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CCP-TP-046

Revision 4

CCP
Mobile IQ3 System
Calibration Procedure

EFFECTIVE DATE: 02/09/2011

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PRINTED NAME

APPROVED FOR USE

RECORD OF REVISION

Revision Number	Date Approved	Description of Revision
0	02/12/2003	Initial Revision
1	03/21/2003	Changes in response to CBFO comments
2	11/16/2006	Revised to make the responsibilities of users consistent with those outlined by the Waste Isolation Pilot Plant Hazardous Waste Facility Permit requirements resulting from the Section 311/RH PMR, to incorporate procedure consistency changes and improve procedure flow.
3	09/14/2009	Revised to reflect movement of system from the Savannah River Site to the Oak Ridge National Laboratory. Revised calibration and total measurement uncertainty documents. Corrected editorial and typographical errors.
4	02/09/2011	Revised to reflect changes to Calibration Procedures due to NDA2000 Software upgrade.

TABLE OF CONTENTS

LIST OF ATTACHMENTS

1.0 PURPOSE

This document contains the operating instructions to calibrate the Mobile IQ3 system using the NDA2000 Operations Software. The IQ3 system is used to quantify the activity of individual radioisotopes present in the waste sample. Three coaxial germanium (SEGe) detectors are used to quantify the activity of individual radioisotopes present in the waste container. The Multi-Group Analysis software and three Low Energy Germanium (LEGe) detectors are used for the relative isotopic analysis. The assay must meet the requirements for CCP-PO-002, *CCP Transuranic Waste Certification Plan,* for shipment to the Waste Isolation Pilot Plant (WIPP).

1.1 Scope

This procedure specifies instructions to operate the MCS Mobile IQ3 system which includes system start up, using NDA2000, and calibration of the equipment.

2.0 REQUIREMENTS

2.1 References

Baseline Documents

- EPA/520/1-80-012, *Upgrading Environmental Radiation Data*, Washington, D.C., Office of Radiation Programs, U.S. Environmental Protection Agency
- MCS-IQ3-CALIB-2009, *Calibration Report for the MCS IQ3*
- MCS-IQ3-TMU-2009, Total *Measurement Uncertainty for the MCS IQ3*
- CCP-QP-005, *CCP TRU Nonconforming Item Reporting and Control*

Referenced Documents

- CCP-PO-002, *CCP Transuranic Waste Certification Plan*
- CCP-QP-002, *CCP Training and Qualification Plan*
- CCP-QP-008, *CCP Records Management*
- CCP-TP-047, *CCP Mobile IQ3 Gamma Scanner Operation*

2.2 Training Requirements

2.2.1 Personnel performing this procedure will be trained and qualified in accordance with CCP-QP-002, *CCP Training and Qualification Plan*, prior to performing this procedure.

2.3 Equipment List

2.3.1 NDA Analysis Computer System and ancillary equipment

2.3.2 Three (3) SEGe detectors

2.3.3 Three (3) LEGe detectors

2.3.4 Attenuator Caps

- Three (3) for SEGe detectors
- Three (3) for LEGe detectors

2.3.5 IQ3 Mechanism including the following components:

- Shield
- Sample rotator
- Three (3) transmission sources (Ba-133, nominally 10 milliCurie [mCi] each)
- Transmission source shutter assembly
- Conveyor system

2.3.6 Nuclear Instrument Module (NIM) bins including:

- Three (3) Power supplies
- Six (6) Analog to Digital Converters (ADCs)
- Six (6) Spectroscopy Amplifiers
- Six (6) High Voltage (HV) Bias Supplies
- Three (3) Acquisition Interface Modules (AIMs)
- Two (2) Dual Multi-Spectrum Storage (DMSS) Modules
- One (1) Reference Pulser

2.3.7 Light Stack

- Amber light indicates system motion
- Red light indicates transmission shutter is open
- White light indicates data acquisition
- Blue light indicates liquid nitrogen fill system is ON
- Cycling through lights indicates an EMERGENCY STOP

2.3.8 Six (6) efficiency calibration standards (line sources) obtained from suppliers maintaining a nationally-accredited measurement program and calibration matrix drums in accordance with the calibration/validation test plan.

