9.2%	400	1	1309.40
		0.4200	9.2%	500	1	1463.96

The formula used for the above calculations is on the right. The value for "m" is 1.645 which corresponds to a confidence level of 95%.

$$MDA = \frac{2m \sqrt{\frac{R_b}{T_s} + \frac{R_b}{T_b}}}{\text{eff. (counting instrument)}}$$

### ATTACHMENT 3. UNRESTRICTED RELEASE SURVEY RECORD

<i>Customer Name:</i>	
<i>Customer ID Number:</i>	
<i>MSC Job Number(s):</i>	

Date:	
Time:	
μR/hr background:	

**RELEASE LIMITS**

Survey Instrument	Surface Contamination Release Limits			Removable Release Limits (dpm/100 cm <sup>2</sup> )
	Plane	Point <sup>f</sup>	Max. <sup>g</sup>	
Pancake G-M				<i>Removable Release Limits are applied to 100 cm<sup>2</sup> wipe samples. Wipe samples are analyzed on low level laboratory counting systems or equivalent.</i>
100 cm <sup>2</sup> α+β scintillator				
CWM-10 conveyORIZED monitor				
100 cm <sup>2</sup> α+β gas proportional				

**SURFACE CONTAMINATION SURVEY TECHNICIANS and INSTRUMENTATION**

Technician	Instrument	Serial Number	Cal. Due Date	Inst. Bkg.	Daily Response

**SURFACE CONTAMINATION SURVEY RESULTS**

MSC Job Number/ Identification Number	Unrestricted Release Holding Container Number/Net Weight Passed	Net Weight Rejected/ Waste Box Number	Tech Initials

**REMOVABLE CONTAMINATION ANALYSIS RESULTS SUMMARY**

Number of Samples	Number Above Limits	Analysis Attached	Reviewer Initials

**RELEASE SUMMARY**

Total Net Weight Released	Release Bin Number	Date Released	Total Net Weight Rejected	Date Waste

**Review:** \_\_\_\_\_

<sup>f</sup> Point source (point) considerations may be evaluated and applied only by qualified survey personal.  
<sup>g</sup> Maximum (max.) surface contamination evaluation will be performed by the Survey Manager or Designee.

**ATTACHMENT 4.**

**CONVEYORIZED MONITOR PRE-OPERATIONAL RESPONSE CHECK FORM**

<i>Date:</i>	<i>Technician:</i>
<i>Instrument:</i>	<i>Instrument S/N:</i>
<i>Calibration Date:</i>	
<i>Calibration Isotope(s)/Configuration/Geometry:</i>	

<i>Background</i>			
<i>Instrument</i>	<i>Reading</i>	<i>S/N</i>	<i>Cal. Due</i>
Ludlum Model 19 MicroR			
Ludlum Model 44-9 G-M			

<i>Diagnostics</i>			
<i>Item</i>	<i>YES</i>	<i>NO</i>	<i>Initial</i>
All indicators/switches responding?			

<i>Alarm Response Check</i>			
<i>Upper Detector Array Height:</i>		<i>Count Time (seconds):</i>	
<i>Alarm Level Activity Setting:</i>			
<i>Check Source Isotope:</i>	<i>Activity:</i>	<i>Configuration:</i>	
<i>Response Check Data</i>			
<i>Group</i>	<i>Trial</i>	<i>Alarm</i>	<i>Comments</i>
1 & 2			
3 & 4			
5 & 6			
7 & 8			
9 & 10			
<i>Alarm History Printout is attached.</i>		<i>Signed:</i>	



## TITLE: UNRESTRICTED RELEASE CALCULATIONS

### 1.0 PURPOSE

The purpose of this Instruction is to outline the methods and calculations utilized to determine unrestricted release limits for particular materials.

### 2.0 SCOPE

This procedure details the calculations and methods utilized to determine unrestricted release limits for Manufacturing Sciences Corporation, Kerr Hollow Road Facilities. The release limits take into consideration the hard-to-detect isotopes.

### 3.0 REFERENCES

- 3.1 Brookhaven National Laboratory, NNDC. *NuDat Database*, January 1996.
- 3.2 Erdtman, G. and Soyka, W. *The Gamma Rays of the Radionuclides*, ISBN 0-89573-022-7, 1979.
- 3.3 U. S. Atomic Energy Commission. *Regulatory Guide 1.86, Termination of Operating Licenses for Nuclear Reactors*, Retyped by U.S. EPA, August 1997.
- 3.4 Manufacturing Sciences Corporation. *Radioactive Material License R-01078-L00 submittal*.

