Protocol # 846.04-0032 SRM Prep 18JUN2010
Date: _____________
Supersedes Date: __________

Protocol for

Preparation & Standardization of Radioactivity Solution SRMs -- excluding NEI (4400 series) and natural matrix (4350 series) materials

Person Responsible for Operating Procedures _________________________________

Group Leader _________________________________

Health Physicist _________________________________

The Protocol has three components:

A. Preparation and storage of master solutions;

B. Preparation of SRM ampoules;

C. Preparation of counting sources

Materials

- All stock radioactive solutions are either (1) delivered from typical vendors (Oak Ridge, Isotope Products, etc.) in a screw-capped bottle; or (2) are obtained from flame-sealed ampoules that are held in the long-term Radioactivity Group inventory. The contents of the bottles or ampoules are typically either (1) carrier-free solutions of the radioactive isotope in either dilute nitric acid or hydrochloric acid, ranging from about 1.0 mol· L^{-1} to 4.0 mol· L^{-1}: or (2) contain carrier solutions consisting of dilute salt solutions of a stable isotopes of the same element at cation concentrations of less than 200 μg per gram of the dilute acid solution. The volumes are typically 5 mL or less. The exact specifications of the activity content, as well as chemical and physical forms are specified on the NIST 364 form.

- All subsequent diluting solutions consist of either HNO_3 or HCl (≤ 3.0 mol· L^{-1}), which may or may not contain stable carrier cations at a concentration of < 50 μg per gram of solution.

- Other materials used for the procedures include:

(1) Glass bottles (50 mL to 2000 mL) to contain master solutions;
(2) other glassware to manipulate non-radioactive solutions (volumetric flasks, beakers, funnels, syringes, pipettes, etc.);
(3) 5 mL glass ampoules;
(4) polyethylene pycnometers for picking up and dispensing solutions gravimetrically;
(5) disposable switching valves, gas-tight syringes, and pick-up and dispensing tubing for Hamilton dispenser;

(6) plastic bags used for opening all ampoules under controlled conditions;
(7) absorbent paper and wipes;
(8) disposable gloves and protective clothing;
(9) waste bags and "sharps" containers;
(10) smears to do surveys for possible contamination at the end of operations.

Facilities

- Radiochemistry laboratories B46, B152, B156, and B157; Storage of sealed solutions in B49.
- Fume hoods, rated for use with radioactivity (covered with trays and/or absorbent materials, adequate to absorb or contain the total volume of the radioactive solution) in B46, B152, and B157 to handle stock material, to open ampoules and dispense solutions into ampoules.
- Balances in B46 and B152.
- Hamilton dispenser set up in one of the hoods in B157 (with strippable coverings).
- Ampoule sealer in hood in either B46 or B156.

Procedures:

(A) Preparation and storage of master solutions

- The stock material bottle or ampoule is opened in a plastic bag in a fume hood. The hood surface is covered with absorbent material.
- All procedures are performed with safety glasses, protective clothing (lab coats) and disposable gloves.
- Monitoring is performed with badging, finger rings, and portable monitors.
- No shielding is required.
- The stock radioactive solution from a vendor's bottle may be transferred by pycnometer to a larger bottle which may contain a pre-dispensed quantity of diluent; or diluent may be added directly to the vendor's bottle by pycnometer. In the former case, the vendor's bottle is rinsed by repetitive pycnometer transfers. The solution in the re-capped stock bottle is allowed to stand for several hours.
- The solution in the stock bottle is transferred by pycnometer to either a small number of preweighed empty master ampoules or to a master solution bottle that contains the diluting carrier solution. Care is taken (through training and experience) to minimize aerosol dispersion during all pycnometer transfers.
- Master ampoules are flame sealed for storage, and future use. Ampoule sealing is covered in Protocol # 846.04-0034 amp.sealing 21JUN2010.
- Master bottle is capped and mixed, for future use.
- All waste materials (including stock bottle and pycnometer) are packaged for pickup by HP. All waste materials are dry prior to packaging.
- All materials, facilities and personnel are surveyed for possible contamination on conclusion of the procedure.