2.4 Software

2.4.1 NDA2000 Operations

2.4.2 Operating System

2.4.3 Genie 2000, Gamma Acquisitions and Analysis

2.5 Precautions and Limitations

2.5.1 The operator shall be aware of the following hazards:

- High-voltage bias supplies
- Liquid nitrogen
- Radioactive transmission sources
- The IQ3 system will start without audible warning. Access to the Equipment Bay shall be controlled by the Nondestructive Assay (NDA) Operator.
- Moving parts (e.g., shield door, conveyer)

2.6 Prerequisite Actions

NOTE
Prerequisite action steps may be performed in any order and may be repeated, as required.

2.6.1 Equipment Bay

[A] Verify power on the electrical cabinet, and all lamps are illuminated (24 volts alternating current [VAC], 24 volts direct current [VDC], 120 VAC, System ON).

[B] Verify that the two EMERGENCY STOP Buttons are operative.

[C] Verify the system interlocks operate properly.

[D] Ensure Central Processing Unit (CPU)/Manual switch is in CPU mode.

[E] Ensure that the attenuator caps are removed from the SEGe and LEGe detectors.

2.6.2 Control Room

[A] Ensure that power to NIM bin, computer, and ancillary equipment is ON.

[B] Verify that the EMERGENCY STOP Button is operative.

[C] Verify the stack light indicators operate (all the lights will cycle ON and OFF).

[C.1] **IF** the lights fail to come ON during this process, **THEN** STOP WORK, notify the NDA Lead Operator (LO), **AND** DO **NOT** proceed without NDA LO concurrence.

[D] Verify that the HV inhibit indicators on the six detectors are OFF.

2.7 Definitions

2.7.1 None.

3.0 RESPONSIBILITIES

3.1 NDA Operator

3.1.1 Performs Calibration measurements.

3.1.2 Notifies the NDA LO as required by this procedure or of any abnormal condition.

3.1.3 Transfers data to the NDA Expert Analyst (EA) for review and reanalysis, as required.

3.2 NDA EA

3.2.1 Reviews the data and determines acceptance of the calibration results and generates the Calibration and Validation Report, or the Calibration Verification Report.

3.2.2 Requests the NDA LO to obtain additional calibration data, if necessary, and specifies the standard content(s) and configuration(s) to be used.

3.3 NDA LO

3.3.1 Reviews the initial data and investigates instances when calibration is stopped or aborted.

3.3.2 Obtains a list of standards to be used in calibration runs, count times, and number of runs, from the NDA EA and ensures the required drum is correctly loaded and available prior to beginning calibration.

3.3.3 Investigates and resolves abnormal occurrences.

3.3.4 Provides direction for operators to mitigate or correct abnormal conditions.

3.3.5 Keeps Vendor Project Manager (VPM) informed of any abnormal occurrences and actions to be taken to mitigate or correct abnormal conditions.

3.4 Facility Records Custodian

3.4.1 Receives, processes, and transmits all records generated by this procedure in accordance with CCP-QP-008, *CCP Records Management*.

4.0 PROCEDURE

NOTE

Calibration and Frequency

Calibration of the IQ3 is performed and documented as described in this procedure. Calibration standards shall be from suppliers maintaining a nationally accredited measurement program. A calibration is performed once prior to assaying drums. The IQ3 must be recalibrated and/or a successful calibration verification performed prior to drum assay if the following conditions occur:

- Major system repairs and/or modifications
- Replacement of measurement's system's components, (e.g., detector, neutron generator or supporting electronic components), that have the capacity to affect data
- Significant changes to the system's software
- Relocation of the system
- Failure of the Quality Assurance (QA) performance measurement
- Any other change that might affect the energy calibration

System calibrations are documented in the operational logbook. Calibration and verification results will be summarized and documented in either a Calibration and Validation Report, or a Calibration Verification Report. The use of the attenuator caps will be specified in the operational logbook. Calibrations, except for energy calibrations, must be performed with and without the attenuator caps.