### 4.0 DEFINITIONS

- 4.1 *Surface Contamination Release Limit* – the allowable surface activity per unit area. The limit is taken from Regulatory Guide 1.86, Table 1 (Attachment 1.) and actual isotopic considerations relative to the material in question.
- 4.2 *Decay Progeny* – in this procedure refers to either alpha particles, beta particles, and/or gamma photons. Particles and/or photons may not be considered in calculations if they are sufficiently low in probability or average energy.
- 4.3 *Unrestricted Release* – an item or items that have met the criteria relative to *surface, removable, and maximum* activity established by Regulatory Guide 1.86. The item(s) is/are considered to be “clean” and may be released to non-radiologically controlled areas for unrestricted use.
- 4.4 *Hard-to-detect* – radioactive isotopes which do not emit readily detectable decay progeny. Some examples are  $^{55}\text{Fe}$ ,  $^{63}\text{Ni}$ , and  $^{241}\text{Pu}$ .
- 4.5 *Inaccessible Area* – an area on an item considered for unrestricted release that cannot be surveyed for unrestricted release with standard survey meters or

techniques. The Survey Manager or Designee shall determine unrestricted release status for those items identified with inaccessible areas.

## 5.0 PRECAUTIONS

None.

## 6.0 PREREQUISITES

6.1 Anyone designated to perform unrestricted release calculations must be technically capable. Determinations of technical competence will be made by the RSO.

6.2 The individual's role proficiency should show authorization to perform and to review unrestricted release calculations.

## 7.0 RESPONSIBILITIES

### 7.1 Radiation Safety Officer (RSO)

7.1.1 The RSO or designee will establish appropriate unrestricted release limits. These established limits will take into account isotopic contamination considerations, material geometry considerations if necessary, material composition considerations if necessary, and any other applicable considerations which may be determined as necessary.

7.1.2 The RSO is responsible for all unrestricted release of materials from MSC.

7.1.3 The RSO may designate responsibilities and/or certain tasks of the unrestricted release program.

## 8.0 PROCEDURES FOR CALCULATIONS OF RELEASE LIMITS

### 8.1 General Process for Determination of Release Limits

8.1.1 Receipt of isotopic information from client, usually shipping manifest

8.1.2 Isotopic information review and calculations based on isotopes to determine suitable release limit

8.1.3 Request for additional analysis if necessary

8.1.4 Review of information on material, i.e. composition, size, shape, and disposition

8.1.5 Review of additional analysis and subsequent adjustments to isotopic ratios

8.1.6 Issuance of release limit for material, specific to instrumentation

8.2 Isotopic Considerations

8.2.1 The Surface Contamination Release Limit derived from Regulatory Guidance 1.86, Table I. The table is broken down into four groups, each with different criteria. The table lists the average and maximum allowable surface and maximum removable activity levels for the different groups.

Special case by case considerations will be given to source terms which have significant amounts (>25% of the total activity for a particular batch) of transuranics, thoriums, radiums, <sup>125</sup>I, <sup>126</sup>I, <sup>129</sup>I, <sup>131</sup>I, <sup>133</sup>I, <sup>227</sup>Ac, <sup>231</sup>Pa, <sup>232</sup>U, or <sup>90</sup>Sr. These considerations will involve ratios and scaling to accommodate the lower allowable surface contamination and removable contamination levels.

8.2.2 All isotopes in a particular source term are ratioed with respect to total activity.

The formula used for determining ratios:

$$\text{Ratio for isotope (I}_r\text{)} = \text{activity for isotope} \div \text{total activity}$$

8.2.3 The ratios (I<sub>r</sub>) of the hard-to-detects are summed. This percentage is removed from the allowable surface activity (from Reg. Guide 1.86, Table I) to determine a scaled allowable surface activity.

*Example:* A shipment has manifest 25% of the total activity as <sup>55</sup>Fe. Applying this to 15,000 dpm per 100 cm<sup>2</sup> would result in a scaled maximum of 11,250 dpm per 100 cm<sup>2</sup>.

$$\text{Allowable} - \% \text{ hard-to-detect} = \text{scaled allowable activity}$$

(15,000)                      (25%)                      (11,250)

*Example:* A shipment has manifest 10% <sup>63</sup>Ni and 10% <sup>55</sup>Fe. Applying this to an allowable average of 5,000 dpm/100 cm<sup>2</sup> over one meter would result in a scaled average surface activity of 4,000 dpm/100 cm<sup>2</sup> over one meter.

$$\text{Allowable} - \% \text{ hard-to-detect} = \text{scaled allowable activity}$$

(5,000)                      (20%)                      (4,000)

The hard-to-detects currently recognized are:

- <sup>3</sup>H ..... soft beta
- <sup>14</sup>C ..... soft beta
- <sup>35</sup>S ..... soft beta
- <sup>51</sup>Cr ..... very low intensity gamma

<sup>55</sup> Fe .....	soft beta
<sup>59</sup> Ni .....	very low intensity/energy gamma
<sup>63</sup> Ni .....	soft beta
<sup>106</sup> Ru .....	soft beta
<sup>129</sup> I .....	soft beta
<sup>241</sup> Pu.....	soft beta

Some of the above listed isotopes are detectable, but due to low efficiencies they are considered otherwise.

