1. **Purpose:** This procedure describes the requirements for instrument calibrations and the steps to calibrate various equipment. Instruments and equipment used for quantitative measurements such as dose rates or effluent monitoring must be calibrated periodically for the radiation measured [10 CFR 20.1501(c)]. MU shall possess calibrated radiation detection and measuring instruments that include survey and monitoring instruments and quantitative measuring instruments needed to monitor the adequacy of radioactive materials containment and contamination control.

2. **Scope:** Radiation Workers both in EHS and on campus must follow this procedure for calibration requirements. This procedure covers the minimum requirements to calibrate equipment. If the authorization wishes to perform calibrations to cover additional radionuclides that may be used, it is their responsibility to perform those calibrations and save the results to their records.

3. **Definitions:**

3.1 Radiation monitoring instrument – any device used to measure the radiological conditions at a licensed facility. Examples include portable or stationary count-rate meters, portable or stationary dose-rate or exposure-rate meters, area monitors, single or multichannel analyzers, liquid scintillation counters (LSCs), gamma counters, proportional counters, solid-state detectors, and hand- and foot-contamination monitors [NUREG 1556 Vol 9 Rev 3].

3.2 Minimum Detectable Activity (MDA) – the smallest amount of activity a detector is capable of detecting. This term describes the capability of a detector, not the detection limits for a single sample. The MDA is a similar concept to a detection limit in other analytical equipment and will have activity-based units such as dpm.

3.3 Preventative Maintenance (PM) – a PM check is performed by the manufacturer of the equipment and generally applies to benchtop analytical equipment. During a PM visit, the manufacturer will evaluate the electronic components of a detector to ensure it can function properly. PM checks are recommended annually but not required unless periodic calibration by the user fails.

3.4 Quench – a process found in scintillation detectors that reduces the efficiency of the energy transfer or causes the absorption of photons. Quenching can lead to lower counts and false negatives. There are two types of quenching: chemical and color. Chemical quenching occurs within the solvent by reducing the number of electrons available to transfer energy. Color quenching occurs when the light produced by the fluorescent scintillation cocktail is absorbed or scattered. When running scintillation samples, it is important to minimize the color quench by having a transparent sample.

3.5 Chi-Square test – a statistical analysis recommended for benchtop analytical equipment to ensure the equipment is operating normally. The test evaluates whether a counter detects a radiation source with an appropriate amount of variability in the data. By using the bounds for a 95% confidence level, the Chi-Square test results should be within the selected confidence level to confirm the equipment is operating normally. If the Chi-Square test results fall outside the selected confidence level range, then a PM visit may be needed.

4. Procedure Details:

4.1 General calibration requirements

4.1.1 Survey instruments for RAM use are calibrated to be sufficiently sensitive to measure the type and energy of radiation used, including survey instruments used to locate low-energy or low-activity seeds if they become dislodged in the operating room or patient's room [NUREG 1556 Vol 9 Rev 3]. Prior to approval of a new Authorized User (AU) or a new radionuclide under an AU's permit, the authorization will be evaluated by the Radiation Safety Staff (RSS) to ensure that appropriate radiation monitoring equipment is on hand.

4.1.2 Requirements for survey instrument calibration generally apply to handheld survey meters and include [10 CFR 35.61]:

4.1.2.1 Calibrations shall be performed before first use, annually, and following a repair that affects the calibration.

4.1.2.2 Scales will be calibrated with readings up to 1000 mrem/hr with a radiation source.

4.1.2.3 Instruments will be calibrated at two separate readings on each scale.

4.1.2.4 The date of calibration must be conspicuously displayed. An example of a sticker used to display meter calibrations can be found in Section 5.

4.1.2.5 Instruments cannot be used if the difference between the indicated exposure rate and the calculated exposure rate is more than 20%.

4.1.3 Radiation workers can choose to have a qualified and licensed vendor calibrate their instrumentation (such as Ludlum Measurements, Inc.) or to perform in-house calibrations. If the latter option is selected, the Radiation Worker shall be trained on the calibration procedures and perform calibrations in accordance with this procedure. On-the-job training for instrument calibration may include observing trained personnel perform survey instrument calibrations and conducting survey meter calibrations under the supervision and in the physical presence of an individual authorized to perform calibrations [NUREG 1556 Vol 9 Rev 3].

4.1.4 The RSS does not calibrate handheld or benchtop equipment as of October 2022. It is recommended that all handheld survey instruments, regardless of medical or campus/veterinarian uses, be sent to a qualified and licensed vendor (such as Ludlum Measurements, Inc) for annual calibration and repair (when needed).