NOTE

Reports generated during this procedure may be printed as needed and included in the appropriate report.

4.1 Gamma Energy Pre-Calibration

NDA Operator

4.1.1 Refer to CCP-TP-047, *CCP Mobile IQ3 Gamma Scanner Operation* as needed for startup sequence.

4.1.2 Double-click on the NDA2000 SETUP icon on the desktop, **AND** perform the following:

[A] Select the GAMMA tab.

[B] Select the CALIBRATION SOURCE CERTIFICATE button.

[C] Select the NEW Button.

4.1.3 Enter certificate name (e.g., All Lines)

4.1.4 USING the Certificates of Calibration for all the line sources, sum the activities, **AND** enter the following:

[A] Nuclide

[B] Energy (e.g., 59.5 kilo-electron Volt [keV])

[C] Summed Rate (e.g., 65866.5 gammas/sec)

[D] Uncertainty (percent [%])

[E] Half-Life (years [yrs])

[F] Uncertainty (yrs)

[G] Units (select years)

4.1.5 Select ADD ROW.

4.1.6 Repeat steps 4.1.2 through 4.1.5 for each nuclide listed on the Certificates of Calibration.

4.1.7 Select SAVE.

4.2 Energy Calibration for Gamma-Ray Analysis

NOTE

Copies of the Certificates of Calibration for the six line sources used for calibration will be included in the Calibration and Confirmation Report.

The gamma-ray energy calibration is set from the GENIE 2000 Software and requires the use of one or more sources that emit gamma-rays with energies between approximately 50 keV and 2,000 keV.

The energy calibration extends from a nominal range of 0 to 2,000 keV and is set manually, using GENIE 2000 Software.

The line sources will be placed in the Calibration Matrix Drum (typically foam) according to NDA EA direction and recorded for all energy calibrations.

Minor adjustments to system components may be made by the NDA Operators during the establishment of system calibration parameters, including system gain and count times.

4.2.1 Place the line sources into the Calibration Matrix Drum as directed by the NDA EA (see Attachment 1 and Attachment 2 for placement reference).

4.2.2 Have the Forklift operator load the Calibration Drum on the IQ3.

4.2.3 Double click the Gamma Acquisition and Analysis icon and select OPEN Data Source from the File drop-down menu, AND perform the following:

[A] Select the DETECTOR SOURCE button.

[B] Selected DET 1, as the Data Source, **AND** click on the OPEN button.

4.2.4 Select ADJUST from the MCA drop-down menu to adjust the settings to produce a 4,000 channel spectrum with approximately 0.5 keV/channel and an approximately ZERO offset.

4.2.5 Select START to obtain a spectrum of the reference sources for at least 300 seconds, **OR** as directed by the NDA EA.

4.2.6 Select SETUP, from the Calibrate drop down menu, **AND** establish the following settings:

[A] Energy Units set to keV.

[B] Tolerance Units set to ENERGY.

[C] Analysis Sequence Description set to ENERGY CALIBRATION REPORT.

[D] Calibration settings:

[D.1] Energy Calibration set to 1.5 keV.

[D.2] Efficiency Match set to 1.0 keV.

[E] Tail Curves set to NONE.

[F] Continuum set to STEP.

[G] Channels set to 4.

4.2.7 Select the OK button to accept the settings.

4.2.8 Select ENERGY ONLY CALIBRATION, from the Calibrate drop down menu, **AND** perform the following:

[A] Position the cursor on the appropriate energy peak, **AND** click on the CURSOR button to automatically determine the Peak Channel.

[B] Enter the energy value in the ENERGY box, **AND** click on the ACCEPT button.

[C] Repeat steps 4.2.8[A] and 4.2.8[B] for the additional energy peaks.

[D] Select the OK button to accept the Two-Point Calibration.

4.2.9 Select ENERGY FULL BY CERTIFICATE FILE from the Calibrate drop down menu, **AND** perform the following:

[A] Open the Certificate File created during Gamma Energy Pre-Calibration.