#### 8.2.4 Considerations for Isotopes with Half-lives of Approximately 30 Days or Less

Often, isotopes with relatively short half-lives will be manifest on incoming shipments. The shipment will be evaluated on how long it has been in storage before processing and the origin of the materials. If sufficient storage time for approximately 10 half-lives is evident, the isotopes *may* be removed from the source term.

Examples of isotopes which *may* be considered for removal:

<sup>51</sup> Cr .....	27.7 days
<sup>67</sup> Cu .....	2.58 days
<sup>95</sup> Nb .....	34.98 days
<sup>103</sup> Ru .....	39.26 days
<sup>141</sup> Ce .....	32.5 days

This list is not all inclusive as there other isotopes which may be considered for removal after half-life considerations.

#### 8.2.5 Equilibrium Decay Chain Considerations

Isotopes which in reality exist as decay chains in equilibrium and not as single isotopes will occasionally be manifest alone. When this occurs, daughters and/or parents will be added to the source term and their respective progeny considered.

<sup>238</sup>U will have additional daughters present in equilibrium, unless the <sup>238</sup>U has been recently separated. <sup>90</sup>Sr will almost always exist with <sup>90</sup>Y present in equilibrium. Natural uranium has many daughters present in equilibrium.

These equilibrium chains exist due to the relatively short half-life of the daughter with respect to the parent, so to exist alone would require recent separation of the parent which is a very remote possibility in most cases.

### 8.2.6 Isotopic Decays, Intensities, and Energies

Isotopic decays, their respective decay progeny, their respective progeny intensities, and their respective progeny energies are considered with respect to total activity.

This relationship is determined for alpha particles, beta particles, and gamma photons for each isotope present. The following formulae are used to express the relationships:

$$S_p = (I_r \cdot I_p)$$

where  $S_p$  = source term probability of a particular decay progeny from a particular isotope

$I_r$  = isotopic activity ratio

$I_p$  = probability for a particular decay progeny to occur for a particular isotope

$$E_w = (I_r \cdot I_p \cdot I_e) \div \Sigma(I_r \cdot I_p \cdot I_e)$$

where  $E_w$  = isotope specific, weighted energy contribution for a particular decay progeny of the source term in question

$I_r$  = isotopic activity ratio

$I_p$  = probability for a particular decay progeny to occur for a particular isotope

$I_e$  = average energy of particular decay progeny

The summation of the  $S_p$  establishes a particular source term's decay progeny intensity and the summation of the relative  $E_w$  establishes a particular source term's decay progeny average energy.  $S_p$  is used in final release limit calculations, but  $E_w$  is for information only at present.

### 8.2.7 Requests for Additional Analysis

In cases where a high percentage of the hard-to-detect isotopes are manifest or the manifest is not trusted for some reason, additional sampling and analysis is warranted. Additional sampling and analysis allows for determination that the shipping manifest is accurate or inaccurate. Accuracy is determined with activity ratios from the analyses and compared with the activity ratios from the manifest. Ratios are used because it is not realistic to measure all of the activity present in a shipment. In situations where the analysis and the manifest do not correlate, a new source term is developed from the sampling and analysis information.

Health Physics Technicians and/or Survey Technicians typically conduct the sampling. Analysis is performed by the MSC Analytical Laboratory and/or sent out for outside determinations.

### 8.3 Instrumentation Considerations

#### 8.3.1 Detector Type

Different detectors may be more efficient at detecting different radiations. For example, NaI detectors are much more efficient at detecting gamma radiation than beta radiation and gas proportional detectors are more efficient at detecting beta radiation than gamma radiation. These differences require considerations with regard to unrestricted release.

MSC primarily utilizes three different detectors for unrestricted release surface contamination surveys. These are (1) 44-9 Ludlum pancake detectors, (2) 43-89 Ludlum 100 cm<sup>2</sup> alpha/beta detectors, and (3) 600+ cm<sup>2</sup> plastic scintillation detectors.

The 44-9 is efficient for alpha and beta radiation, but not very efficient for gamma radiation. If gamma radiation is considered with this probe/detector arrangement, gamma efficiencies will be established prior. Only alpha and beta radiation is considered for this detector, unless otherwise noted.