4.1.5 The RSS can provide initial on-the-job training, upon request, to an AU or delegated RW who wants to calibrate benchtop analytical equipment such as LSCs or gamma counters. After the RSS provides training, the newly qualified AU or RW can perform calibrations independently and can use this SOP to train their own groups without EHS assistance. This training does not have to be documented.

4.1.6 Calibrations of all equipment shall be performed in accordance with NRC regulations, annually, or at the frequency recommended by the manufacturer, whichever period is shorter. MU considers annual calibrations of handheld and benchtop equipment sufficient. Other instruments that need to be calibrated more frequently will be specified in authorization-specific protocols or other the RSS procedures (i.e. quarterly linearity for dose calibrators). Calibrations shall also be performed after any repairs that may affect the operating characteristics of the instrument. Battery changes are not considered repair or alteration.

4.1.7 The RSS does not require routine maintenance of instruments. The AU is responsible for following manufacturer's instructions for routine maintenance. If periodic calibrations are not sufficient, it is recommended that the AU contact a service provider for a PM check.

4.1.8 Records of calibration shall be retained for 3 years [10 CFR 35.2061]. The record must include the model and serial number of the instrument, the date of calibration, the results of the calibration, and the name of the individual who performed the calibration.

4.1.9 All calibration records for both handheld and benchtop equipment must be sent to the RSS for documentation to fulfill the requirements in 10 CFR 35.2061. Email records can be sent to rad@missouri.edu.

4.2 Calibrations for handheld survey meters to identify fixed and removable contamination should be performed as follows:

4.2.1 Fill out the Ludlum Calibration Form which can be found on the Ludlum website or EHS website.

4.2.2 The company name and contact person are not MU EHS; list the AU as the Company Name and list the person responsible for the meter as the Contact Person.

4.2.3 EHS does not need to receive the meters after calibration. They can be returned directly to the AU's lab.

4.2.4 Under the special instructions section, list any additional requirements needed. Ludlum typically calibrates handheld meters with a Cs-137 source. Instruments should be calibrated with a radionuclide that emits radiation of identical or similar type and energy as the environment in which the calibrated device will be used. If Cs-137 is not appropriate for the radiation field, list additional radionuclides in the special instructions section so Ludlum can perform additional calibrations with appropriate sources. Contact EHS for assistance in identifying which additional calibration sources to list.

4.2.5 When preparing meters for shipment to Ludlum, ensure that the check source is not attached to the side of the instrument. Some of these check sources are above the Department of Transportation (DOT) definition of exempt and require DOT training and paperwork to ship. If a source is still permanently attached to a meter, contact EHS for assistance with removing the source.

4.3 General requirements for calibrations of benchtop analytical equipment used to identify removable contamination include:

4.3.1 Manufacturer procedures and industry standard practices should be followed to calibrate benchtop analytical equipment such as LSCs and gamma counters.

4.3.2 The calibration should use radioactive sealed sources that are suitable for the geometry of the samples being analyzed. The sources should have known activities and be of similar type and energy as the radioactive materials to be analyzed. The analysis should be sensitive enough to detect the lowest levels of radioactivity desired. For example, if an LSC is being used for analyzing H-3 contamination on swipes in 20 mL scintillation vials, the calibration should use a H-3 20 mL scintillation vial standard.

4.3.3 Calibrations should include calculations of the efficiency and the MDA for radionuclides with a similar emission and energy type as those being used in the lab. Per the MU NRC Radioactive Materials License, removable contamination limits in unrestricted areas are 200 dpm for beta and gamma emissions and 20 dpm for alpha emissions. Therefore, the MDA for an instrument must be at or below these values to detect any contamination. See section 4.6.9 and 4.6.10 to calculate MDA..

4.3.4 A Chi-Square test is not required but can be a useful tool for determining if the instrument is operating properly. See section 4.7 for information on performing a Chi-Square test.

4.3.5 Quench curves on LSCs can also be performed by the user to account for any potential quenching in the sample. The manufacturer will not perform these during a PM visit, and it is the responsibility of the AU to perform this procedure if desired.

Most manufacturers sell quench standard sets for H-3 and C-14. The AU can prepare additional quench sets for other radionuclides that may be used in the lab such as P-32. Perkin Elmer has a guide on how to prepare quench curve standards for use in an LSC.

