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**CP5-ND-2500/FR0**

# Method Manual for Qualitative Sodium Iodide Scans at the Paducah Gaseous Diffusion Plant, Paducah, Kentucky

FOUR RIVERS
NUCLEAR PARTNERSHIP, LLC

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CP5-ND-2500 FR0

# Method Manual for Qualitative Sodium Iodide Scans at the Paducah Gaseous Diffusion Plant, Paducah, Kentucky

Date Issued—11/25/2024

U.S. DEPARTMENT OF ENERGY
Office of Environmental Management

Prepared by
FOUR RIVERS NUCLEAR PARTNERSHIP, LLC,
Managing the
Deactivation and Remediation Project at the
Paducah Gaseous Diffusion Plant
Under Contract DE-EM0004895

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# APPROVALS

**Method Manual for
Qualitative Sodium Iodide Scans
at the Paducah Gaseous Diffusion Plant,
Paducah, Kentucky**

**CP5-ND-2500 FR0**

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# ACRONYMS

| | |
|---|---|
| CAP | Corrective Action Plan |
| CI | Criticality Incredible |
| D&R | Deactivation and Remediation |
| DOE | U.S. Department of Energy |
| DQO | Data Quality Objective |
| FRNP | Four Rivers Nuclear Partnership, LLC |
| JHA | Job Hazard Analysis |
| NaI | Sodium Iodide |
| NCS | Nuclear Criticality Safety |
| NDA | Nondestructive Assay |
| MCA | Multichannel Analyzer |
| PDP | Performance Demonstration Program |
| PGDP | Paducah Gaseous Diffusion Plant |
| PPPO | Portsmouth Paducah Project Office |
| QA | Quality Assurance |
| QC | Quality Control |
| QSNDA | Quality System for Nondestructive Assay |
| RPD | Relative Percent Difference |
| SME | Subject Matter Expert |
| TMU | Total Measurement Uncertainty |
| WAC | Waste Acceptance Criteria |
| WRM | Working Reference Material |

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# EXECUTIVE SUMMARY

The manual serves as a technical overview of Sodium Iodide (NaI) scanning to provide support for the characterization of process gas piping, equipment, and waste components. This will be used as a qualitative method to develop a data assisted approach to characterization. It ensures sufficient information is provided to the user in order to enable duplication of the method. It also outlines the requirements for the generation of valid and defensible data. Additionally, this manual provides the references to the applicable Paducah Gaseous Diffusion Plant (PGDP) Deactivation & Remediation (D&R) contractor's procedures utilized to implement various portions of the method.

Utilizing the requirements and limitations specified within this manual maintains compliance with CP2-ND-1001, *Quality System for Nondestructive Assay Plan at the Paducah Gaseous Diffusion Plant, Paducah, Kentucky*.

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# 1. INTRODUCTION

The Paducah Gaseous Diffusion Plant contractor's Quality System for Nondestructive Assay (QSNDA) Plan (CP2-ND-1001) requires that the nondestructive assay (NDA) program requirements shall be incorporated into the implementing procedures to ensure a quality program as well as ensuring the requirements for valid and defensible data. It also addresses the programmatic requirement for the development of a method manual or equivalent documentation for each NDA method used for providing data to a client. The intent of this method manual is to capture the overall NDA methodology applied to measurements utilizing the NaI detectors and provide either reference to or explanation of the key areas identified in CP2-ND-1001, as applicable:

1. Identification of the NDA measurement method (Section 2)
2. Summary of the NDA method (Section 2)
3. Method scope and application (Section 4)
4. Compatible containerized waste and process component geometries and matrices (Section 4 & 5)
5. Methods of determination for detection limits and minimum detectable values (Section 3)
6. Sources of interference for the expected application and radionuclide(s) (Section 4)
7. Calibration techniques and standards needed to perform calibrations (Section 8)
8. Description of data acquisition and reduction techniques used (Section 10.1)
9. Measurement uncertainty determination technique (Section 6)
10. Calibration confirmation method and performance information and data for source/matrix configurations nominally representative of the actual measurement item population (Section 8.3)
11. Quality control techniques, data assessment, and acceptance criteria for QC measures (Section 9)
12. Description of verification process (Section 10.2)
13. Description of data reporting and approval process (Section 10)
14. Corrective actions for out-of-control data (Section 10)
15. Contingencies for handling out-of-control or unacceptable data (Section 10)
16. Safety considerations (Section 11)
17. Applicable tables, diagrams, flowcharts, and confirmation data (Pages v & vi)
18. Data validation method (Section 10)
19. References (Section 12)

This manual supports those key areas for the sodium iodide gamma analysis for Operations, Environmental Services, and other customers as described in the applicable Data Quality Objectives (DQO's). DQO general requirements are contained in the QSNDA Plan and specific DQO's are customer driven for their specific measurement needs.

# 2. NDA QUALITATIVE NAI SCAN METHOD SUMMARY

NDA is an analysis of an item in which the chemical and physical properties of that item and container remain essentially unaltered. Use at PGDP involves analysis based on observing spontaneous or stimulated nuclear radiation without affecting the physical or chemical form of the material. The purpose of this document is to provide a detailed description of the methodology for using sodium iodide gamma-ray detector systems to support activities related to removal and/or disposition of process gas piping and equipment. It will also be used to ensure the quality and consistency of the data.

The sodium iodide gamma ray detection systems provide a simple and portable method for supporting the characterization of uranium hold-up within process gas piping and equipment. Gamma ray methods rely on capturing gamma photons emitted from the item as part of radioactive decay of the radionuclide species present. Gamma methods are effective when a measurable amount of gamma photons are able to penetrate the items being measured. More specifically, uranium bearing matrices produce a well-known range of gamma rays with energies that are detectable using NaI detectors.

## 2.1 NAI DETECTORS

Sodium iodide systems used for this method are highly portable and efficient, hand-held detector systems. These systems are comprised of two major components: the detector itself, and a scaler/ratemeter unit. The crystal within the detector, where the interactions with gamma radiation occur, is predominately composed of NaI. This is the reason these detector systems are commonly referred to as "NaI detectors". The efficiency of NaI detectors is high, relative to other crystal types, which allows them to produce results rather quickly.

The NaI detector is attached to a scaler/ratemeter unit via a coaxial cable. The scaler/ratemeter provides high voltage to the detector, and displays the count rate. The observed count-rate and/or any other applicable information can be read from the detector system's display, and recorded onto a field worksheet.

The NaI detector is also fitted with a collimator. Collimation is discussed in section 5.4.

This method is designed for use of 2-inch by 2-inch (2x2, e.g., Ludlum 44-10 with a model 2241 or model 3000 meter) or 3-inch by 3-inch (3x3, e.g., Ludlum 44-20 with model 3000 meter) detectors. Due to efficiency differences in detector sizes, results will differ depending on the detector that is chosen. For this reason, the size of the detector used will be noted in each data package this method produces.

**Figure 1: Sodium Iodide Detector Systems. [2x2 (left), 3x3 (right)]**

A test was conducted to compare the two types of detectors on site, 2x2 and 3x3. This was accomplished utilizing working reference material (WRM) known as "mouse pads" (silicone sheets containing UO2F2 powder). A surrogate was loaded and then scanned using each type of detector. See Table 1 and Figure 2 for the comparison of 2x2 and 3x3 detectors. In this case, the 3x3 detector produces roughly three times the counts of the 2x2 detector. Depending on the surrogate and WRM this value is known to fluctuate and should not be used as a standard, rather an example of the possible difference.