[B] Selected the AUTO button.

[C] Select the SHOW button and set the “Order of the Polynomial” to 3.

NDA EA

[D] Review both the Energy and Shape Curves to determine if any discordant points exist.

[E] Delete any points that are discordant, for assignable cause, **AND,** include in the Calibration and Confirmation Report.

NDA Operator

[F] Print the amended Energy and Shape Calibration Curves to be included in the Calibration and Confirmation Report.

[G] Select the USE-RESULTS button to accept the Energy and Shape Calibration Curves.

[H] Select the OK button to finish the Energy Calibration.

4.2.10 Select SAVE, from the File drop down menu, to preserve the Data Source new Energy Calibration.

4.3 Reference Peak Calibration

4.3.1 Unload the Calibration Drum with mixed-nuclide gamma sources from the IQ3.

4.3.2 Double-click the Gamma Acquisition and Analysis icon and select OPEN Data Source from the File drop down menu, **AND** perform the following:

[A] Select the DETECTOR SOURCE button.

[B] Select DET 1, as the Data Source **AND** click on the OPEN button.

4.3.3 Set the Pulser Amplitude to produce a spectral peak at approximately 1800 keV.

4.3.4 Set the Pulse Rate to approximately 100 Hertz (Hz).

4.3.5 Acquire a spectrum of the Pulser peak alone, for at least 100 seconds.

4.3.6 Select PEAK LOCATE>UNIDENTIFIED 2nd Diff., from the Analyze drop down menu, **AND** perform the following:

[A] Check "☒" for the GENERATE REPORT box.

[B] Select the EXECUTE button.

4.3.7 Select PEAK AREA>SUM/NON-LINEAR LSQ FIT from the Analyze drop down menu, **AND** perform the following:

[A] Check "☒" for the GENERATE REPORT box.

[B] Select the EXECUTE button.

4.3.8 Select PRINT REPORT WINDOW, from the File drop down menu, to generate a printout.

4.3.9 Forward the printout to the NDA EA for review and inclusion in the Calibration and Confirmation Report.

4.3.10 Double-click on the REFERENCE PEAK CALIBRATION icon on the desktop.

4.3.11 Using the File menu, **OR** the OPEN TOOLBAR icon on the desktop, OPEN the COAX Gamma Detector 01 as Data Source.

4.3.12 From the Reference Peak Calibration Program perform the following:

[A] Select the spectral peak at approximately 1,800 keV, **AND** designate it as the REFERENCE PEAK.

[B] Select the REFERENCE PEAK from the peaks in the lower grid, **AND** check "☒" for the SELECT AS REFERENCE box.

[C] Select PULSER as the Source Type.

4.3.13 From the File drop down menu save the Reference Peak information in the Detector Data Source.

4.3.14 Repeat Steps 4.3.1 through 4.3.13 for each applicable detector.

4.3.15 EXIT the NDA2000 Reference Peak Program.

4.4 Transmission Peak Calibration

4.4.1 Have a Forklift Operator load a empty 55-gallon drum on the IQ3.

4.4.2 Double-click on the NDA2000 SETUP icon on the desktop, **AND** perform the following:

[A] Select the GAMMA tab.

[B] Select the TRANSMISSION SOURCE CERTIFICATION button.

[C] Select the NEW button.

[D] Enter the CERTIFICATE DESCRIPTION

[E] Select the EXTRACT FROM LIBRARY button to populate the table.

[F] Select the SAVE button to EXIT the editor.

4.4.3 Double-click on the NDA2000 OPERATIONS icon on the desktop to start the count.

4.4.4 Select the GREEN light on the tool bar, **AND** perform the following:

[A] Select the TRANSMISSION CALIBRATION.

[B] Set the Assay Count Time to 100 SECONDS, or as appropriate, **AND** select the REAL button.

[C] Set Scanning Mode to NORMAL SCAN, **AND** select the AUTOMATIC button.

[D] Set Transmission Mode to TWO PASS, Source to CERTIFICATE DESRCIPTION NAME, **AND** Count Time to 100 SECONDS, or as appropriate.