4.4 To calibrate LSCs:

4.4.1 Perkin Elmer recommends running the Self-Normalization and Calibration (SNC) protocol with the H-3, C-14, and background standards at a frequency befitting the use of the instrument. In general, running the SNC protocol with these standards once a month should be sufficient. However, running the SNC protocol once a year will satisfy the RSS calibration requirement for the instrument. A printout of the SNC shall be sent to the RSS as a record of the calibration. The SNC protocol will calculate the efficiency for each radionuclide as well as perform a Chi-Square test. The AU is responsible for purchasing and maintaining the H-3, C-14, and background standards set. These sets typically expire after 5 years, however the manufacturer does not require LSC standards to be active. Using expired standards is acceptable as long as results continue to indicate a properly functioning LSC.

4.4.1.1 For the Chi-Square test, each sample is counted 20 times.

4.4.1.2 The degrees of freedom are calculated as n-1, or in this case 19.

4.4.1.3 Using the Chi-Square distribution chart at a 95% confidence level, the SNC chi-square test should be between 10.117 - 30.14 for both H-3 and C-14. This value will change if the standards are not counted 20 times. However, the standard for the SNC protocol is to count 20 times and should not be changed.

4.4.1.4 If the Chi-Square test does not fall within the 95% confidence level (i.e. the 10.117 - 30.14 range), contact the manufacturer to schedule a PM visit. After the PM, run the SNC protocol again to ensure the instrument is performing properly. Any time maintenance is performed on the instrument, a new calibration must be performed, and the record must be sent to the RSS.

4.4.2 Older LSCs, such as Beckmans, may not have the SNC protocol available. In this case, it is recommended that appropriate LSC standards are counted to determine the efficiencies and MDAs for each radionuclide that may be analyzed using the instrument. Typical radionuclides include H-3, C-14, and P-32, all of which have premade NIST traceable standards available via the manufacturer. Since P-32 is a short-lived radionuclide, it is improbable that a NIST traceable standard exists. Manufacturers sell Si-32 as a standardfor P-32. See section 4.6 for instructions on calculating the efficiency and MDA.

4.4.3 When using H-3, C-14, and Si-32 (in lieu of P-32) for annual calibration, the efficiencies will cover the entire energy spectrum of an LSC from 0 - 2000 KeV. Using these three standards is sufficient for determining efficiencies for any radionuclide to be counted on an LSC. AUs should not need to purchase additional standards to determine efficiencies unless their research requires it.

4.4.4 Quench curves are not required for annual calibration. The Perkin Elmer manual recommends performing the quench curve for a specific radionuclide only once. The quench curve allows the instrument to calculate the efficiency for a specific sample if the radionuclide is known; it compares the count rate to the curve then determines activity in units of decays per minute. This method may be useful for research applications when an LSC is being used to quantify research samples. However, quench curves are generally not necessary when an LSC is used for counting contamination swipes if the annual calibration is performed to determine efficiencies for H-3, C-14, and P-32.

4.4.5 If a quench curve is not performed, the contamination swipe protocol should be set up with a calculated efficiency to convert cpm to dpm. This efficiency will be the one calculated using the H-3, C-14, and P-32 standards. When the radionuclide of concern is unknown, it is most conservative to set the efficiency to the lowest calculated efficiency from the annual calibration. This will typically be the efficiency for H-3, which is ~60%. P-32 will have the highest efficiency.

4.4.6 Be aware that calculated efficiencies may need to be updated on individual swipe survey protocols to ensure that cpm is being converted to dpm properly.

4.5 To calibrate gamma counters, including well counters and gamma counters with automatic samplers:

4.5.1 Always refer to the instrument's manual for specific procedures on calibration.

4.5.2 In general, a gamma counter can be calibrated similarly to an older LSC. Use a sealed radioactive source of similar type and energy to the radioactive material used in the laboratory. If needed, consult with EHS for assistance with sealed source selection. In order to cover a broad energy spectrum, typical radionuclides used in gamma counter calibrations are Co-57 (~120 KeV for Tc-99m) and Ba-133 (~356 KeV for I-131). Refer to section 4.8 for additional radionuclide information.

4.5.3 Some manufacturers recommend a counting window of +/-20% from the peak energy of the radionuclide to be analyzed. To be more conservative and account for unknown contamination, EHS recommends an open window count unless the MDA is over the removable contamination limits of 200 dpm for beta and gamma emitters and 20 dpm for alpha emitters.

4.5.4 When collecting a background count, Perkin Elmer recommends using an empty sample holder and counting for 10 minutes.