**Table 1: Comparison of Responses of Detectors with Varying NaI Crystal Size**

| Comparison of 2x2 and 3x3 Data | | | | | |
|---|---|---|---|---|---|
| Surrogate utilized: NDA-SURR-12PIPE (12" Pipe) | | | NAI Count Rate (kcpm) | | 2x2 to 3x3 Ratio |
| Source ID(s) | Surrogate | gU | 2X2 Detector | 3X3 Detector | |
| PPPO-WRM-MP-4.4-031 | 12" Pipe | 14.9 | 12.7 | 37.0 | 1 : 2.9 |
| PPPO-WRM-MP-4.4-031 thru 032 | 12" Pipe | 29.6 | 22.7 | 73.3 | 1 : 3.2 |
| PPPO-WRM-MP-4.4-031 thru 034 | 12" Pipe | 59.9 | 38.7 | 126.3 | 1 : 3.3 |
| PPPO-WRM-MP-4.4-031 thru 038 | 12" Pipe | 120 | 60.0 | 192.7 | 1 : 3.2 |
| PPPO-WRM-MP-4.4-031 thru 046 | 12" Pipe | 238 | 82.3 | 260.0 | 1 : 3.2 |
| PPPO-WRM-C00001 thru C00003 | 42" Pipe | 565 | 232.0 | 663.0 | 1 : 2.9 |

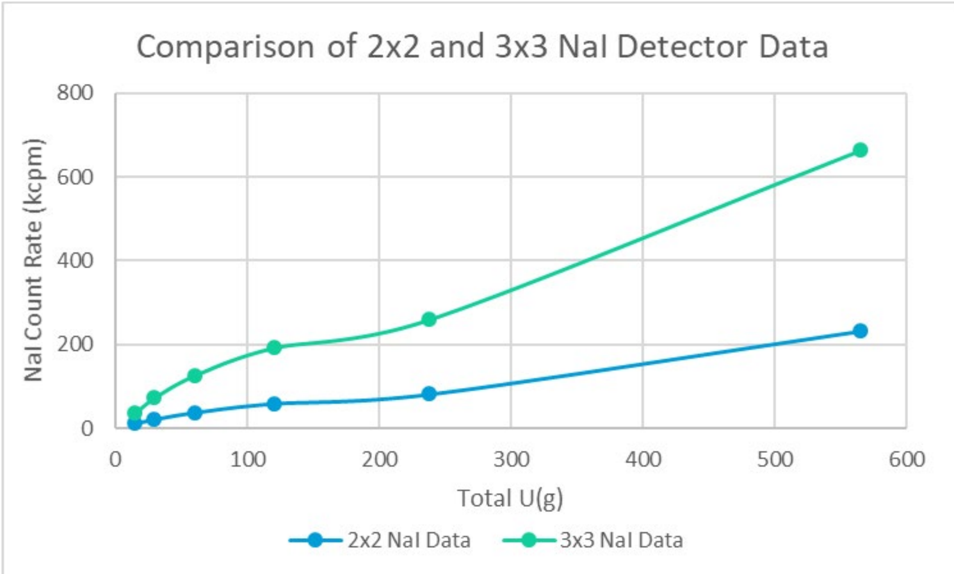



**Figure 2: Graph of Data Associated with the Comparison of 2x2 and 3x3 Detectors**

It should be noted that both detector types are capable of readouts up to 999kcpm. Any reading above this value will continue to display the maximum. Readings of this type are recorded as >/= 999 kcpm.

# 3. DETECTION LIMITS

Detection limits, normally associated with quantification, are not applicable to this method. While count rates are obtained, the associated detection limit for a mass is highly dependent upon the exact system being measured (geometry, wall thickness, container composition, etc.) It is also dependent upon deposit chemical composition, enrichment, geometry, deposit thickness, etc. These values are not determined via current methodology.

# 4. SCOPE AND APPLICATION

The scope of this method is applicable to routine deposit scans or surveys of equipment. NaI scans are normally performed on items where depleted, normal, and enriched $UF_6$, $UO_2F_2$, $UF_4$, and other uranium bearing compounds are contained. Scanning is a qualitative measurement. This method utilizes gross gamma counting in units of kcpm to generate defensible, qualification data for pipe and process gas equipment that will enhance the characterization approach. The method may also be utilized to measure containerized waste of all types.

The results of the method are utilized to support determination of the following:

- Criticality Incredibility (CI)
- Waste Acceptance Criteria (WAC)
- Optimal intrusive sampling locations

- Items that may contain significant uranium deposition
- Ideal placement of other NDA systems
- Uranium hold-up distribution within an item

### 4.1 ASSUMPTIONS

Within process gas equipment and piping at PGDP, uranium isotopes and their associated daughter products are typically the predominate gamma radiation emitters that NaI detectors measure. It is therefore assumed that the majority of the NaI count rates are attributed to these uranium isotopes and their daughter products. However, if other gamma emitting isotopes are present, the increased activity will conservatively bias the NaI measurement results.

### 4.2 LIMITATIONS

1. Detectors with 2x2 or 3x3 inch crystals shall be used.
2. The detector must remain as close to on contact as possible with the measurement item while scanning readings are collected.
3. The lead collimation/shielding shall be configured so that the detector face is flush with the collimator or recessed the minimum amount possible.
4. The maximum scan rate shall not exceed 2"/second.
5. Systems with different enrichment cannot be grouped like for like for comparative purposes.
6. Items with differing wall thickness cannot be grouped like for like for comparative purposes.
7. A background must be obtained without the influence of adjacent equipment adding to the count-rate unless it is also in the background of the item measured.
8. Large deposits which are significantly self-shielding must not be utilized for absolute comparison to other deposits.

## 5. MEASUREMENT METHODOLOGY

### 5.1 DATA QUALITY OBJECTIVES

The Data Quality Objective (DQO) process is intended to be a collaborative effort between the NDA organization and the customer. As such, established DQOs shall be agreed on by both parties and documented. Any changes to established DQOs will be agreed on and documented. DQOs are determined by and derived from the needs and requirements of the customer. NDA evaluates the needs and requirements and details the DQOs in terms relevant to its current capability. NDA notifies the customer if current capabilities are not able to meet the DQO. NDA will work closely with the requesting organization to ensure that data is being completed as requested. The primary goal of NDA measurements is to arrive at a quality result, that is, one that satisfies the user's measurement needs. Adequately analyzing problems and applying appropriate measurement techniques support this goal.

## 5.2 SCANNING WITH HANDHELD DETECTORS

Routine scanning will be performed with the instrument in ratemeter mode. This mode continually updates with a real time value and is used when scanning since it allows you to search for and pinpoint areas with elevated counts. The detector should remain as close to on contact as possible with the measurement item while scanning readings are being collected. This ensures consistent readings.

Total coverage of all measurement items is **NOT** required. In instances where measurement obstructions exist, they must be clearly documented on field sheets and included in the data package for full review.

The following is a list of commonly scanned items and suggested scanning methodologies.

Waste Containers (5.5g drum, 55g drum, ST-90, etc.) Due to the storage array of these containers, i.e. double stacked and side by side, all sides of the container may not be accessible. As much of the container as possible should be scanned, including the top and sides.

Pipe –Any accessible surface area should be scanned. Special attention should be given to expansion joints, elbows/bends, flanges, and weld joints.

Valve- The valve should be scanned from weld joint to weld joint on the pipe stubs and along the valve body. For G-17 valves, if scanning only the subassembly, the valve seats should be scanned and special attention should be given to the bellows where material can accumulate and not be seen visually.

Large Equipment (Converter, Compressor, Holding Drum, Trap, Freezer Sublimer, etc.) - Due to the large size and often thick walls of these containers it is best to scan all sides of the vessel. Special attention should be given to the bottom of the vessel along with any inlet and outlet ports or connections. Some of the larger equipment types have seen upgrades over time. Any extra weld joints or previously opened areas should be checked thoroughly.

There are two stages of scanning with NaI detectors, dynamic (moving) and static (stationary) readings. Upon noting an increase in the number of counts, the measurement performer will pause briefly and then decide whether to move on or take further measurements. The first stage is characterized by continuous movement of the probe, the surveyor has only a brief "look" at potential sources. The second stage occurs only after a positive response was made at the first stage. Movement of the probe will slow greatly with the surveyor holding the probe stationary for a period of time until the readings begin to level out. The detector is set to Auto-Response Rate/Fast Mode. While this setting allows for variable response time, the meter automatically reduces to a 1 second response time when a sudden change in count rate is observed. It should be noted that the maximum scan rate shall not exceed more than 2 inches per second.

