
GAMMA-RAY-EMITTING RADIONUCLIDES IN SOLUTION

Purpose

The purpose of this procedure is to describe the measurement and reporting procedures for gamma-ray-emitting radionuclides using the NIST "4 π "- γ ionization chamber.

Scope

The NIST "4 π "- γ ionization chamber (IC) is a well-type pressurized instrument which is used for measuring the radioactivity (disintegrations per second) of gamma-ray-emitting radionuclides. Scheduled calibration services for submitted samples of gamma-ray emitters are offered as a complement to the Standard Reference Material (SRM) program. These calibration services are 43010C (half-lives longer than 15 days) and 43020C (half-lives shorter than 15 days). The range of activity for the gamma-ray-emitting radionuclides is 0.4 - 60 MBq and the nominal uncertainty range is 0.47 - 2.27 percent (expanded uncertainties, $k = 2$).

The IC uses calibration factors (K-values) for each radionuclide previously established by direct methods of activity measurements (Hoppes 1984; NCRP 1985). Records of the direct measurements of activity used to determine K-values are kept in the K-value Record Book. For further information on specific methods refer to NIST SP 250-10. IC calibrations are checked with those of other national standardizing laboratories through international measurement comparisons, particularly those conducted by the Bureau International des Poids et Mesures (BIPM) with the Système de Référence (SIR; <http://www.bipm.org/en/bipm/ionizing/radionuclides/sir/>). Results of these comparisons are obtainable from the Appendix B ("Key and Supplementary Comparisons") of the Key Comparisons Database (KCDB, http://kcdb.bipm.org/AppendixB/KCDB_ApB_search.asp) maintained by the BIPM. Results of NIST participation are recorded and summarized in the SIR Intercomparison Notebook.

The gamma-ray-emitting radionuclides which may be submitted to NIST for IC calibrations are those listed in the K-value Record Book.

Definitions

K-value: The relative calibration factor, K_R , is given by R_R/R , the ratio of the measured responses of the ^{226}Ra reference source and a known one becquerel (1 Bq) of a stated radionuclide.

Equipment

Equipment manuals are maintained in Building 245, Room B47. Current instrument models and serial numbers are listed in the IC Lab Notebook. Current software and database versions are also listed in this notebook and referenced by name and file date.

Hardware

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- The NIST "4 π "- γ (re-entrant) cylindrical ionization chamber (described in NIST SP 250-10)
- Multi-range electrometer
- Comparator
- Start Stop scaler
- Calendar scaler
- Printout controller
- Sample holder
- A series of 10 radium-226 reference sources

Equipment Quality Control

Multi-range electrometer: Individual measurements of submitted sources are evaluated as ratios to the appropriate reference source, therefore normally fluctuating environmental conditions do not impact the measurement result. In order to scale the background measurements to the source measurements, the sensitivity ratios (referred to as "capacitor ratios" for historical reasons) should be known and remain consistent. The capacitor ratios are measured approximately every five years by observing the ratios of measurements of a single radium-226 reference source (RRS) on multiple ranges, using an RRS that is appropriate for the range. The result of a check for consistency is recorded in the IC lab notebook.

Radium-226-reference sources: K-values are sometimes determined for only one RRS. K-values for other RRSs are calculated using RRS ratios. RRS ratios are checked approximately every five years by measuring more than one RRS on the same capacitor sensitivity setting. To reduce hand-dose to the operator, it is also acceptable to measure the RRS ratios using the NIST automated ionization chamber. The results will be equivalent. The result of the measurements and comparison with historical data is recorded in the IC Lab notebook. Failure of an individual RRS would be identified as a change in ratio with two other RRSs.

Software

Iconline.exe: Iconline is a FORTRAN program running on a desktop computer. It is used to collect data from the ionization chamber as well as input by the user. Data is stored offline for further processing

Ionol.exe: Ionol is a FORTRAN program running on a desktop computer. It is used to process the data file created by Iconline using further input from the user and information from ICDATBAS.

ICDATBAS: ICDATBAS is a data file containing the most recent K-values listed in the K-value Record Book.