4.5.5 Newer gamma counters, such as the Wizard 2470, have an operation similar to the SNC on an LSC. It is called the IPA (Instrument Performance Assessment) test. The IPA test can be performed at a frequency deemed appropriate by the AU depending on the frequency of RAM use, or no longer than a period of one year. However, the IPA test works best with multiple data points, so monthly runs may be the best option. Refer to the equipment manual for instructions on performing the IPA test.

4.5.5.1 The IPA test determines the efficiency of the radionuclide of choice. Auto sampler gamma counters frequently come with an I-129 sealed source for this purpose. The I-129 source can be used for the IPA test and is an appropriate option for authorizations that use low-energy photon emitters such as I-125.

4.5.5.2 If the IPA test is used to determine efficiency, a copy of the results must be sent to the RSS annually.

4.5.5.3 The MDA must also be calculated. Use the background count rate and the equation found in section 4.6.9 or 4.6.10.

4.5.6 Alternatively, the AU may wish to use a separate generic protocol that counts an I-129 source with no efficiency and outputs the results in cpm. In this instance, count the source and background for one minute, then calculate the efficiency and MDA by using the equations found in section 4.6.

4.5.6.1 If this method is chosen, update the swipe survey protocol with the updated I-129 efficiency so the results output in DPM.

4.5.6.2 The AU may choose not to update swipe protocols in the system but instead determine the count rate which correlates to 200 dpm beta/gamma or 20 dpm alpha based on the new efficiency. To do this, use the equation found in section 4.6.8.

4.5.7 When the calibration is complete, send the record to the RSS.

4.6 To determine counting efficiencies and MDA:

4.6.1 Use a source, or multiple sources, of the same radionuclide or of similar energy to the radionuclides used under the authorization.

4.6.2 Sources used for calibration should be traceable to a primary radiation standard such as those maintained by NIST.

4.6.3 Count each source, including background, for one minute with an open window (i.e. the entire energy spectrum capability of the instrument, typically 0-2000 KeV for an LSC). Choosing an open window is a more conservative approach to determining efficiencies and MDAs. However, the AU may choose to narrow the window around a specific energy peak to increase the efficiency for a specific radionuclide (i.e. for I-131, the window could be set to 316-416 Kev which has the peak around 356 KeV).

4.6.4 When calculating efficiency, use the cpm output, not the dpm output. Some LSCs automatically correct cpm to dpm and have an option to input an efficiency correction factor. When determining efficiency, ensure that the efficiency correction factor is 1. This option is more common in older LSCs, such as Beckmans, and is not typically applicable in newer Perkin Elmer models, such as a Tricarb.

4.6.5 To calculate the efficiency for each radionuclide, use the following equation:

$$Efficiency = \frac{cpm\ from\ standard - cpm\ from\ background}{decay\ corrected\ activity\ of\ standard\ in\ dpm}$$

4.6.6 Make sure the activity of the standard is decay corrected for the date the standard is analyzed.

4.6.7 If the authorization chooses to narrow the energy window to increase efficiency for a specific radionuclide energy, then the efficiency equation should be modified as follows:

$$Efficiency = \frac{cpm\ from\ standard - cpm\ from\ background}{decay\ corrected\ activity\ of\ standard\ in\ dpm \cdot yield}$$

4.6.7.1 Here, the yield refers to the percentage of time that a specific energy is emitted from the radionuclide. For example, the emission of a 356 KeV photon from I-131 occurs 81.5% of the time. Thus, the resulting efficiency should be divided by .815 to increase the counting efficiency in the narrowed window.

4.6.7.2 EHS does not recommend narrowing the window unless the user needs this for research purposes or the MDA is too high when calculated using an open window.

4.6.8 If the AU does not want to update the survey protocols in the software to auto correct for DPM using the newly acquired efficiency, the following equation can be used to calculate the count rate that is equivalent to 200 dpm beta/gamma or 20 dpm alpha.

$$CPM = Efficiency \cdot 200\ dpm\ beta/gamma$$

$$CPM = Efficiency \cdot 20\ dpm\ alpha$$

4.6.9 The following equation can be used to calculate the MDA at a 95% confidence level when the counting times for the background and sample are different:

$$MDA_{95\%} = \frac{2.71 + 3.29\sqrt{R_b t_s\left(1 + \frac{t_s}{t_b}\right)}}{t_s E}$$

Where:

R_b = background count rate in cpm

t_s = sample count time in minutes

t_b = background count time in minutes

E = detector efficiency in counts per disintegration (DPS or Bq)

4.6.10 When the background and sample counting times are equal, which is the more common method, then the MDA calculation at a 95% confidence level can be modified as follows:

$$MDA_{95\%} = \frac{2.71 + 4.65\sqrt{R_b t_b}}{t_b E}$$

Where:

R_b = background count rate in cpm

t_b = background count time in minutes

E = detector efficiency in counts per disintegration (DPS or Bq)

4.7 To calculate the Chi-Square test:

4.7.1 The Chi-Square test is used to verify a detector is operating normally. It is recommended that a chi-square test be performed after calibration annually, but it is not required. If, during calibration, the results seem to be inconsistent, this test should be used prior to scheduling a PM visit with the vendor. The following equation can be used to calculate the chi-square value:

$$\chi^2 = \sum_{i=1}^{n} \frac{(N_i - \bar{N})^2}{\bar{N}}$$

Where:

n = Number of measurements

χ^2 = Chi-Square result

N_i = Individual measurement

$\overline{N}$ = Mean

4.7.2 Count the background 20 times.

4.7.3 Count the source 20 times.

4.7.4 Use the Chi-Square equation to calculate the results.

4.7.5 With a sample number of 20, the degrees of freedom is n-1, or 19.

4.7.6 Using a chi-square probability chart, the result for 19 degrees of freedom at a 95% confidence level should be between 10.12 - 30.14.

4.7.7 If the chi-square results do not fall within this range, run the test again.

4.7.8 If, after multiple runs, the chi-square test continues to fail, contact the vendor for a PM request.

4.8 The following is a list of common radionuclides that can be used for calibration of benchtop analytical equipment and their surrogates (where surrogates are longer-lived radionuclides that have similar energies to shorter-lived radionuclides which do not typically have calibration sources)

4.8.1 H-3 LSC standard can be purchased through a manufacturer.

4.8.2 C-14 LSC standard can be purchased through a manufacturer.

4.8.3 Si-32 LSC standard can be used for P-32 calibration in an LSC.

4.8.4 Co-57 has a similar energy to Tc-99m (~120 KeV) and can be used in gamma counters.

4.8.5 Ba-133 has a similar energy to I-131 (~350 KeV) and can be used in gamma counters.

4.8.6 Na-22 is a positron emitter (511 KeV) and can be used for other positron emitters in gamma counters.

4.8.7 Cs-137 is a mixed beta/gamma source that is often used for all types of calibrations (662 KeV) and especially for counting higher energy and PET radionuclides.

4.8.8 I-129 is recommended by the manufacturer to calibrate gamma counters for lower energy (~30-40 KeV photons).

4.8.9 For medical doses which are typically shorter lived, a unit dose for a patient can be used for calibration. For example, a Cu-64 dose for a patient can first be used in a gamma well counter to determine an efficiency and energy window prior to administration of the dose.

4.9 To use handheld instruments for monitoring dose rates, exposure rates, and contamination, an operational meter check must be performed.

4.9.1 Verify the meter has been calibrated within the past year. If the calibration is greater than one year, do not use the meter. Prepare the meter for shipment offsite for manufacturer calibration.

4.9.2 Verify the batteries are still working. To do this, switch the meter to the “BAT” setting. If the needle moves on the screen into the region that says “BAT TEST,” then the batteries are sufficient for proper operation. If the needle falls outside of the “BAT TEST” range, replace the batteries. The AU, not EHS, is responsible for battery replacement. Testing the battery may look different on different model types. Consult with the RSS if there are questions or concerns when checking the battery.

4.9.3 The RSS considers the requirements described in 10 CFR 35.61 to be fulfilled during the annual calibration process with the vendor. As such, the last step in performing an operational meter check is to verify the meter responds to a known source of radiation. The RW is not required to ensure that the meter reads +/- 20% of the measured reading to complete this step. Simply place the meter face against a check source to verify the detector responds.

4.9.4 If at any point the above steps cannot be completed, do not use the meter. Contact the RSS for assistance if necessary. If the meter fails any of the steps in the operational check, repairs via the manufacturer may be necessary.

5. Additional Information:

5.1 Example calibration sticker to post on equipment.

Radionuclide	Efficiency %	MDA (DPM)	BKG (CPM)

Protocol for Surveys: _________________

Efficiency for Unknowns: _____________%

Calibrated by:____________ Date:____________

6. References:

6.1 10 CFR 20.1501(c)

6.2 NUREG 1556 Vol. 9 Rev. 3

6.3 10 CFR 35.61

6.4 10 CFR 35.2061

6.5 NCRP 112

6.6 Perkin Elmer Tri-Carb LSC Manual series

6.7 Perkin Elmer Wizard Manual Series

7. Revisions

7.1 Rev 1 – 2023-5-10 – New SOP.