All components should be scanned so that readings are collected on all accessible surfaces. This method relies on personnel collecting as much data as possible for each component and properly identifying areas that are not accessible. Personnel will utilize maps, drawings, photos, or descriptions to record scan values. When using standard equipment drawings, any deviation from the normal should be noted in detail.

Potential interferences that might impact the actual measurement include, but are not limited to:

- Background radiation such as that from adjacent piping, process equipment, etc.
- Radionuclides other than uranium – Example Neptunium-237

### 5.3 BACKGROUND MEASUREMENTS

It is required for a background to be taken with each measurement item. The method used for measuring background often determines how much or how little conservatism will be added to a measurement.

The best background method should be chosen for each unique measurement application. Area background is normally measured by scanning a 270° to 360° area directly around the item being measured (e.g. all cardinal directions, up, and down, as applicable). These directions along with the corresponding background values are all recorded. The direction of scanning is also indicated so that the correct background may be applied. The detector is usually held perpendicular to the body at waist or shoulder height, depending on the item and then parallel to the body facing the floor and then ceiling. Pointing the detector toward open areas for a general area background is best but not always possible. If the item being measured has elevated readings, the items directly adjacent should also be surveyed to ensure they are not elevated and causing interference. Items that are large in size or items with considerable lengths may require multiple backgrounds.

### 5.4 COLLIMATION/SHIELDING AND FIELD OF VIEW

Sodium iodide detector systems utilized for this method are shielded using lead sleeves, often referred to as collimators. This collimation significantly reduces the incident gamma radiation from the sides of the detector, allowing the system to be used in areas with high background or next to other items with significant uranium deposits. The collimators are open on the end of the detector where the crystal is located, which allows radiation from the measurement item to enter the detector without being impeded. Some detectors are equipped with collimator sleeves that can be adjusted such that the detector face is flush, recessed, or exposed relative to the collimator. The collimator is configured such that the detector face is flush with the collimator sleeve (i.e. – zero collimation) or recessed the minimum amount possible.
The 2” detectors (Ludlum 44-10) utilize a standard lead factory collimator (Part # 4260-076) with a wall thickness of 0.23”. The 3” detectors (Ludlum 44-20) are wrapped with a 1/16” lead sheet.

## 6. TOTAL MEASUREMENT UNCERTAINTY

CP2-ND-1001 requires the evaluation of the total measurement uncertainty for each method. It also states that “the TMU is determined for all reported measurement values, as required by the data end user”.

This method is not directly quantitative and does not reasonably permit the traditional reporting of total measurement uncertainty for a mass value. However, the uncertainty of the count rate measurement ($\sigma_{crm}$) associated with the scan data is calculated according to the equation below:

$$\sigma_{crm} = \sqrt{\frac{count\ rate}{2 * response\ time}}$$

With a response time of 1 sec and a count rate of 3 kcpm, the calculated $\sigma_{crm}$ is 10%.

As the response time and/or count rate increases, the calculated uncertainty decreases. Thus, the TMU is estimated at 10%, as a maximum. As the DQO indicates, the uncertainty may be calculated for a given measurement situation to arrive at a more accuracy based value.

Precision and bias are discussed in section 8.3.

# 7. MEASUREMENT CONSIDERATIONS

Data was collected by measuring certified uranium-bearing WRM. WRMs were placed inside various surrogates in order to realistically simulate the attenuation of x-rays and gamma rays that exist within process equipment. The WRMs/surrogates were arranged in multiple configurations such that enrichment and WRM mass loading were varied throughout the acquisition of measurements. Replicate measurements were also taken for configurations in order to better examine the system's performance and limitations. These replicate measurements show the reproducibility of NaI readings when scanning items.

## 7.1 ENRICHMENT

When comparing count rate versus uranium mass, lower enrichments produce lower count rates per gram of uranium. This can be seen in Table 2 and Figure 3 below. Testing was conducted using roughly the same amount of Total gU at three different U-235 enrichments, depleted (0.231 wt. % U-235), normal (0.711 wt. % U-235), and enriched (4.96 wt. % U-235). Each setup was placed in a 12in surrogate pipe. As can be seen, the total gU of the enriched material was lower than that of the normal and depleted material, and it still produced higher counts than either of the latter.

**Table 2: NaI Detection at Varied Enrichments of U-235**

| Comparison of Enriched, Normal, and Depleted Deposits 2X2 | | | | |
|---|---|---|---|---|
| Surrogate Utilized: NDA-SURR-12PIPE (12" Pipe) | | | | |
| Source IDs | | PPPO-WRM-C00708, 00709, 00710, 00711, 00718 | PPPO-WRM-C00003, 00012, 00022, 00029, 00032 | PPPO-WRM-C00114, 00116, 00118, 00121, 00143 |
| wt% U-235 | | Enriched | Normal | Depleted |
| Max NaI Counts (kcpm) | 1 | 461 | 338 | 339 |
| | 2 | 466 | 346 | 341 |
| | 3 | 454 | 354 | 340 |
| AVG Max NaI Counts (kcpm) | | 460 | 346 | 340 |
| Total U (g) | | 937 | 956 | 963 |
| Total U-235 (g) | | 46.5 | 6.80 | 2.23 |