4.4.5 Select START ASSAY, **AND** perform the following:

[A] Enter the ITEM ID at the Basic Information Screen.

[B] Select DONE when completed.

4.4.6 **AFTER** the data are acquired,
THEN double-click the NDA2000 CALIBRATION icon on the desktop to perform the calibration.

4.4.7 In the Select Calibration Window, Select TRANSMISSION.

4.4.8 Select PERFORMANCE CALIBRATION from the CALIBRATIONS OPERATIONS menu.

4.4.9 Select the detector category, geometry, container, and transmission source, as appropriate from the screen.

NOTE

The Transmission Source Wizard displays the Transmission Calibration Data, which is the observed count rate for each transmission peak through the empty container.

4.4.10 Select NEW TRANSMISSION CALIBRATION to start the Transmission Source Wizard.

4.4.11 Enter a description of the new calibration, **AND** press NEXT.

4.4.12 Check the box next to the date the calibration counts were performed, **AND** press NEXT.

4.4.13 Select FINISH to save the Transmission Calibration Data.

4.4.14 Unload the empty 55-gallon drum from the IQ3.

4.5 Efficiency Calibration

4.5.1 Ensure that all calibration sources are traceable to a nationally accredited measurement program.

4.5.2 Assemble a Calibration Drum as indicated by the NDA EA, **AND** insert the calibration sources into their assigned tube positions, as necessary.

NOTE

A uniform distribution of source material is approximated by using line sources (sources with uniformly deposited radioactivity along the length of the rod) placed vertically in the drum at specific radial distances such that when the drum is rotated the radiation response seen by the detector is uniform. The system will automatically perform a full efficiency calibration and display a plot of the results. The process is repeated for all four Calibration Matrix Drums. The count time for each Calibration Matrix Drum will be determined by the NDA EA and entered in accordingly. The NDA EA will provide tare and gross weights of the Calibration Matrix Drums.

4.5.3 Have the Forklift Operator load the selected Calibration Drum onto the IQ3.

4.5.4 Double-click on the NDA2000 OPERATIONS icon on the desktop, **AND** perform the following:

[A] Click on the first Toolbar icon (a GREEN light) **AND** select EFFICIENCY CALIBRATION from the Count Type drop down menu.

[B] On the Assay-Routine Screen perform the following:

[B.1] Select the appropriate Type.

[B.2] Select the appropriate geometry.

[B.3] Set the appropriate Gamma Preset Counting Time.

[B.4] Select Start Assay

[C] On the Item Identification screen, enter the following:

[C.1] Item ID.

[C.2] For Description 1, enter Reference Source ID Number(s).

[C.3] For Description 2, enter Reference Source ID Number(s).

[C.4] Site Location.

[C.5] Select the appropriate Matrix Type.

[C.6] Drum Gross Weight.

[C.7] **IF** the tare weight DOES **NOT** match the tare weight provided by the NDA EA,
THEN perform the following:

(a) Click on the NDA2000 Setup icon on the NDA2000 main screen tool bar.

(b) Click on the GENERAL tab.

(c) Click on CONTAINER TYPE button.

(d) Select “55 Gallon.”

(e) Change tare weight to the tare weight provided by the NDA EA.

(f) Select SAVE, CLOSE, **AND** click on the “x” at the top right of the screen to EXIT setup.

[C.8] For Sample Type, select NONE.

[C.9] Select the appropriate Declaration.

[C.10] Click on the DONE button to start the measurement.

[D] Upon completion of the measurement, print the results, **AND** forward the printout to the NDA EA for review and inclusion into the Calibration and Confirmation Report.

[E] Remove the Calibration Drum from the IQ3.

[F] CLOSE the NDA2000 Operations Software.

[G] Double-click on the NDA2000 CALIBRATIONS icon on the desktop, **AND** perform the following:

[G.1] Select EFFICIENCY from the Calibration drop down menu.

[G.2] Select PERFORM CALIBRATION from the Calibration Operations drop down menu.