The 43-89 is efficient for alpha and beta radiation, but will detect gamma much more efficiently than the 44-9. It is considered to be similar to the 600+ cm<sup>2</sup> plastic scintillation detectors and the same scaling is utilized for both, with the exception of alpha radiation. The 43-89 also contains a ZnS scintillation material and is efficient for alpha.

The 600+ cm<sup>2</sup> plastic scintillation detectors are part of a conveyORIZED system. These detectors are efficient for beta and gamma radiation. They have no efficiency for alpha radiation.

It must be noted that actual instrument efficiency is utilized when converting cpm from the instrument to dpm. The delectability considerations above are for establishing release limits for the types of radiation present in a particular source term.

#### 8.3.2 Detector Size

Detector probe area is also a necessary consideration with regard to unrestricted release. Limits are typically considered over 100 cm<sup>2</sup> and many probes are not 100 cm<sup>2</sup> in size. Scaling is done to adjust for probe areas.

*Example 1:* A Ludlum Model 44-9 (pancake probe) has an active window of 15 cm<sup>2</sup>. This is 15% of 100 cm<sup>2</sup>. Applying this to the average allowable of 5,000 dpm/cm<sup>2</sup> gives a result of 750 dpm/probe area.

If the scaled allowable of 4,000 dpm/100 cm<sup>2</sup> from the example above is used, the result is 600 dpm/probe area.

*Example 2:* A probe area of 600 cm<sup>2</sup> is 600% of 100 cm<sup>2</sup>. Applying this to the average allowable of 5,000 dpm/100 cm<sup>2</sup> gives a result of 30,000 dpm per probe area. But, due to the size of the probe area (600 cm<sup>2</sup>) and the fact that it is larger than 100 cm<sup>2</sup>, the average allowable is set no greater than the 15,000 dpm maximum allowable. This is somewhat more conservative than probe area considerations will allow.

#### 8.4 Material Geometry and Composition Considerations

Material geometry must be considered as surfaces must be accessible. Material with areas which are not accessible must be considered on a case by case basis. Considerations will be given regarding process knowledge, material origin, suspected contaminants, and defensibility of those considerations.

Material composition may effect release limit considerations. Considerations may be allowed for contributions of detectable activity which passed through the material from the side not being assayed. This would apply to low density, thin materials such as aluminum sheet or plate. Material composition will be considered on a case by case basis.

#### 8.5 Surface Contamination Release Limit Issuance

After the release limits are scaled for hard-to-detects, adjustments are made for source term probabilities ( $S_p$ ) and for the particular instrumentation to be used. Instrumentation considerations are based on a particular source term's decay progeny and an instrument's ability to detect certain decay progeny. A final release limit is determined.

The Surface Contamination Release Limit is issued as part of the Unrestricted Release Survey Record. The information is supplied by the individual performing the calculations.

It should be noted that under typical circumstances, contamination distribution is considered to be in the form of a plane source, i.e. the contamination is distributed relatively evenly over the surface of the material. In this situation, probe areas must be considered such as in the case of the 44-9 G-M. The following limits are calculated using the assumption of plane source contamination geometry. Point source contamination geometry (i.e. the contamination distribution is not

relatively uniform and is confined to a small area of less than approximately 1 cm<sup>2</sup>) limits are not dependent on probe area and thus the probe area is not considered.

#### 8.5.1 Limits for the 44-9 Pancake G-M (Plane Sources)

Active area considerations for the detector establish a maximum average allowable activity per probe/detector area of 750 dpm (8.3.2 *Example 1.*).

Scaling for hard-to-detects is taken from active area considerations (750 dpm).

Detection capability considerations of this detector show an efficiency for alpha and beta radiation, but not for gamma radiation, thus allowing only the alpha and beta components to be considered.

The  $\Sigma S_p$  alpha component (not to include any hard-to-detect) is added to the  $\Sigma S_p$  beta component (not to include any hard-to-detect). The product of this figure, the detector area scaling, and the % hard-to-detect is the release limit for this probe. The formula is:

$$\text{Release Limit} = (\Sigma S_p \text{ alpha} + \Sigma S_p \text{ beta}) \cdot (750 \text{ dpm} \cdot \% \text{ hard-to-detect})$$

#### 8.5.2 Limits for the 600+ cm<sup>2</sup> Plastic Scintillation Detector (Plane Sources)

Active area considerations for the detector establish a maximum average allowable activity per probe/detector area of 15,000 dpm (8.3.2 *Example 2.*).

Scaling for hard-to-detect is taken from active area considerations (15,000 dpm).