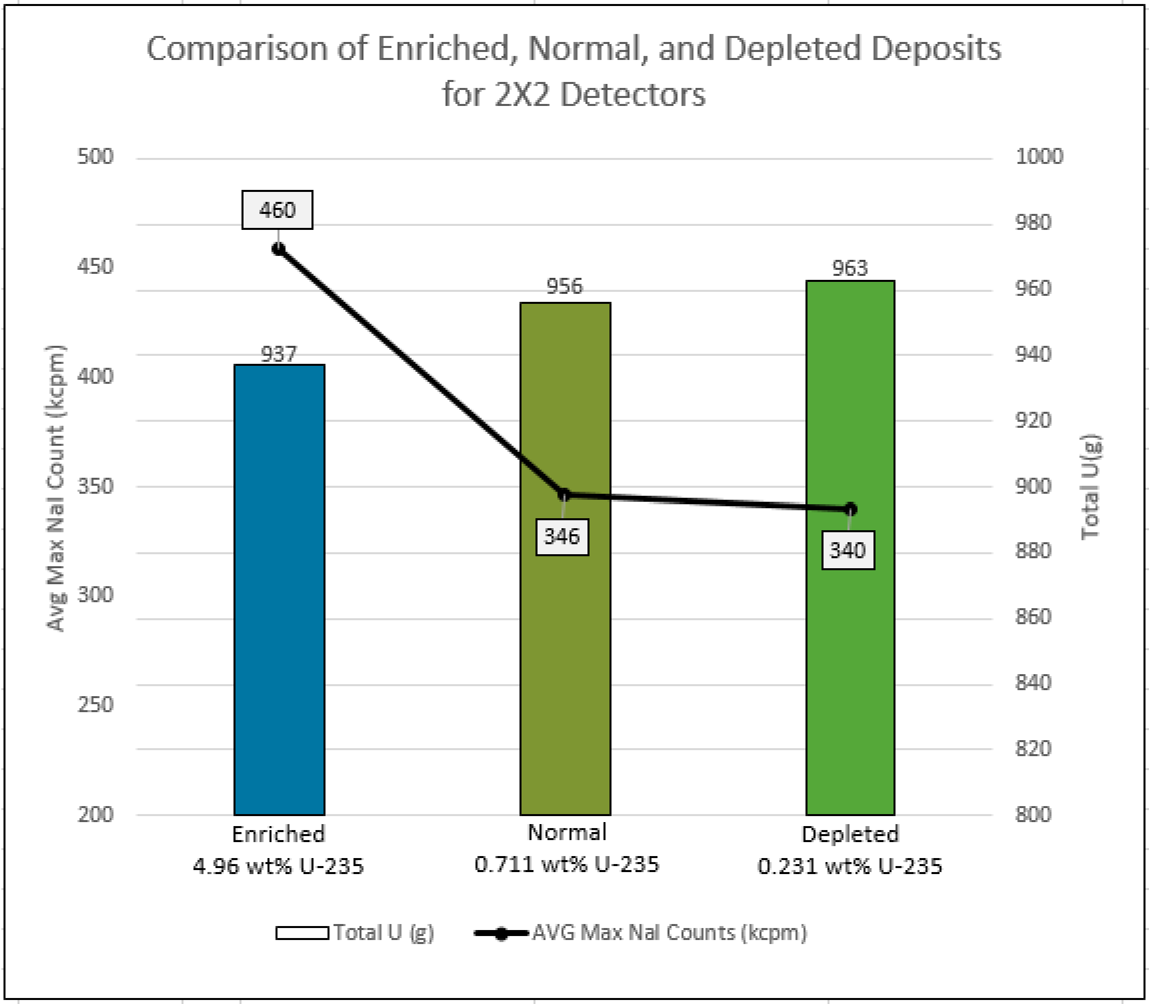



**Figure 3: Comparison of Enriched, Normal, and Depleted Deposits using 2x2 Detectors**

It should be noted that when comparing observed count rate versus $^{235}U$ mass, as opposed to total uranium, the higher enrichments tend to produce lower count rates per gram. Looking at Table 2, the maximum count rate for the normal and depleted material is lower than that for the enriched. However, the U-235 mass for the normal material is only a fraction of that for the enriched material. Thus, comparing the relative U-235 mass, the lower enrichment will yield a much greater count rate than the enriched uranium, resulting in a conservatively high scan rate for the same U-235 mass. When looking at similar U-235 masses, as the enrichment decreases the total uranium mass will increase significantly.

## 7.2 WALL THICKNESS

The equipment covered in this method is made up of many different wall thicknesses. The wall thickness of a measurement item impacts the amount of photon attenuation (i.e., absorption of photons) between the source and the detector. A thick component wall correlates to more attenuation and lower counts than a measurement of the same deposit in a component with a thinner wall.

Table 3 and Figure 4 evaluate the effects of wall thickness on deposits of the same makeup. The same WRM (3 normal 0.711 wt. % U-235 tubes) were placed in surrogates of varying wall thicknesses. The tubes were laid flat, side by side, within each surrogate. The surrogates were scanned and the highest NaI reading was recorded. As seen in Figure 4, the effect of wall thickness on attenuation is not linear. As the wall thickness increases, the percentage of gamma rays attenuated lessens. This is true because the lower gamma energies are more easily attenuated.

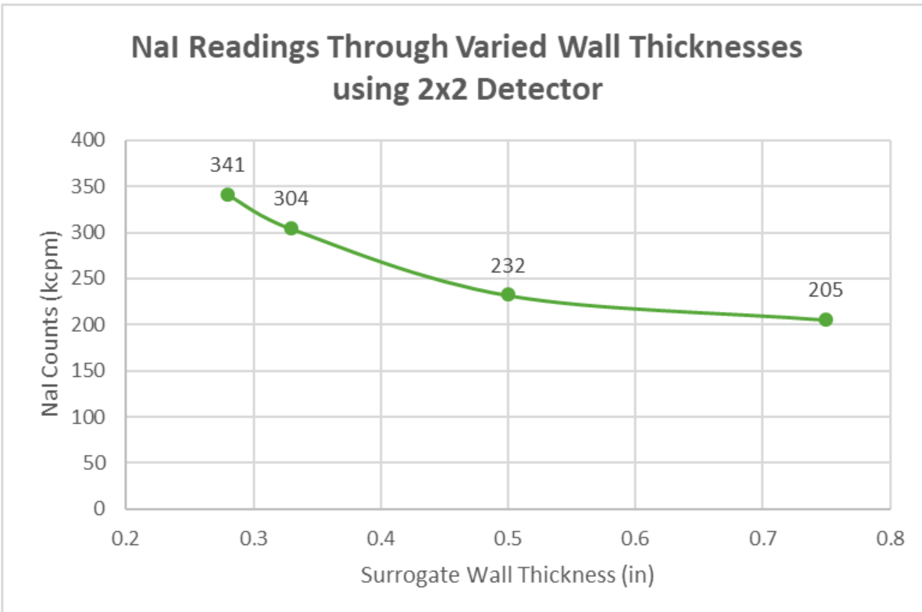



**Figure 4: Chart of NaI Readings through Varied Wall Thicknesses using 2x2 Detector**

**Table 3: NaI Readings through Varied Wall Thicknesses using 2x2 Detector**

| NaI Readings Through Varied Wall Thicknesses using 2x2 Detector | | | | | |
|---|---|---|---|---|---|
| Sources Used: PPPO-WRM-C00001 thru C00003 (3 normal tubes in bottom) | | | | | |
| Enrichment wt%: 0.711 Total U (g): 565 | | | | | |
| **Surrogate** | **Wall Thickness** | NaI Count (kcpm) | | | |
| | | 1 | 2 | 3 | Average |
| 6" Pipe | 0.28 | 335 | 339 | 349 | 341 |
| 12" Pipe | 0.33 | 309 | 303 | 300 | 304 |
| 42" Pipe | 0.5 | 236 | 231 | 228 | 232 |
| 42" Valve | 0.75 | 203 | 206 | 206 | 205 |

## 7.3 BACKGROUND CONSIDERATIONS

Depending on location of item measurement, the background value can vary greatly. There are several locations on plant site that are known to have increased background counts. There are also instances where a high background is not expected but occurs. For this reason, testing was conducted to consider the effects of background values on item scan values.

Testing was accomplished by placing two surrogate pipes in close proximity of one another (2.5ft) to simulate neighboring pipes in the process gas system (Pipe 1: 12 inch diameter, Pipe 2: 30 inch diameter). The 12 inch pipe was loaded with a constant amount of UO2F2 while the 30 inch pipe was filled with varying materials. The background recorded was the highest value when conducting background measurement in the direction of Pipe 2. Results are shown in Table 4.

**Table 4: NaI Scanning and Background Effects**

| NaI Scanning and Background Effects | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Setup | | | | NaI Count Rate (Pipe 1) | | | | |
| Pipe 1: NDA-SURR-12PIPE | gU | Pipe 2: NDA-SURR-30PIPE | gU | Background | 1 | 2 | 3 | Average |
| Two Normal tubes PPPO-WRM-C00041 AND C00042 | 379 | Empty | 0 | 3 | 145 | 143 | 145 | 144 |
| Two Normal tubes PPPO-WRM-C00041 AND C00042 | 379 | One Normal tube PPPO-WRM-C00001 | 187 | 4 | 145 | 142 | 146 | 144 |
| Two Normal tubes PPPO-WRM-C00041 AND C00042 | 379 | Five Normal tubes PPPO-WRM-C00001 THRU C00005 | 943 | 6 | 146 | 147 | 149 | 148 |
| Two Normal tubes PPPO-WRM-C00041 AND C00042 | 379 | Ten Normal tubes PPPO-WRM-C00001 THRU 00010 | 1870 | 9 | 151 | 149 | 149 | 149 |
| Two Normal tubes PPPO-WRM-C00041 AND C00042 | 379 | Twenty Normal tubes PPPO-WRM-C00001 THRU 00020 | 3740 | 14 | 151 | 149 | 152 | 150.5 |
| Two Normal tubes PPPO-WRM-C00041 AND C00042 | 379 | Forty Normal tubes PPPO-WRM-C00001 THRU 00040 | 7510 | 17 | 152 | 151 | 153 | 152 |
| Two Normal tubes PPPO-WRM-C00041 AND C00042 | 379 | Eighty Normal tubes PPPO-WRM-C00001 thru C00082 (EXCLUDING C00056 & C00071) | 15000 | 28 | 154 | 153 | 153 | 153 |

As one can see, when the amount of UO2F2 increases in Pipe 2, the count rate for Pipe 1 also begins to increase. Background values are important to each measurement and personnel performing scans should note directions of higher backgrounds.