[G.3] Select Detector Category, Container, and Geometry, as appropriate.

[G.4] Select NEW EFFICIENCY CALIBRATION to start the Efficiency Calibration Wizard.

[G.5] Enter an appropriate Calibration description, **AND** click on the NEXT button.

[G.6] On the Counts screen, check "☒" for all the relevant efficiency measurements, **AND** click on the NEXT button.

[H] With NDA EA supervision, examine the plot and identify any discordant data points.

[I] With NDA EA supervision, click on the OVERPLOT button to display multiple calibration curves.

[I.1] **IF** any discordant data points are noted and can be deleted for assignable cause,
THEN click on the BACK button, **AND** uncheck them for the list.

[I.2] **IF** data points were deleted,
THEN click on the NEXT button, followed by the FINISH button to save the new calibration.

[J] Print out the report, **AND** forward it to the NDA EA for approval, designation as the default calibration for the appropriate matrices, and inclusion in the Calibration and Confirmation Report.

4.5.5 Repeat steps 4.5.1 through 4.5.4 for each Calibration Matrix Drum, as required.

4.6 Confirmation of Calibration

NOTE

Calibration Confirmation shall be performed after each calibration or re-calibration using at least one non-interfering, representative matrix. Matrices representative of the calibration range may also be tested as required.

A minimum of two standard strengths or loadings is required: One to cover the lower one-third of the operating range, and one to cover the upper one-third of the operating range. Standards used for calibration will **NOT** be used for confirmation.

The NDA LO will obtain the detailed information concerning the number and strength of the standards, the matrix drum configurations, and the number of replicates from the calibration/verification plan.

4.6.1 Obtain the appropriate standard/matrix configuration as indicated by NDA LO.

4.6.2 Verify that the standard(s) is from a supplier maintaining a nationally accredited measurement program.

4.6.3 Locate the standard(s) within the matrix as indicated by the NDA LO.

4.6.4 Perform Background/Transmission, and Instrument Performance checks as described in CCP-TP-047, as required.

4.6.5 Perform replicates for each standard/matrix configuration as indicated by the NDA LO per the standard assay procedure described in CCP-TP-047.

4.6.6 Evaluate the confirmation measurement results using the calibration confirmation criteria specified in CCP-PO-002.

[A] **IF** the criteria are met,
THEN the calibration is confirmed and NO further action is required.

[B] **IF** the criterion is NOT met,
THEN evaluate the cause.

4.6.7 Submit the report(s) to the Facility Records Custodian.

4.7 Verification of Calibration

NOTE

Verification of the calibration is performed using at least one standard matrix configuration. Calibration standards or secondary standards that have been correlated with the calibration standards can be used.

Calibration Verification should be performed after any one of the following occurs: major system repairs and/or modifications, replacement of the measurement system's components, (e.g., detector, supporting electronic components that have the capacity to affect data, significant changes to the system's software, and relocation of the system).

The NDA LO will obtain the detailed information concerning the number and strength of the standards, the matrix container configurations, and the number of replicates from the calibration/verification plan or an NDA EA.

4.7.1 Obtain the appropriate standard matrix (e.g., calibration drum) configuration as indicated by NDA LO.

4.7.2 Verify that the source standard(s) is from a supplier maintaining a nationally accredited measurement program.

4.7.3 Locate the standard(s) within the matrix as indicated by the NDA LO.

4.7.4 Perform Background/Transmission and Instrument Performance checks as described in CCP-TP-047, as required.

4.7.5 Perform replicates for each standard/matrix configuration as indicated by the NDA LO as per the standard assay procedure described in CCP-TP-047.

4.7.6 Evaluate the verification measurement results using the calibration verification criteria specified in CCP-PO-002.

[A] **IF** the criteria are met,
THEN the calibration is verified and NO further action is required.

[B] **IF** the criteria are NOT met,
THEN evaluate the cause.