Calculate Chamber A Height Effect V9.xlsx: Calculate Chamber Height Effect is an excel spreadsheet used to calculate the correction required if the height of the source holder is different during the measurement than it was at the time of determination of the K-value (see Fitzgerald 2012).

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Validation

The validation of equipment is performed upon installation and subsequent to any change in equipment. The proper recording of data is validated by observing the results on the calendar scaler and comparing this value to the value recorded by the software.

Validation of processing software is performed by manual calculation of an experimental result and comparison of this value to that obtained from the program. This is performed upon the initial version and subsequent to any changes in the program.

Results of validations are recorded in the IC Lab notebook.

Safety*Radiation safety*

Radiation safety training and assessment services are provided by the NIST Gaithersburg Radiation Safety Division (GRSD). The room used for ionization chamber calibrations is designated as a Radiation Area. A countertop lead cave is used to house samples during measurement and to store the ^{226}Ra reference sources. In the case of high-activity sources, a lead-lined "L" shield might be used while changing samples.

Procedures*Source Container and Contents*

The solution to be assayed in the NIST IC should have as nearly as possible the same composition as the standards used to calibrate the chamber (described in NIST SP 250-10). The gamma-ray-emitting radionuclide solution should be 5.0 ± 0.2 mL in a standard 5 mL ampoule. The specifications of these ampoules are as follows:

body diameter	16.5 ± 0.5 mm
wall thickness	0.60 ± 0.4 mm
barium content	less than 2.5 percent
lead oxide content	less than 0.02 percent
other heavy elements	trace quantities

Description of the Actual Measurement Process

The stability of this chamber, although excellent, is always monitored by measuring its response to a reference source of ^{226}Ra in equilibrium with its daughters whenever calibrations are made. The activity of the sample is taken to be proportional to the ratio of the chamber response (current) for the sample to that for the reference source. The relative calibration factor, K_R , is

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given by R_R/R , the ratio of the measured responses of the ^{226}Ra reference source and a known one becquerel of a stated radionuclide.

The activity, A , of the radionuclide in a sample to be measured, S , is given by $K_R R_S$, where R_S is the response (current) for the sample relative to that for the radium source. Background currents, measured just before and after counting the sample and radium, must be subtracted from the response for each. K_R diminishes from the value at the time of the original calibration with the 1600 year half life of the ^{226}Ra , and a decay correction for this must be made.

Because the activity of the sample is always measured as a proportional response to a reference source of ^{226}Ra , maintenance of the standard is not dependant on the integrity of specific electronic components.

Preliminary

- Customer contact: give specifications for chemical composition and activity limits, source must either be provided as 5 mL solution in a NIST-type 5 mL flame-sealed ampoule, or transferred to one by NIST.
- NIST paperwork and acceptance procedure: submit completed NIST 364, Radioactive Material Request, for approval before arrival of materials. Specifically notify the Chief of GRSD or designate of the arrival and departure of all isotopes of plutonium and uranium. Complete any other necessary forms (NIST 64, Test Record; NIST 77, Calibration and Test Fee Computing Form; NIST 796A, Shipper's Declaration for Radioactive Materials, etc.).
- Test folders: request test folder on receipt of material to be calibrated. Note dates of material received and returned.
- Run program KVUPD or examine database to determine appropriate electrometer range and ^{226}Ra reference source to be used for measurement.
- Schedule time to use the IC by blocking and initialing the required amount of time on the calendar in 245/B47. Possible conflicts should be resolved in person with other users. Generally shorter half-life sources take precedence.
- Deliver source, or a related sample, to the appropriate person to be measured on a calibrated germanium spectrometry system according to instructions for an impurity measurement. The results and source are delivered to the analyst at the completion of this measurement.

Receiving Instructions for Radioactive Solution Samples

All radioactive sources arriving at NIST are delivered to GRSD where they are examined to determine the radiation level and possible contamination of containers and packaging.