Detection capability considerations of this detector show an efficiency for beta and gamma radiation, but not for alpha radiation, thus allowing only the beta and gamma components to be considered.

The  $\Sigma S_p$  beta component (not to include any hard-to-detect) is added to the  $\Sigma S_p$  gamma component (not to include any hard-to-detect). The product of this figure, the detector area scaling, and the % hard-to-detect is the release limit for this probe/detector. The formula is:

$$\text{Release Limit} = (\Sigma S_p \beta + \Sigma S_p \gamma) \cdot (15,000 \text{ dpm} \cdot \% \text{ hard-to-detect})$$

### 8.6 Release Limit Issuance for Removable Contamination

Removable contamination levels will be evaluated with low level counting instrumentation located in the MSC Analytical Laboratory. Limits will be determined by scaling calculations considering hard-to-detect isotopes from the allowable limits of Reg. Guide 1.86 for removable contamination. A dpm/100 cm<sup>2</sup> limit will be derived. Removable contamination wipes will cover 100 cm<sup>2</sup>.

*Example:* A shipment manifest with 10% <sup>3</sup>H and having a source term which is not "special case," thus allowing a maximum removable contamination activity level of 1,000 dpm/100 cm<sup>2</sup> for β/γ emitters would be scaled back to 900 dpm/100 cm<sup>2</sup> for removable contamination activity.

### 8.7 Example of Complete Calculation (Plane Source)

A shipment arrives and the manifest is faxed to the appropriate personnel. The manifest shows the following:

Co-60 .....	2.35 mCi
Cs-137 .....	1.34 mCi
Fe-55 .....	0.81 mCi
 Total .....	 4.5 mCi

From this, the ratios are calculated. Reporting is typically in percentage. The ratios for this source term are:

Co-60 .....	52.2 %
Cs-137 .....	29.8 %
Fe-55 .....	18 %

From this source term, 18 % (from <sup>55</sup>Fe) must be considered hard-to-detect or *not detectable* and therefore must be considered by scaling.

Scaling is taken from the instrument *maximum* average allowable activity. For the 44-9 pancake G-M, the *maximum* average allowable average activity is 750 dpm (this comes from probe active area considerations as shown in 8.3.2 *Example 1.*). 18% from 750 dpm leaves 615 dpm. For the conveyORIZED system with 600+ cm<sup>2</sup> detectors, the instrument *maximum* average allowable activity is 15,000 dpm (8.3.2, *Example 2.*). 18% from 15,000 dpm leaves 12,300 dpm.

The source term decay progeny probability is determined. Each contributing isotope is considered (Hard-to-detects are considered to be non-contributing).

<sup>60</sup>Co has three primary decay progeny: two gammas and one beta

beta .....	95.8 keV average.....	100%
gamma .....	1173.2 keV .....	100%
gamma .....	1332.5 keV .....	100%

The beta contribution to the source term is:

I<sub>r</sub> for <sup>60</sup>Co = 52.2%  
I<sub>p</sub> for <sup>60</sup>Co beta = 100%

$$S_p = (52.5\%) \cdot (100\%) = 52.2\%$$

The gamma contribution to the source term is:

$$\frac{1173.2 \text{ gamma:}}{I_p \text{ for } ^{60}\text{Co} = 100\%}$$

$$S_p = (52.2\%) \cdot (100\%) = 52.2\%$$

$$\frac{1332.5 \text{ gamma:}}{I_p \text{ for } ^{60}\text{Co} = 100\%}$$

$$S_p = (52.2\%) \cdot (100\%) = 52.2\%$$

Thus the total gamma contribution is 104.4% (52.2 + 52.2).

<sup>137</sup>Cs has two primary decay progeny: one gamma and one beta

beta .....	156.8 keV average.....	94.6%
gamma .....	661.6 keV .....	90%

The beta contribution to the source term is:

$$I_r \text{ for } ^{137}\text{Cs} = 29.8\%$$

$$I_p \text{ for } ^{137}\text{Cs} \text{ beta} = 94.6\%$$

$$S_p = (29.8\%) \cdot (94.6\%) = 28.19\%$$

The gamma contribution to the source term is:

$$I_p \text{ for } ^{137}\text{Cs} = 90\%$$

$$S_p = (29.8\%) \cdot (90\%) = 26.82\%$$

The summations of the probabilities for the beta and gamma constituents:

$$\Sigma S_p \text{ beta} = 52.2 \text{ (from } ^{60}\text{Co)} + 28.19 \text{ (from } ^{137}\text{Cs)} = 80.39\%$$

$$\Sigma S_p \text{ gamma} = 104.4 \text{ (from } ^{60}\text{Co)} + 26.82 \text{ (from } ^{137}\text{Cs)} = 131.22\%$$

This source term does not have any alpha constituents, but they would be calculated the same way.