4.7.7 Submit the report(s) to the Facility Records Custodian.

Facility Records Custodian

4.7.8 Receive, process and transmit the report(s) in accordance with CCP-QP-008.

5.0 RECORDS

5.1 Records generated during the performance of this procedure are maintained as QA records in accordance with CCP-QP-008. The records are the following:

5.1.1 QA/Non-Permanent:

[A] IQ3 Calibration and Validation Report

[A.1] Energy Recalibration, as applicable

[A.2] Reference Peak Calibration, as applicable

[A.3] Efficiency Calibration, as applicable

[A.4] Transmission Calibration, as applicable

[A.5] Confirmation of Calibration, as applicable

[A.6] Calibration Verification, as applicable

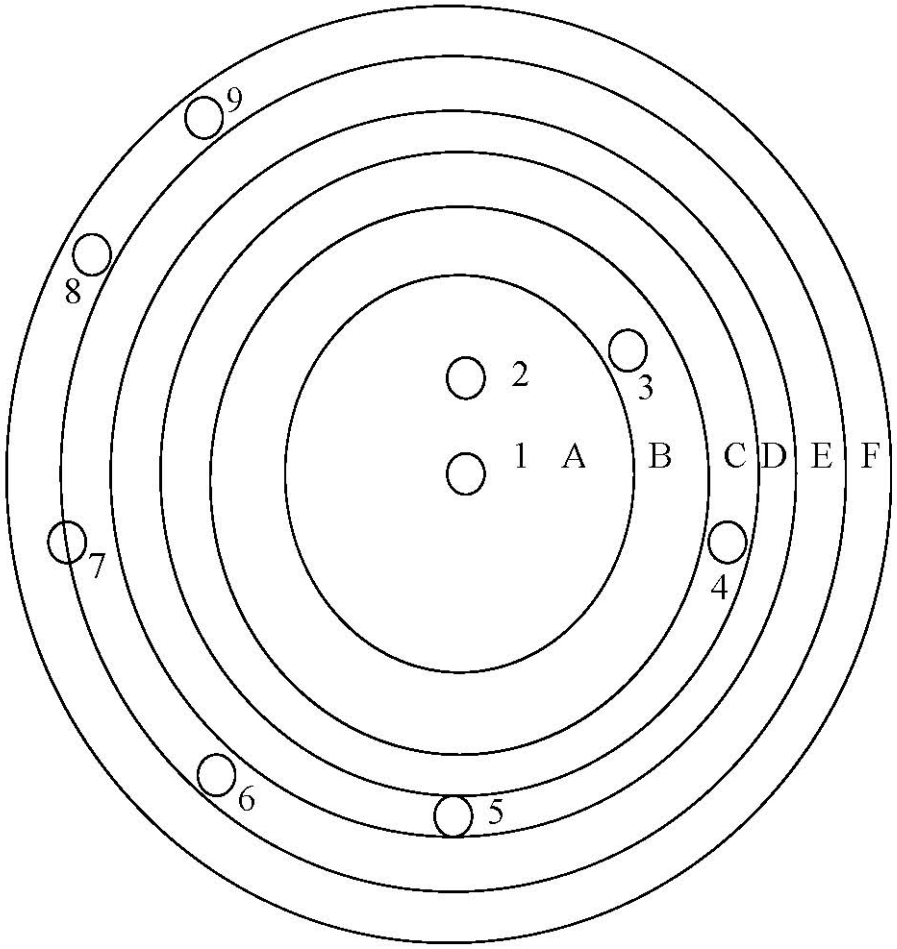
[B] IQ3 Calibration Verification Report

Attachment 1 – Tube Location and the Distance from the Center

Tube Location	Distance from Center (inches)*
1	0
2	2.3
3	5.6
4	7.3
5	8.6
6	9.8
7	10.2
8	10.5
9	10.8

*The radial distance from the drum center to the center of each of the tubes is provided in this table.

Attachment 2 – Top View Cross Section of a Calibration Drum



Each calibration drum is divided into six shells of equal volume labeled A, B, C, D, E, and F. The individual tubes are number 1 through 9. The radial distance from the drum center to the center of each of the tubes provided in Attachment 1, Tube Location and the Distance from the Center.