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After GRSD releases the source to the analyst, the analyst in turn should verify that the correct radionuclide has been shipped in the activity range which has been previously discussed with the customer. Quality control procedures that should be implemented by the analyst are as follows:

1. Re-examine the source for pin holes of any type and check the ampoule seal.
2. Inspect the label attached to the primary container. This helps to insure that it is the required radionuclide and the activity amount is correct.
3. Read technical data sheets and package inserts accompanying the radionuclide package carefully for special instructions on stability, quality inspection, and chemistry of the product. This information should conform to the calibration guidelines specifying the physical and chemical properties of the submitted sample solution.

Providing that no defects are observed, the sample may be transported for measurement in the NIST "4 π "- γ ionization chamber. In the case of above criteria not being met, contact customer.

Step-by-Step Operating Instructions for the NIST "4 π "- γ Ionization Chamber

Sign in using the IC Log Book.

Starting the Ionization Chamber System

The system is started from the connected computer using the following procedure:

1. Check schedule and observe system to verify it is not currently in use.
2. Verify computer system time. If necessary, correct the time using NIST network time.
3. Click on desktop icon "iconline." The system is started.
4. Answer Y or y for default values, N or n to change and supply answers. Generally use default values. Press return after each answer.

Questions are as follows:

New Bkg-O; Skip 1

BKGS=-1

Time: Day#

Input Start of Calendar Scalar HR, MIN, SEC

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Reset clock on clock scalar

Computer Time: HR: MIN: SEC

Statement of agreement between computer and calendar clock

Answer Y or y if OK, N or n to reenter cal. start time

Enter Sample #: O-BKG, 999-DONE, -999 change CAPR, PTS.

Setting the Electrometer

The coulomb dial on the electrometer must be set to 10^{-9} for all measurements.

Set the sensitivity dial to the proper setting. The range of sensitivity is 1 to 1000. The sensitivity setting for background is 1. Refer to Table 1 for determining the 1-1000 range settings.

Table 1. Sensitivity Range Settings

“Capacitor Ratio”	Radium Reference Source*
1	Background
10	3, 10, 20
100	20, 50, 100, 200
1000	200, 500, 1000, 5000

*The number of the radium reference source used is approximately equivalent to the number of micrograms of radium per source.

The “capacitor ratio” and the ^{226}Ra reference source to be used are determined based on the activity provided by the supplier when the sources are submitted, with appropriate corrections for decay.

Background Measurements

Background measurements are made initially and should be examined before continuing. If the background is inconsistent, or abnormally different, investigate the cause. High background may be due to an unshielded source in the vicinity. It is possible to make measurements under high background conditions by measuring the background more often.

Measuring Reference Source

1) Place the ^{226}Ra reference source into the rigid, tight-fitting Lucite source holder and lower into the chamber. Align the markings on the source holder with those on the instrument shielding. (Note – The source holder is an integral part of the IC system. Care must be taken to place the

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holder shaft in a position where it will not roll and fall. If a cart is used, it must have walls or a tray to restrict movement of the holder)

2) Change the timer controller switch from the manual to the automatic mode. Press Start. (This step is repeated each time a radium reference source or solution sample is measured.)

3) After the designated number of measurements has been made on the ^{226}Ra reference source, or on a sample, return the timer controller switch to manual. This is done at the end of every set of measurements. The program will display the average count time and standard deviation. The sample number will be requested for the next sample to be counted. To exit before the requested number of cycles, press the <CTRL>-C on the computer.

4) Remove the holder from the chamber and replace the ^{226}Ra reference source with the sample to be assayed.

Measuring the Sample

When placing the solution sample in the chamber, observe the ampoule to make certain that no liquid is in the neck of the ampoule. The sample is then counted the same number of times in each set of measurements as the radium reference source. At least four sets of 10 measurements should be made on the sample, with an equivalent number of measurements made on the radium reference source.

After all measurements have been completed on the submitted sample and the radium reference source, it is recommended that background measurements be repeated.

Data Collection

The data collection is under control of Iconline.exe. The start of the calendar scalar and other default parameters are entered, as prompted, by the operator. The operator enters the sample number, number of data points to be collected, and capacitor ratio. If results are not acceptable, the program will notify the user and will not proceed. All data is collected and stored to a disk for off-line analysis.