## 7.4 INFINITE THICKNESS

Larger uranium deposits have increased self-attenuation; meaning large deposits can significantly shield their own gamma rays. Due to this concern, an evaluation of infinite thickness must also be conducted to examine the range of uranium masses that can be reliably detected using NaI detectors.

The chart below, Figure 5, shows the relationships between total grams U and the NaI count rate. Silicone sheet WRM (mouse pads) were used to show this relationship. Starting with one mouse pad inside a 12in surrogate pipe, the highest reading was recorded. One mouse pad was added on top of the previous material during each scan until a total of 20 mouse pads were present. The pipe was scanned each time additional WRM were added to the surrogate.

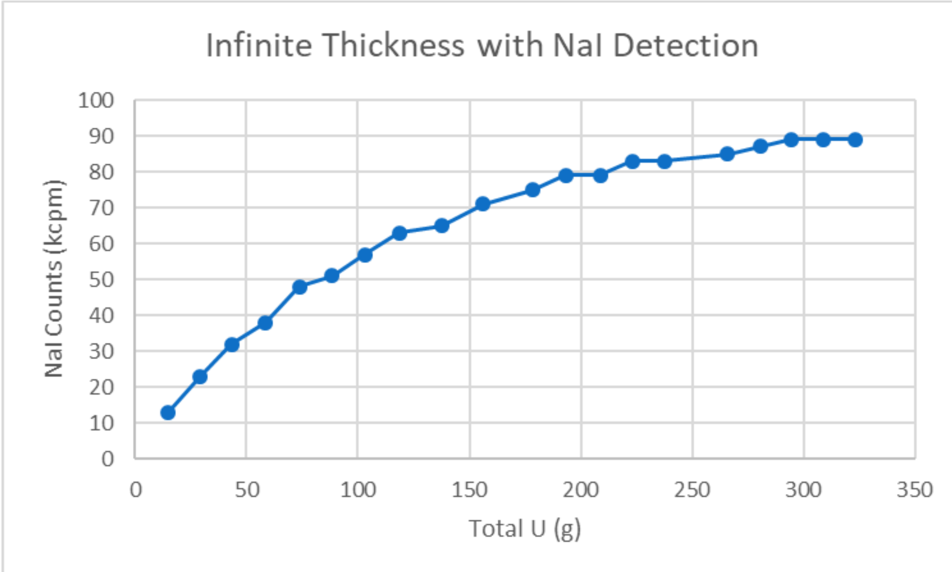



**Figure 5: Graph of Infinite Thickness Testing**

As additional mouse pads are presented, the counts no longer increase at the same rate. Eventually the addition of more material doesn't increase the count rate at all. Count rate is dependent on deposit size and distribution within the item. For example, the same amount of material configured in a different way will cause a variation in the number of counts seen. For this particular method, we wish to identify items that contain hold-up but aren't necessarily concerned with the amount of hold-up that is present. For example, a customer could request scans to determine optimal intrusive sampling locations. Therefore, a deposit that is optimal as a location for sampling may be identified, regardless of whether it's infinitely thick or not. An infinitely thick deposit will always produce a significant count rate resulting in further analysis using alternate methods.

# 8. CALIBRATION AND PERFORMANCE

| |
|---|
| Note: Collimators are not typically utilized during initial calibration. They are typically used during normalization. |

## 8.1 INITIAL CALIBRATION

NaI detectors and meters are calibrated annually, at minimum, by the manufacturer, qualified vendor, or qualified company personnel to factory specifications using traceable equipment and traceable reference standards. The detector and associated electronics are calibrated and paired as "married" sets, and are not to be used interchangeably. This calibration evaluates the detector to ensure that it is in acceptable working condition, the optimum voltage is being used, and certifies the detector's efficiencies for certain traceable isotopes are within normal ranges. The optimum voltage should be selected at all times. This calibration is conducted initially, and at least annually thereafter. Calibrations are also required with a major system repair or replacement of a vital NDA measurement component such as the collimator. Calibration documentation is maintained, whether in-house or vendor.

At a minimum, the following will be performed/provided:

- Perform a calibration check with a Cesium-137 (Cs-137) reference standard traceable to the NIST or other nationally recognized supplier of traceable standards.
- Provide calibration data ("As-Found" and "As-Left" count rates, tolerances and uncertainties of the calibration).
- Provide acceptable count rates tolerance for meters/NaI(Tl) systems.
- Perform high voltage plateau measurements and determine the optimum operating high voltage.
- Gross efficiency (%) and error at a 95 % confidence level with a Cs-137 source as well as the precision to the measurement.
- Inspect hardware and perform any necessary general maintenance.

## 8.2 NORMALIZATION TESTING

> NOTE: Normalization is not required for a detector system utilized for single event or item testing. For example, scanning an item in support of a neutron quantification does not require a detector to belong to a family of detectors. Normalization supports multiple detectors which are utilized for comparative data.

A group or "family" of detectors utilized for generating a set of comparative data will be tested to ensure that they produce results that are not statistically different for a given measurement. This process is referred to as normalization and ensures that the detectors are not statistically different. The family of detectors are typically set up with like detectors, meters and shields/collimators.

### 8.2.1 Normalization Measurements

A set of detector systems is established as a family. An equivalent uranium working reference material or certified reference material is measured a minimum of 30 times using each detector system. The measurements are carried out over multiple days in order to take into account uncertainty associated with variable daily conditions. The data is entered into the NaI Normalization spreadsheet.xls to ensure the detector or detectors results fall within the normalization limits.

### 8.2.2 Example data

A set of data for a family of 11 detector systems was analyzed to determine the initial limits utilized to determine if each detector fit into the family. Statistical evaluation of the data determined that all detectors were acceptable as a family. Appendix A contains the statistical evaluation report for the data.

### 8.2.3 Normalization Limits

From this evaluation, limits were established to be utilized for detector acceptability.

Detectors may be adopted into a family of detectors. The detector must undergo the same measurement campaign and be within the limits as set for the original family.

A single family of detectors exists when the following limitations are met:

**A.** Individual results (single net counts per minute) fall within 5% of the mean of the group of detectors.

OR

**B.** A single detector standard deviation based on 30 point initial setup falls within 2% of the average of all detectors.

The initial normalization limits are based upon a set of data that was generated during a relatively short timeframe and with a limited number of detectors. As more data is collected it may be necessary to update the limits.

## 8.3 CALIBRATION CONFIRMATION

The calibration confirmation process is designed to produce objective evidence demonstrating the applicability and correctness of the "initial" calibration relative to the measurement of interest. The recommended method is to assemble test item surrogates (pipes, expansion joints, valves, etc.) consisting of source/matrix configuration(s) nominally representative of the items to be characterized. The surrogates contain a known and traceable radioactive element/isotope mass/activity in a known and representative configuration. The confirmation test item(s) are then measured using the "initial calibration" of the NDA system. The number of differing test item configurations used to confirm the calibration is to be determined by the NDA service provider and documented. The reported "calibration confirmation" measurement result must agree (with criteria as established by the NDA service provider) with the known element/isotope mass/activity of the confirmation test item(s). As stated in CP2-ND-1001 alternate acceptance criteria may be used, when approved by the NDA QA Manager and the independent third party review.

Six replicate measurements will be performed of each calibration confirmation setup for measurement. As this method does not utilize direct quantification, confirmation will be represented by the ability of gamma analysis to determine any areas of concern and produce a scan map for characterization purposes. The method does not have specific requirements for bias since a quantitative value is not produced. Precision may be calculated by taking the highest measurement value recorded for each of the six replicates. The standard deviation (Std Dev) and average of the six values is calculated. From that, the precision (%RSD) is calculated as 100% x (Std Dev/Average).

The relative precision limit is **43.45%**, calculated for a population of six replicates according to approved alternate method. Approval for use of the alternate calculation method can be found in PPPO-02-4627705-18. All calibration confirmation measurement sets must meet this requirement.

The analysis of each calibration confirmation measurement will compare the location and the distribution of radiological material within the test item to the location and distribution identified in the scan map. The comparison will result in a “pass/fail” evaluation of the data.