The release limit for the 44-9 pancake G-M:

Since the 44-9 is not considered efficient for gamma, only the beta constituent is considered. From the hard-to-detect scaling, a *maximum* average activity of 615 dpm per probe/detector area has been determined. Following the formula in 8.1.1:

$$\text{Release Limit} = (\Sigma S_p \text{ alpha} + \Sigma S_p \text{ beta}) \cdot (615 \text{ dpm}) =$$

$$= (0 + 80.39\%) \cdot (615 \text{ dpm}) = 494.4 \text{ dpm or } \underline{494 \text{ dpm}}$$

The release limit for the 600+ cm<sup>2</sup> plastic scintillation detector:

This detector is efficient for gamma and beta radiation, thus the beta and gamma constituents are considered. From the hard-to-detect scaling, a *maximum* average activity of 12,300 dpm per probe/detector area has been determined. Following the formula in 8.1.2:

$$\begin{aligned} \text{Release Limit} &= (\Sigma S_p \text{ beta} + \Sigma S_p \text{ gamma}) \cdot (12,300 \text{ dpm}) = \\ &= (80.39\% + 131.22\%) \cdot (12,300 \text{ dpm}) = 26,028 \text{ dpm} \end{aligned}$$

Since this number is greater than 15,000 dpm, the release limit is set at 15,000 dpm for this detector.

## 8.8 Special Case Isotopic Source Terms

If a shipment manifest greater than 25% of transuranics, thoriums, radiums, <sup>125</sup>I, <sup>126</sup>I, <sup>129</sup>I, <sup>131</sup>I, <sup>133</sup>I, <sup>227</sup>Ac, <sup>231</sup>Pa, <sup>232</sup>U, <sup>90</sup>Sr, or any combination of the above, then it falls under the special case considerations.

The ratios of the special case isotopes will be combined into like groups as broken down by *Table I* of Reg. Guide 1.86 (Attachment 1). Their contributions to total source term are determined. Release limits will be established to consider their contribution.

## 8.9 <sup>238</sup>U and <sup>235</sup>U Source Term Considerations and Enrichment Issues

Enrichment issues are more of a concern for license limit requirements for SNM than they are for unrestricted release monitoring. Due to the SNM site inventory restrictions, incoming materials may be sampled to verify isotopic source terms.

Source terms manifest with uranium source terms, either natural uranium, depleted uranium, or enriched uranium, will be evaluated with the same methods as described above.

### Example of Enriched Source Term Calculation

A shipment arrives and the manifest is faxed to the appropriate personnel. Analysis of the manifest shows the following ratios:

U-238 .....	90 %
U-235 .....	10 %

This source term would be considered enriched.

Since <sup>238</sup>U is in equilibrium with <sup>234</sup>Th, <sup>234m</sup>Pa, and <sup>234</sup>U, the source term is adjusted to the following:

U-238 .....	24.3 %
Th-234 .....	24.3 %
Pa-234m .....	24.3 %
U-234 .....	24.3 %

U-235..... 2.7 %

When the decay progeny of the source term is evaluated, the following is determined for this source term:

Alpha decay ..... 51 % @ 4481 keV average  
Beta decay ..... 41.6 % @ 497.1 keV average  
Gamma decay..... 2 % @ 180 keV average

The release limit for the 44-9 pancake G-M (Plane Source):

Since the 44-9 is not considered efficient for gamma, only the alpha and beta constituents are considered. Following the formula in 8.1.1:

$$\begin{aligned} \text{Release Limit} &= (\Sigma S_p \text{ alpha} + \Sigma S_p \text{ beta}) \cdot (750 \text{ dpm}) = \\ &= (51 + 41.6\%) \cdot (750 \text{ dpm}) = 694.5 \text{ dpm or } \underline{694 \text{ dpm}} \end{aligned}$$

The release limit for the 600+ cm<sup>2</sup> plastic scintillation detector:

This detector is efficient for gamma and beta radiation, thus the beta and gamma constituents are considered. Following the formula in 8.1.2:

$$\begin{aligned} \text{Release Limit} &= (\Sigma S_p \text{ beta} + \Sigma S_p \text{ gamma}) \cdot (12,300 \text{ dpm}) = \\ &= (41.6\% + 2.02\%) \cdot (15,000 \text{ dpm}) = \underline{6543 \text{ dpm}} \end{aligned}$$

## 9.0 RECORDS

Release Limit calculations will be part of the Unrestricted Release Record. This record shall consist of the Unrestricted Release Survey Record, *materials isotopic evaluations*, removable contamination wipes analysis data, and conveyORIZED survey monitor data if applicable. All Unrestricted Release Records are MSC QA records and shall be maintained in accordance with approved instructions.