Measurement of impurities

The following gives a summary of the procedures documented in the *Procedure for gamma-ray spectrometry measurements for activity calibration and impurity measurements using High Purity Germanium (HPGe) detectors*.

Impurity-check measurements of radioactive sources to be calibrated by the ionization chamber are performed using the Radiation Physics Division (RPD) Gamma-Ray Spectrometry System located in Building 245, Room C25 (basement). This system consists of five High Purity Germanium (HPGe) detectors and one Silicon-drifted detector. These detectors are calibrated

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using NIST Standard Reference Materials (SRMs), which are, in turn, validated through measurement comparisons with other National Metrology Institutions and the BIPM through the periodic submission of measured sources to the SIR for gamma-ray emitting sources. The calibration of each of the six detectors is done through the development of efficiency curves for different source geometries and various source-to-detector distances. These efficiency curves are verified monthly or more frequently if needed (such as when there has been a change in the instrument configuration), using previously calibrated ^{57}Co and ^{60}Co point sources to determine the efficiency value, shape (FWHM) and energy calibration reproducibility. Efficiency curves for point sources (solids), 5 mL ampoules (liquids) and 33 cm³ spheres (gas) for gamma-ray emitting sources for the energy range $14 \text{ keV} < E < 3.6 \text{ MeV}$ are available for impurity and activity calibration measurements.

Gamma-ray spectra are acquired using GammaVision™ (version 6.06, as of this writing), a commercially available software developed and marketed by EG&G ORTEC. Data analysis is performed using Genie 2000 from Canberra (operational manual located in laboratory). The choice of data analysis software was based on in-house comparisons of the results obtained by several (3) different software packages and by hand. In addition, the data analysis software used by customers was taken into account. Such commercial products are identified in order to specify these procedures accurately; the RPD does not recommend or endorse any commercial product, nor intend to imply that these products are necessarily the best available for the purposes of these procedures.

The uncertainty on the activity values for the impurities and/or source measurement is obtained by the expanded uncertainty as given by

$$\sigma_{\text{imp / source}} = 3\sqrt{\sigma_{\text{eff}}^2 + \sigma_{\text{measure}}^2}$$

where σ_{eff} is the uncertainty of the efficiency for the measured gamma-ray energies for the impurity and/or source, and σ_{measure} is the uncertainty calculated based on the gamma-ray lines used to determine the activity of the measured impurity and/or source. As impurity levels are generally much lower than the nuclide being calibrated, the uncertainty on the impurity measurement is often on the order of 5 % to 20 %.

Processing the IC Data

The data processing is done using Ionol.exe. The program averages the data for each sample and corrects them for background and radioactive decay (using the established half-life). The sample activity is calculated using the K-value for that radium reference source producing a current approximating that of the sample. The results, with intermediate averages if desired, are printed out.

Correction for impurities is handled by the processing program. Results of impurities are entered as an activity ratio relative to the main radionuclide and a measurement time.

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For K-values determined prior to January 2010, a radionuclide-specific correction factor must be calculated to account for the difference between the current position of the source holder within the ionization chamber and the position of the source holder at the time of the determination of the K-value. The correction factor is described by Fitzgerald (2012). Calculation of the correction factor can be performed with the spreadsheet Calculate Chamber A Height Effect V9.xlsx. The output from Ionol.exe is multiplied by the correction factor, f . Alternatively, the correction factor can be incorporated into the K-value. If this is the case, it is documented in the K-value notebook.

Acceptance Criteria

The data is examined to see if the results show the expected activity, and the standard deviation of repeated measurement on the source is checked to see if it is in the range expected for the radionuclide (normally less than 0.1 percent at 1 sigma). If there is a discrepancy in activity, the source may be measured in a commercially re-entrant ionization chamber, or “dose calibrator,” as a confirmatory measurement. This measurement is performed by placing the sample in the dose calibrator source holder and entering the correct instrument setting for the radionuclide in question. Settings are found on a list attached to the instrument or in the instrument manual. The analyst must be familiar with the use of the dose calibrator and the accuracy of the settings. If the standard deviation is abnormally high, the data is examined for possible trends. The customer is contacted if a discrepancy cannot be resolved.