The calibration confirmation is performed periodically, at least annually, as a reassessment of the system as well as when significant changes to the system indicate a possible change in the initial calibration. Where QC trending analyses and calibration verification data indicate that a full "calibration confirmation" is not warranted, NDA management will petition the NDA QA Manager for an extension on an NDA instrument basis.

### 8.4 CALIBRATION VERIFICATION

The calibration verification is a measure designed to evaluate the long-term stability of the "initial calibration." The calibration verification is a point check within the initial calibration space using test items assembled from the traceable WRMs and waste matrix surrogates and/or process component mock-ups. The calibration verifications will follow the same criteria as the calibration confirmations, with the exception of the precision.

The maximum of 30 operational days will be used as the calibration verification performance requirement for this method. The operational period is a rolling tally of 30-days where an NDA system is in active operational mode, but not necessarily consecutive days. The use of uranium sources to perform quality control checks proves that the system remains stable between each calibration verification. See section 9.1, Daily Performance Checks, for more information.

The calibration verification is blind to the extent possible.

### 8.5 PERFORMANCE DEMONSTRATION PROGRAM

The sodium iodide scanning systems shall participate in performance (capability) demonstration programs (PDP) as specified in CP2-ND-1001. The capability demonstration testing is a single-blind test, and uses test samples representative of the waste matrix types, process component configurations and radioactive materials that the system shall characterize. Once the initial PDP has been completed successfully, a PDP is required annually, or at a frequency determined by the DOE PPPO.

The method will not follow typical PDP requirements but will be expected to reproduce similar results with each replicate using a Pass/Fail acceptance criteria similar to the calibration confirmations.

## 9. QUALITY ASSURANCE

Data quality is ensured through compliance with the CP2-ND-1001, *Quality System for Nondestructive Assay Plan at the Paducah Gaseous Diffusion Plant, Paducah, Kentucky*, CP4-ND-1000, *Nondestructive Assay Quality Implementation*, and other quality documents and international standards. Further, by achieving compliance with the FRNP QSNDA, this document maintains a sufficient level of quality to satisfy the quality requirements of requesting parties. Data quality objectives (DQOs) and method requirements that are not ensured by the design or limitations of this method manual shall be identified and flowed down into operating procedures.

## 9.1 DAILY PERFORMANCE CHECKS

Quality control (QC) measurements are required each day the NaI system is used to perform QSNDA measurements. These quality control checks must be performed before and after any reportable data is collected, and are commonly referred to as “pre-QC” and post-QC” measurements, respectively. Unless otherwise directed by an operating procedure, in the event that a “post-QC” fails and it is determined that the instrument is out of control, all measurement data collected since the most recent acceptable QC must be either discarded, or reviewed by a subject matter expert (SME) and the NDA Quality Assurance (QA) Manager to determine any limitations to be placed on the use of the data.

After calibration, QC limits are established based upon a minimum of 30 measurements of a source, background subtracted. A $UO_2F_2$ WRM standard is utilized to establish the warning and control limits at the two and three standard deviations from the mean of the data. A control chart is generated from the data for tracking and trending.

Daily performance measurements and requirements are detailed further in CP4-ND-1000, *Nondestructive Assay Quality Implementation*.

## 9.2 REPLICATE MEASUREMENTS

The replicate measurement is acquired by randomly selecting one measurement item from a given batch to be processed through the NDA measurement. The item may be a piece of equipment, a section of pipe or a defined subset of a given pipe. This measurement item is then measured twice within a batch using the same NDA system, software, and acquisition/reduction parameters (i.e., it is not an independent measurement). The second measurement becomes the replicate that is reported for that batch. The value utilized for the replicate analysis will be the highest value obtained during the item scan.

The replicate data is evaluated using relative percent difference (RPD) according to the following equation:

$$RPD = 100\% \cdot \left|\frac{D-S}{S}\right|$$

where $D$ is the duplicate measurement result, and $S$ is the initial measurement result.

If both values are at background, the replicate will be considered a pass. Otherwise, the RPD must be within 25% to be considered a valid replicate for the batch, unless otherwise stated in CP2-ND-1001. Failure of this test will require an investigation, and where applicable, implementation of a corrective action plan. At low background (</= 5 kcpm), two replicate values near background may fail the 25% RPD but be determined by the SME to be a pass during the minor issue process. For example, at a background of 3 kcpm, replicate values of 4 and 6 would fail but be essentially equal with respect to a given DQO.

The replicate data is monitored and tracked to allow for continued assessment of instrument reproducibility.

# 10. DATA MANAGEMENT

Data acquisition planning serves to identify and document appropriate requirements and responsibilities for the management, quality assurance, use, and archival of NDA data collected by the project.

## 10.1 DATA ACQUISITION AND REDUCTION

NDA measurements are performed, and data recorded per instructions according to CP4-ND-1014, *Nondestructive Assay Data Flow and Review Process* and CP4-ND-1003, *Nondestructive Assay Scans*.

The data sheets are typically provided to customers as a scan map. This map was produced in the field with little or no data reduction.

No software is utilized by the user during data acquisition.

## 10.2 DATA EVALUATION, ASSESSMENT, VERIFICATION, VALIDATION, AND ACCEPTANCE CRITERIA

Data requirements to ensure consistent and quality assured data, including data tracking, data collection, review, verification, reporting and approval are found in CP4-ND-1014, *Nondestructive Assay Data Flow and Review Requirements*. The process ensures that all data released for decision making and/or external use have received adequate quality assurance reviews. The procedure provides guidance and controls for data in the various stages of data development, including the tracking of the data packages and item measurements. The NDA measurement data package is compiled and reviewed in accordance with CP4-ND-1014. All records are controlled in accordance with CP3-RD-0010, *Records Management Process*.

## 10.3 CORRECTIVE ACTIONS

Guidance for monitoring and establishing out-of-control data is provided in CP4-ND-1000. Data that is out-of-control is identified and evaluated according to CP4-ND-1014 prior to re-analysis. Any issue management or corrective action undertaken will be in accordance with applicable procedures.

# 11. SAFETY CONSIDERATIONS

Safety issues are covered in the Precautions and Limitations section of CP4-ND-1003. In addition, job safety hazards are listed, and mitigation controls are analyzed under Job Hazard Analysis (JHA) documents. Applicable JHA documents are referenced in the operating procedure, CP4-ND-1003.

# 12. REFERENCES

CP2-ND-1001, *Quality System for Nondestructive Assay Plan at the Paducah Gaseous Diffusion Plant, Paducah, Kentucky*

CP2-QA-1000, *Quality Assurance Program Description for the Paducah Gaseous Diffusion Plant, Paducah, Kentucky*

CP4-ND-1000, *Nondestructive Assay Quality Implementation*

CP4-ND-1003, *Nondestructive Assay Scans*

CP4-ND-1012, *Nondestructive Assay Method Development and Detector Testing Guidelines*

CP4-ND-1014, *Nondestructive Assay Data Flow and Review Process*

PPPO-02-4627705-18, *Approval of Request for Alternative Method for Percent Relative Standard Deviation Calculation for Calibration Confirmation Acceptance Criteria.*

# APPENDIX A

Statistical Summary of Analysis:

The quality of the 11 Ludlum NaI detectors is very good. Statistical diagnostics tests were performed on the data sets. The data reveals no outliers, the overall data set is not statistically different from a normal (Gaussian) distribution, and the 11 detector standard deviations are not statistically different and can be pooled to generate a single estimate that represents the entire family of detectors. Based on this data analysis it can be concluded that a single family of detectors exists when (1) Individual results (single net counts per minute) fall within 5% of the mean or (2) a single detector standard deviation based on 30 point initial setup falls within 2% of the average.

A detailed discussion of the results are described below.

**Outlier Test**: A Grubbs outlier test was performed on each set of 30 setup points on each of the 11 detectors. All outlier tests were insignificant indicating no values are a significant outlier at the 5% significance level. This is indicative of data that are well behaved and generated from a consistent single distribution.