## 10. ATTACHMENTS

Table 1. from U. S. Atomic Energy Commission Regulatory Guide 1.86

# **Attachments**

## ATTACHMENT 1.

**Table 1. from U. S. Atomic Energy Commission  
Regulatory Guide 1.86**

TABLE I

ACCEPTABLE SURFACE CONTAMINATION LEVELS

NUCLIDE <sup>a</sup>	AVERAGE <sup>b c</sup>	MAXIMUM <sup>b d</sup>	REMOVABLE <sup>b e</sup>
U-nat, U-235, U-238, and associated decay products	5,000 dpm $\alpha$ /100 cm <sup>2</sup>	15,000 dpm $\alpha$ /100 cm <sup>2</sup>	1,000 dpm $\alpha$ /100 cm <sup>2</sup>
Transuranics, Ra-226, Ra-228, Th-230, Th-228, Pa-231, Ac-227, I-125, I-129	100 dpm /100 cm <sup>2</sup>	300 dpm /100 cm <sup>2</sup>	20 dpm /100 cm <sup>2</sup>
Th-nat, Th-232, Sr-90, Ra-223, Ra-224, U-232, I-126, I-131, I-133	1,000 dpm /100 cm <sup>2</sup>	3,000 dpm /100 cm <sup>2</sup>	200 dpm /100 cm <sup>2</sup>
Beta-gamma emitters (nuclides with decay modes other than alpha emission or spontaneous fission) except Sr-90 and others noted above.	5,000 dpm $\beta$ - $\gamma$ /100 cm <sup>2</sup>	15,000 dpm $\beta$ - $\gamma$ /100 cm <sup>2</sup>	1,000 dpm $\beta$ - $\gamma$ /100 cm <sup>2</sup>

<sup>a</sup> Where surface contamination by both alpha- and beta-gamma-emitting nuclides exists, the limits established for alpha- and beta-gamma-emitting nuclides should be applied independently.

<sup>b</sup> As used in this table, dpm (disintegrations per minute) means the rate of emission by radioactive material as determined by correcting the counts per minute observed by an appropriate detector by background, efficiency, and geometric factors associated with the instrumentation.

<sup>c</sup> Measurements of average contaminant should not be averaged over more than 1 square meter. For objects of less surface area, the average should be derived for each object.

<sup>d</sup> The maximum contamination level applied to an area of not more than 100 cm<sup>2</sup>.

<sup>e</sup> The amount of removable radioactive material per 100 cm<sup>2</sup> of surface area should be determined by wiping that area with dry filter or soft absorbent paper, applying moderate pressure, and assessing the amount of radioactive material on the wipe with an appropriate instrument of known efficiency. When removable contamination on objects of less surface area is determined, the pertinent levels should be reduced proportionally and the entire surface should be wiped.

TITLE: LABORATORY ANALYSIS OF NICKEL FOR TECHNETIUM-99 AND URANIUM UTILIZING LIQUID SCINTILLATION PDA METHODS

1.0 PURPOSE

The purpose of this instruction is to describe the method of analysis of contaminated Ni after the removal of Tc-99 and Uranium isotopes by electro-refining process to a level that the Nickel may be free-released for use in the private sector.

2.0 SCOPE

This procedure and method applies to analysis of nickel material obtained after electro-refining.

3.0 REFERENCES

3.1 Reference Documents

- a. Sampling and Analysis Plan
- b. Packard Liquid Scintillation Analyzer Instruction Manual
- c. Oxford Gamma Spectrometry Analyzer Instruction Manual
- d. Risk Assessment Analysis

4.0 DEFINITIONS

- 4.1 RFA: Request for Analysis form
- 4.2 COC: Chain of Custody form

5.0 PRECAUTIONS

- 5.1 Use caution when handling chemicals for dissolution of nickel metal. Safety glasses, face shield and protective clothing required.
- 5.2 Safety glasses or Face shield required when drilling metal ingot.

6.0 PREREQUISITES

- 6.1 Surface of metal should be cleaned prior to obtaining drillings for analysis.

7.0 RESPONSIBILITIES

- 7.1 The process engineer is responsible for collection of cathode material and transfer of the material to the laboratory, along with a completed RFA/COC form.
- 7.2 The Laboratory Manager or his designee is responsible for receiving the material from the process area.

## 8.0 PROCEDURE

All metallic samples collected during either phase of the study will be treated in the same manner. Alpha analysis will be utilized for uranium determination. Beta analysis will be utilized for Tc-99 determination; betas originating from uranium prompt daughters will be considered in final Tc-99 results.