Calibration Report

At the end of a calibration exercise, the customer will be given a Report of Calibration stating primarily:

- a) the principal radionuclide
- b) reference time and date
- c) method of calibration
- d) certified values for activity
- e) decay-scheme assumptions
- f) assessment of radionuclidic purity
- g) overall uncertainty determinations for calibration

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Reported values are checked against the data on the IC printout and this check is noted by initialing the printout. Drafts of reports are stamped as such and identified by draft number. Results of draft proofreading are noted and initialed on the draft.

Signatures are obtained at the appropriate level and the original report is embossed with a Department of Commerce seal. A copy marked file copy and two marked copies are maintained with the calibration printouts, stored in folders identified by company, radionuclide, date, and service number. The original report is sent to the customer.

Customer Sources

When the calibration service is completed, the technical users are notified, and the calibrated sample returned, if desired by customer. Short-lived radionuclide samples are usually measured by the customer only before transmittal to NIST, for the activity would be too low for measurement after the NIST calibration.

Uncertainty Analysis

The bases for the determination of uncertainties are the *ISO Guide to the Expression of Uncertainty in Measurement* and the *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*.

The purpose of this section is to explain the derivation of the various components of uncertainty. Measurements performed with the "4 π "- γ ionization chamber (IC) must be done under completely fixed conditions. This requires a stable background, no saturation losses due to incomplete charge collection because of recombination at high rates, and corrections for all impurities. Ampoules must be of the standard 5 mL type, the volume in ampoules must be 5.0 ± 0.2 mL, and the ^{226}Ra reference source must be corrected for decay.

Uncertainty in activity measurements may be increased if the contribution of the background is variable, large, or both. The uncertainty quoted is derived from measurement of the variations. The operator of the chamber should make frequent measurements of the background and look for the source of any unusual variations observed.

- K-value (Type A and Type B) – uncertainty in the determination of the K-value, including the measurement of activity by primary methods and measurements to transfer that calibration to the ionization chamber (specific uncertainties for individual K-values are listed in the K-value Record Book)
- RRS ratio (Type A) - uncertainty in the ratio of radium reference sources, when the radium source used in the measurement is different from the radium source used for the determination of the K-value
- Half life (Type A) – uncertainty in the half-life value used for decay corrections

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- N measurements on this sample (Type A) – standard deviation determined from n measurements of activity ratios
- Impurities in this sample (Type A and Type B) - Radionuclidic impurities may or may not have a significant effect, depending on the radiations of the principal and impurity radionuclide and the amount of impurity present. If their half-lives are relatively short, measurements at different times may be used to check consistency. High purity germanium detector systems are used to identify and quantify the significant radionuclidic impurities that may be present. The uncertainty in these measurements is propagated through the calculated effect on the IC results.
- Gravimetric (Type B) – typical uncertainties from gravimetric transfers of solution, if necessary
- Source-height correction (Type B) – uncertainty in the calculation of the correction for the source height, if necessary

Records

Calibration printouts and report copies are stored in folders identified by company, radionuclide, date, and service number.

If the source is returned, the date that the source is shipped back to the customer should be entered into the folder.

Folders of calibration records are maintained in 245/C136 for at least five years.

Documentation

K-Value Record Book – maintained in 245/B109

Ionization Chamber Laboratory Notebook – maintained in 245/B47

Ionization Chamber Log Book – maintained in 245/B47

SIR Intercomparison Notebook – maintained in 245/B109

Procedure for gamma-ray spectrometry measurements for activity calibration and impurity measurements using High Purity Germanium (HPGe) detectors – maintained in 245/C25

References

Hoppes, D. D., Basic Radionuclide Measurements at the United States National Bureau of Standards, Environmental Int. 10, 99-107 (1984).

NCRP Report 58, A Handbook of Radioactivity Measurements Procedures,

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National Council on Radiation Protection and Measurement, Bethesda, Maryland, (1985).

Taylor, B.N., and Kuyatt, C.E. "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297 (1994).