Method: Grubbs' Test

| | |
|---|---|
| Null hypothesis | All data values come from the same normal population |
| Alternative hypothesis | Smallest or largest data value is an outlier |
| Significance level | $\alpha = 0.05$ |

| Variable | N | Mean | StDev | Min | Max | G | P |
|---|---|---|---|---|---|---|---|
| NaI-5 Net | 30 | 16358 | 148 | 15987 | 16650 | 2.50 | 0.251 |
| NaI-6 Net | 30 | 16706 | 203 | 16152 | 17040 | 2.74 | 0.104 |
| NaI-12 Net | 30 | 16342 | 199 | 15893 | 16585 | 2.26 | 0.554 |
| NaI-13 Net | 30 | 16812 | 222 | 16445 | 17221 | 1.84 | 1.000 |
| NaI-15 Net | 30 | 16557 | 242 | 16170 | 17090 | 2.20 | 0.667 |
| NaI-16 Net | 30 | 16625 | 230 | 16150 | 17050 | 2.07 | 0.993 |
| NaI-17 Net | 30 | 16406 | 240 | 15760 | 16960 | 2.69 | 0.124 |
| NaI-18 Net | 30 | 16581 | 230 | 16150 | 17100 | 2.25 | 0.572 |
| NaI-19 Net | 30 | 16539 | 235 | 16000 | 16930 | 2.29 | 0.502 |
| NaI-21 Net | 30 | 16586 | 171 | 16200 | 16890 | 2.25 | 0.577 |
| NaI-22 Net | 30 | 16764 | 165 | 16420 | 17010 | 2.09 | 0.930 |

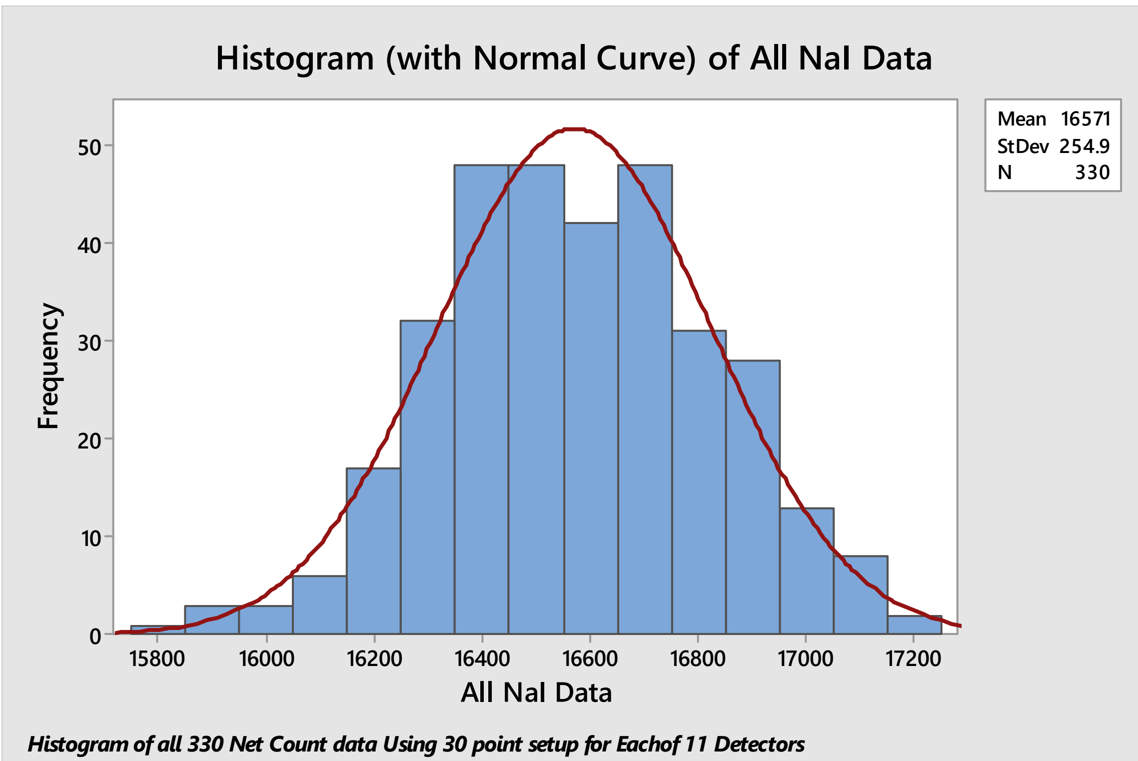
*** NOTE * No outlier at the 5% level of significance**

**Normality Test:** Normality tests were performed on the dataset as a whole since there were no outliers in the individual datasets. Since the smallest P-value amongst the tests performed is greater than or equal to 0.05, we cannot reject the idea that All NaI Data comes from a normal distribution with 95% confidence. A histogram of the entire data set is shown below. Based on the histogram and the normality tests, the data appear to be normally distributed.

Data variable: All NaI Data
330 values ranging from 15760 to 17221.

**Tests for Normality for All NaI Data**

| *Test* | *Statistic* | *P-Value* |
|---|---|---|
| Shapiro-Wilk W | 0.98381 | 0.48064 |
| Anderson-Darling | 0.30515 | 0.567 |



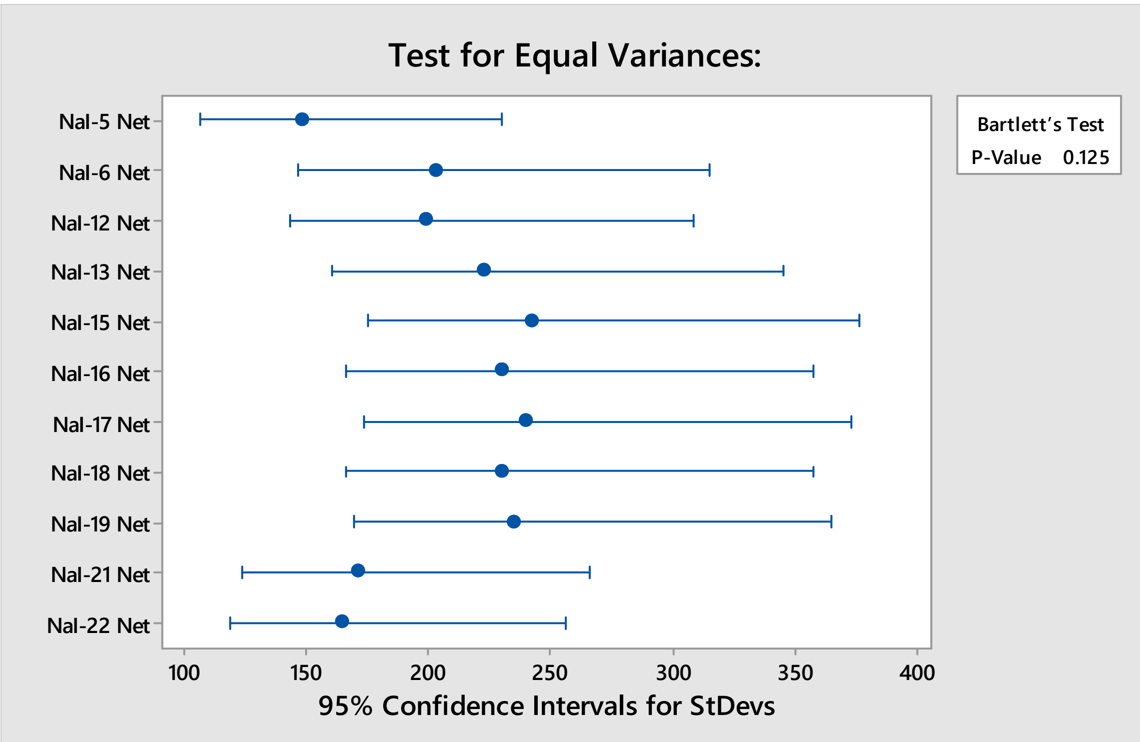

**Variation Test:** Variability tests were performed on the individual datasets to determine whether any of the variances (or standard deviations) of the 11 groups differ. Since the normality tests determined the data were normally distributed the Bartlett's test for equal standard deviations assuming normality was employed. Since the P-value is greater than or equal to 0.05, we cannot reject the null hypothesis that the variances (standard deviations) are equal.