(B) Preparation of SRM Ampoules

- This procedure is largely identical to that previously described in procedure Protocol # 846.04-0038 4400low 23JUN2010.
- All procedures are performed with safety glasses, protective clothing (lab coats) and disposable gloves.
- Monitoring is performed with badging, finger rings, and portable monitors.
- No shielding is required.
- A series of ampoules (typically 20 to 400) are filled with a master solution, obtained from either from a master solution bottle, previously prepared (see above), or from a master solution ampoule (described next)
- The recommended procedure for opening flame-sealed glass ampoules is described on the NIST website at http://physics.nist.gov/Divisions/Div846/srm.html

- If starting with a master solution ampoule (5 mL) to prepare a master solution:
    1. The ampoule is opened in a plastic bag located in a hood. The "sharps" are disposed of in designated containers.
    2. The entire solution contents of the opened ampoule are picked up into a pycnometer.
    3. The pycnometer is weighed and handle throughout with long forceps
    4. Some portion of the pycnometer contents is dispensed in a prepared master solution bottle that already contains a known mass of the diluting solution. Care is taken (through training and experience) to minimize aerosol dispersion during pycnometer transfers.
    5. The pycnometer is reweighed.
    6. The remaining portion of the solution in the pycnometer is transferred to a clean and empty 5 ml ampoule.
    7. The empty pycnometer is disposed of as radioactive waste.
    8. The residual ampoule is flame sealed. Ampoule sealing is covered in Protocol # 846.04-0034 amp.sealing 21JUN2010
    9. The master solution bottle is capped and mixed.

- The master solution bottle is uncapped in the hood where the Hamilton dispenser is located and the pickup tube is inserted into the bottle.
- The solution is recycled through the dispenser and bottle several times for mixing.
- Empty ampoules are handled with long tweezers and are filled by inserting the dispenser's dispensing tube into the ampoule neck.
- Each filled ampoule is capped and is placed in a tray until all ampoules are filled.
- All ampoules are flame sealed.
- All ampoules are sterilized (at a subsequent time) prior to packaging and storage.
- All waste materials (including master solution bottle and disposable dispensing parts) are packaged for pickup by HP. All materials are dry prior to packaging.

- All materials, facilities and personnel are surveyed for possible contamination on conclusion of the procedure.

(C) Preparation of counting sources

- All procedures are performed with safety glasses, protective clothing (lab coats) and disposable gloves.
- Monitoring is performed with badging, finger rings, and portable monitors.
- No shielding is required.
- Two types: (1) deposited point sources on thick backings; (2) LS cocktails. If any other types are needed and are to be prepared they will be specified and described on the NIST 364 or in separate, specific protocols.
- Gravimetric aliquants from sealed ampoules after opening are taken by pycnometer, using methods identical to that described in the procedures above. Care is taken (through training and experience) to minimize aerosol dispersion during pycnometer transfers.
- Deposited sources (point sources) are made from either dilute nitrate solutions or chloride solutions and are not volatile for the nuclides covered by this procedure. Solution is deposited onto non-conductive polyester tape on an aluminum ring. After deposition, the source is covered with a mesh cloth for drying in the fume hood. When dry, the deposited source is covered with a protective film non-conductive polyester tape.
- Solution aliquants for LS sources are added directly by pycnometer to pre-prepared LS cocktails contained in LS vials and are tightly capped.
- All waste materials are packaged for pickup by HP. All materials are dry prior to packaging.
- All materials, facilities and personnel are surveyed for possible contamination on conclusion of the procedure.
- Use of the prepared counting sources in various instruments is covered in other protocols.

General Hazards and General Hazards Mitigation

- The main general hazard involves the use of an opened radioactive solution. An '"opened radioactive solution" refers to a solution of any radionuclide that (1) is intentionally being opened for subsequent use (e.g., to make a counting source, to dilute it, or to prepare a master solution, etc.) or (2) had been contained in a suitably sealed glass container (bottle or flame-sealed ampoule) that has broken or spilled.

- Opened radioactive solutions should only be used or cleaned up in the presence of two persons, one of whom is designated as the person actually handling the radioactive material. The presence of the other individual ensures safety.

- Handling of radioactive sources is to be done only by persons having been adequately trained and who have demonstrated their competence to the SRM coordinator.

- All persons handling opened radioactive solutions must wear protective clothing, which includes but is not limited to a lab coat, gloves, and safety eyeglasses or goggles. Closed shoes and garments fully covering the legs must also be worn (i.e., no sandals or flip-flops, and no shorts or skirts). Monitoring is performed with badging, finger rings, and portable monitors.

- Operations with opened radioactive solutions that may result in the unintentional or accidental release of radioactivity include:
    1. Discovery of broken ampoules or bottles of solutions held in storage.
    2. Breaking or cracking of a flame-sealed ampoule during use, which may include measurement of the ampoule in an ion chamber or with small solid angle photon detectors.
    3. Opening of a flame-sealed ampoule and filling of a plastic pycnometer.
    4. Dropping a filled pycnometer during weighing operations or during source preparation.
    5. Spills from an opened bottle (accidental tipping over, or dropping) during preparation of master solution dilutions.
    6. Spills during dispensing or from filled, but unsealed ampoules.
    7. Releases during flame sealing of ampoules.