Calhoun, J.M., Radioactivity Calibrations with the NIST "4 π " Gamma Ionization Chamber, NIST Special Publication 250-10, 1987

Fitzgerald, R. NIST Ionization Chamber "A" Sample-Height Corrections, J Res Natl Inst Stand Technol Vol 117 (2012).

Filing and Retention

Calibration printouts are stored in folders identified by company, radionuclide, date, and service number.

If the source is returned, the date that the source is shipped back to the customer should be entered into the folder.

For customer calibration, prepare calibration report and obtain required signatures. Make a copy for customer file and send original.

The RPD Quality Manager shall maintain the original and all past versions of this RPD Procedure.

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Appendix A. Sample Calibration Report



National Institute of Standards & Technology

REPORT OF CALIBRATION

for

COMPANY, INCORPORATED
TOWNESVILLE, STATE

Radionuclide	Linolium-100
Source identification	1234-abc
Source description	Liquid in a NIST borosilicate-glass flame-sealed 5-mL ampoule ^{(1)*}
Solution composition	LoCl ₂ in 0.1 mol·L ⁻¹ HCl ⁽²⁾
Activity	1.235 x 10 ⁵ Bq
Expanded uncertainty (<i>k</i> =2)	1.5 percent ⁽³⁾
Reference time	1200 EST 30 February 2015
Photon-emitting impurities (Activity ratios at reference time)	¹⁰¹ No/ ¹⁰⁰ Lo: (1.2 ± 0.3) x 10 ⁻⁵ ⁽⁴⁾
Half life	123.45 ± 0.06 days ⁽⁵⁾
Measuring instrument	NIST “4π”-γ ionization chamber calibrated by apple π(e+x)-γ coincidence counting

Measurements Performed by

Jeffrey T. Cessna, Physicist

For the Director,
National Institute of Standards and Technology byMichael P. Unterweger, Leader
Radioactivity Group
Physical Measurement LaboratoryLisa R. Karam, Chief
Radiation Physics Division
Physical Measurement LaboratoryGaithersburg, MD 20899
Report Issued: 1 April 2015
Service ID No.: 43010C
NIST Folder No.: xxxxx-xx

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NOTES

- (1) The solution was prepared by Company, Incorporated (CI), Townesville, State. CI reported the mass to be 4.9938 grams.
- (2) Description provided by CI.
- (3) The uncertainty analysis methodology and nomenclature used for the reported uncertainties are based on uniform NIST guidelines and are compatible with those adopted by the principal international metrology standardization bodies [cf., B.N. Taylor and C.E. Kuyatt, *NIST Technical Note 1297* (1994)].

The combined standard uncertainty, $u_c = 0.75$ percent, is the quadratic combination of the standard deviations (or standard deviations of the mean where appropriate), or approximations thereof, for the following component uncertainties:

a) 40 ionization-chamber measurements on this sample	0.02 percent
b) k - value	0.72 percent
c) photon-emitting impurities in this sample	0.02 percent
d) radium reference sources ratio	0.08 percent
e) radium reference source positioning	0.10 percent

The expanded uncertainty, $U = 1.5$ percent, is obtained by multiplying u_c by a coverage factor of $k = 2$ and is assumed to provided an uncertainty interval of approximately 95 percent confidence.

- (4) Limits of detection for impurity gamma rays are:

$$\begin{aligned} &1.2 \times 10^5 \gamma \cdot s^{-1} \text{ between 30 and 110 keV,} \\ &3.8 \times 10^4 \gamma \cdot s^{-1} \text{ percent between 130 and 350 keV, and} \\ &4.6 \times 10^3 \gamma \cdot s^{-1} \text{ percent between 400 and 1800 keV,} \end{aligned}$$

provided that impurity photons are separated in energy by five keV or more from those emitted in the decay of linolium-100. Limits are as of impurity measurement time, 32 January 2015.

- (5) Evaluated Nuclear Structure Data File (ENSDF), February 2015.

For further information, please contact Jeffrey T. Cessna at (301) 975-5539.

NIST Folder No.: xxxxx-xx

Source Identification: 1234-abc

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