- 8.1 Transfer the 20 grams of the nickel sample to a large beaker and slowly add the 125 ml of the nitric acid. (CAUTION- this reaction generates heat and NO<sub>3</sub> fumes). Gently heat on a hot plate at low temperature to increase dissolution of the nickel. If needed, add approximately 1 ml of hydrogen peroxide drop wise to the solution. Gently swirl the solution to mix the material.
- 8.2 After the nickel has dissolved, continue gentle heat to slowly evaporate the sample to reduce the nitrate concentration. Cool the solution and transfer the material to a 250 ml volumetric flask and dilute to the mark with distilled water. Stopper the flask and mix thoroughly.
- 8.3 Remove 2 ml and add to a liquid scintillation vial containing 12-15 ml of cocktail. Prepare a duplicate in the same manner and mix the sample thoroughly.
- 8.4 Place the samples, along with a reagent blank and standards, in the Packard Liquid Scintillation Analyzer and start the count. Use the counting parameters determined using the Alpha/Beta standards.
- 8.5 Complete the reporting form and transfer a copy to the appropriate staff members. The tabulated form and the original data will be kept on file in the laboratory.

## 9.0 RECORDS

Chain of Custody forms, Original Data Sheets and Completed Analysis Forms will be maintained as quality records and archived in accordance with MSC's Records Management program.

## 10.0 ATTACHMENTS

None.

TITLE: VOLUMETRIC SAMPLING OF REFINED NICKEL

1.0 PURPOSE

The purpose of this Work Instruction is to provide guidelines for the sampling of refined nickel ingots.

2.0 SCOPE

This Work Instruction details the methodology for volumetric sampling of ingots, specifically nickel ingots, and the procedures for obtaining volumetric samples of ingots.

3.0 REFERENCES

Cole, Les and Auxier and Associates, Inc. *Risk Analysis: Nickel Contaminated with <sup>99</sup>Tc and Uranium*, October 1998.

Manufacturing Sciences Corporation Work Instruction. *Unrestricted Survey of Materials*, July 1998.

Manufacturing Sciences Corporation. *Sampling and Analysis Plan for Nickel Recycle*, November 1998.

4.0 DEFINITIONS

*Ingot* means refined metal solid directly from either a melt furnace operation or an electro-refining operation.

*Unrestricted Release* means an item or items that have met the criteria relative to surface, removable, and maximum activity established by Regulatory Guide 1.86. The item(s) is/are considered to be "clean" and may be released to non-radiologically controlled areas for unrestricted use. Volumetric criteria will also be a consideration with regard to refined nickel.

5.0 PRECAUTIONS

Eye protection as a minimum should be worn when performing volumetric sampling.

6.0 PREREQUISITES

Performance of volumetric sampling of nickel ingots shall be conducted by approved personnel. Approval will be given by the RSO or Designee.

7.0 RESPONSIBILITIES

7.1 Radiation Safety Officer (RSO)

7.1.1 The RSO is responsible for all free release of materials from MSC.

7.1.2 The RSO may designate responsibilities and/or certain tasks of the free release program.

7.2 MSC Analytical Laboratory will perform volumetric analysis.

## 8.0 PROCEDURES FOR VOLUMETRIC SAMPLING OF REFINED NICKEL

8.1 The sampling schedule will be determined by Project Manager.

8.2 Ensure the surface of the electro-refined nickel ingot (also referred to as "finished cathode") is clean and dry. Ingot should be clearly identified prior to sampling.

8.3 Volumetric sampling should occur after the removable contamination survey has been completed and prior to unrestricted release. All materials will remain in radiologically controlled areas until a surface and removable contamination survey has been completed.

8.4 A volumetric sample will be collected using a carbide tipped or equivalently hard drill bit to reduce the contribution of material from the bit to the sample. The bit diameter shall be large enough to ensure an adequate sample volume is achieved.

8.5 The drill bit shall be free of removable and surface contamination. If contamination is present, the bit shall not be used for sampling.

8.6 The sample will consist of drill shavings associated with a drilling completely through the ingot. All shavings will be collected. Each drilling will represent one sample.

8.7 After sample collection, the sample should be labeled and packaged appropriately to minimize cross contamination potential.

8.8 Submit the sample(s) to the laboratory for evaluation following appropriate laboratory procedures for sample submission.

## 9.0 RECORDS

Sampling information and analysis will be part of the free release record and shall be maintained in accordance with appropriate MSC Instructions, Instruction Guides, and/or Procedures. Records of all measurements and evaluations will be properly identified and maintained for any regulatory or customer review.

## 10. ATTACHMENTS

None.