Test for Equal Variances:

| | |
|---|---|
| Null hypothesis | All variances (or standard deviations) are equal |
| Alternative hypothesis | At least one variance (or standard deviation) is different |
| Significance level | α = 0.05 |

*Bartlett's method is used. This method is accurate for normal data only.*

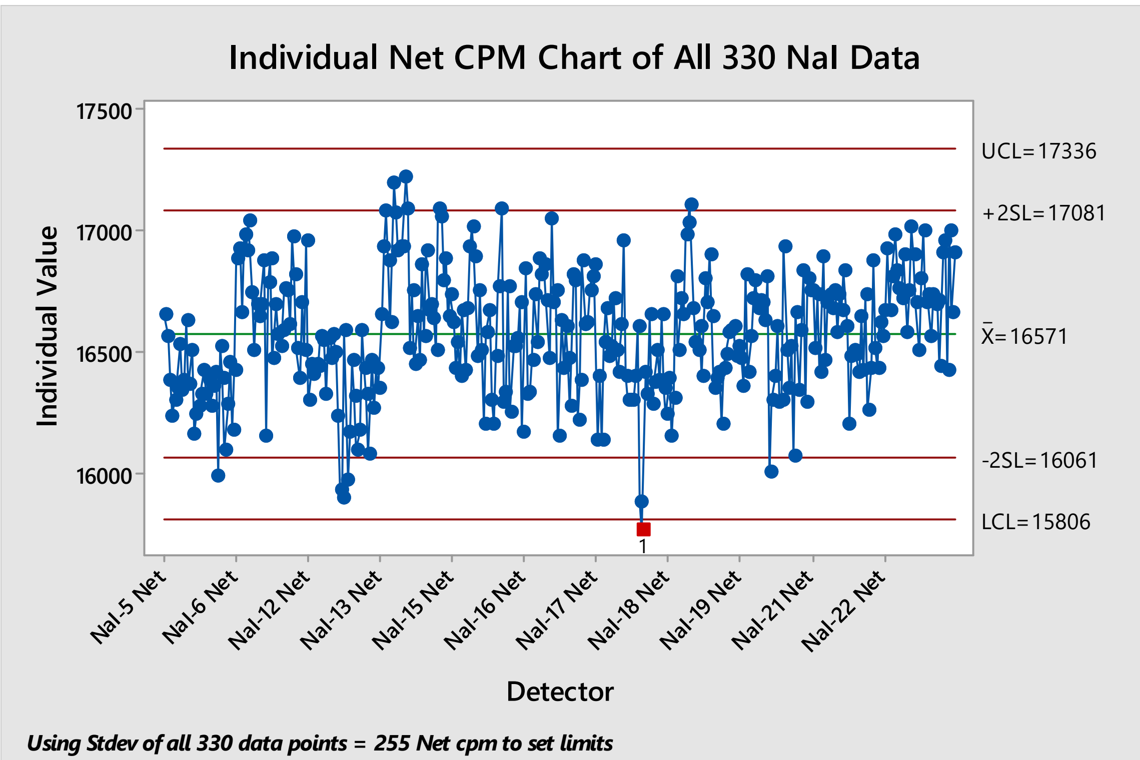
| Method | Test Statistic | P-Value |
|---|---|---|
| Bartlett | 15.19 | 0.125 |

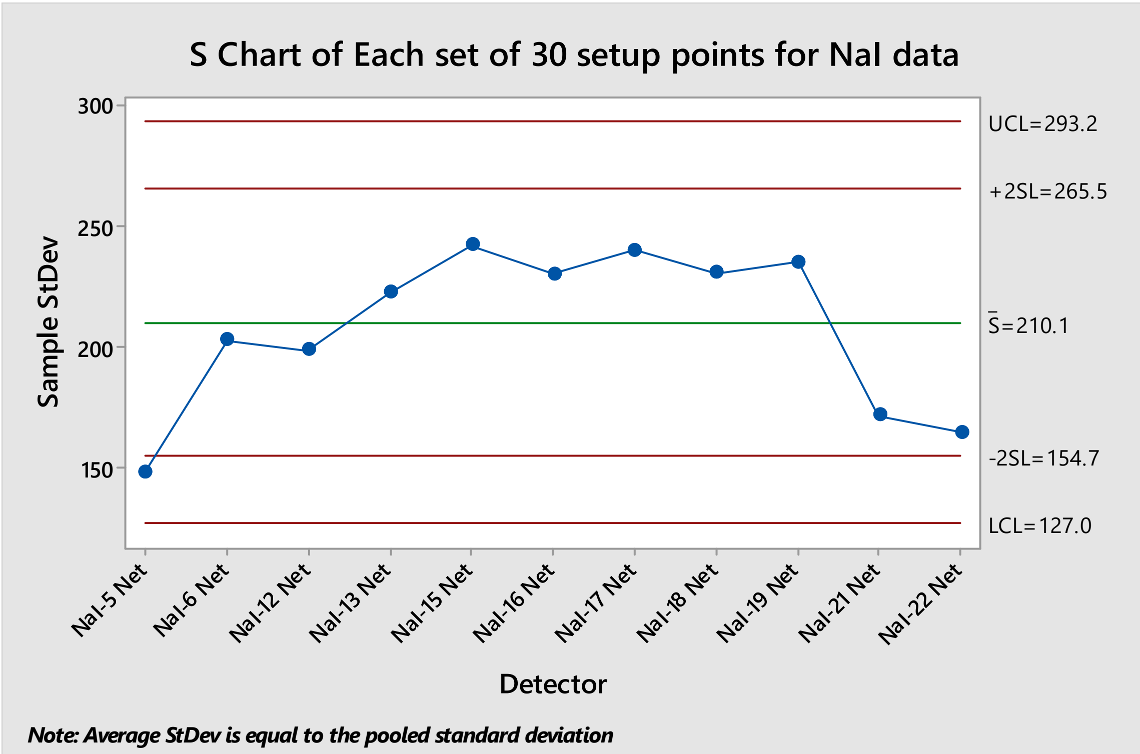



**Rule for Normalized Family of Detectors**: Since there were no outliers, the standard deviations are not statistically different, and the entire set of 330 results can be represented by a single normal distribution, the data can be considered belonging to a single normalized family. A typical Shewhart control chart of the entire data set can now be developed to determine the normalized limits for a detector that belongs in this family. The control chart with all 330 data points plotted has UCL (3S) limits = 17336 Net CPM based on the overall standard deviation of 255 Net CPM and the overall grand average of 16571 Net CPM. As shown on the chart there is only 1 result outside 3S which is expected using a standard 3S Shewhart Control Chart. (expectation is 3 in 1000 or 1 every 333 points will be outside 3S). Using this standard deviation of 255, the normalized rule should be that all data will fall within 3S (3*255) = 765 Net CPM or 4.6% of the mean for a family of detectors.

Additionally, the S chart for monitoring the standard deviation among the 11 detectors employs the pooled standard deviation of 210.1 net CPM as the average standard deviation (using the classical pooled standard deviation formula). The upper 3S limit indicates a normalized detector should have a standard deviation no larger than 293.2 net CPM or 293.2/16571 = 1.77% of the average (when initial setup is based on 30 points). The normalized rule should be that a detector belongs to the family if an initial setup standard deviation of 30 points is less than or equal to 1.77% of the average.

Minitab Statistical Software and Statgraphics Centurion Statistical Software were employed to analyze the detector data.




S Chart of Each set of 30 setup points for NaI data
Sample StDev
300
250
200
150
UCL=293.2
+2SL=265.5
S̄=210.1
-2SL=154.7
LCL=127.0
NaI-5 Net
NaI-6 Net
NaI-12 Net
NaI-13 Net
NaI-15 Net
NaI-16 Net
NaI-17 Net
NaI-18 Net
NaI-19 Net
NaI-21 Net
NaI-22 Net
Detector
Note: Average StDev is equal to the pooled standard deviation