- In each of the above cases, the radioactive solution must be handled throughout all operations inside a constraining plastic bag and/or above a sufficient quantity of absorbent paper to completely contain the total volume of the radioactive liquid. Secondly, any transfer of the contained solution must be done in a secure way (with a tray, carrying case, or even guarding a filled pycnometer with a gloved hand) so that the container can not tip or fall any great distance.

- Most personnel exposure occurs with solutions when the radiations are shielded by glass containers or by the plastic pycnometer (in worst case).

- Typical handling times:

    Less than one minute to open ampoule and fill pycnometer.
    Less than two minutes of handling pycnometer to make master solution or to prepare one counting source.
    Less than 30 minutes in front of dispenser to fill 50 ampoules.
    Less than 15 minutes to seal 50 ampoules.

- Inventory control:

    All sealed sources are labeled as to nuclide, activity, reference date & researcher responsible.
    Long-term storage is in B49.
    Sources used for ongoing work are stored in B157.

- The materials used for this protocol also include chemical hazards, principally the corrosive nature of the dilute acids. The carrier salts are not volatile and are not toxic at the levels specified in the NIST 364 and as specified below for the specific radionuclide.

- The radioactive solutions covered by this protocol are stable can be handled <u>as solutions</u> with reasonable safety in fume hoods.

- The procedures covered here incorporate handling of sharps (opened ampoules with rough glass edges), possibly contaminated with radioactive material. The sharps are always handled with gloved hands and with additional wadded absorbent paper for protection.

- Natural gas/oxygen torches are used for flame sealing of ampoules. The torch heads are secured in fixed positions in the Collé-Cavallo ampoule sealer and the flames are never near combustible materials. The compressed-oxygen tanks use approved regulators and are secured to bench tops with approved gas cylinder holders.

Emergency Procedures

- All laboratory procedures involving the use of radioactive materials are done in the presence of two qualified persons.

- All laboratories are equipped with suitable portable instruments for surveying exposures and detecting possible contaminations.

- In the event of a non-radiological accident involving facilities or personal injury, the severity of the accident must be assessed and addressed accordingly. For emergency situations, all personnel are trained to call (2222) the appropriate authorities. Depending on the situation, either the location should be immediately controlled and evacuated or first aid action should be administered to anyone injured (2222), e.g., turning off dangerous equipment (torches, electrical sparking), use of fire extinguishers, use of emergency showers and eye wash stations, or stopping of bleeding, or CPR.

- When unplanned exposures are discovered, laboratory workers must immediately remove themselves from the radiation source and must immediately contact the Health Physics office.

- In case of source breakage or leakage, the spill must be isolated and Health Physics (HP) immediately notified (call extension x5800 or x2222 after hours). The laboratory should be posted as having potential contamination and secured until cleared by HP. If any individuals are contaminated measure the contamination, note the level and begin immediate washing in a stopped sink or basin (collecting the wash water for later analysis). Cleanup of the spill is to follow normal HP practices. If the radiation level is not a hazard to the individual involved in the incident, the individual shall remain in the facility until he/she is cleared by HP.

Radionuclides

This protocol is applicable for use with the following radionuclides and their attendant chemical form.

Radionuclide (includes progeny in decay chains)	SRM Number	Maximum Activity	Chemical Form	Physical Form
Nickel-63	4226C	20 MBq	chloride/$NiCl_2$ in dilute HCl	liquid
Cesium-137	4233E	0.1 GBq	chloride/CsCl in dilute HC	liquid
Strontium-90 / Yttrium-90	4239	20 MBq	chloride/YCl_3/$SrCl_2$ in dilute HCl	liquid
Barium-133	4251C	0.1 GBq	chloride/$BaCl_2$ in dilute HC	liquid
Holmium-166m	4274	20 MBq	chloride/$HoCl_3$ in dilute HCl	liquid
Curium-244	4320A	2 kBq	nitrate in dilute HNO_3	liquid
Natural Uranium (234, 235,238)	4321C	0.6 GBq	chloride/$UO_2(NO_3)_2$	liquid
Americium-241	4322C	50 kBq	nitrate in dilute HNO_3	liquid
Plutonium-238	4323B	30 kBq	nitrate in dilute HNO_3	liquid
Uranium-232	4324B	0.2 MBq	chloride/$UO_2(NO_3)_2$ in dilute HCl	liquid
Thorium-229	4328C	50 kBq	nitrate in dilute HNO_3	liquid
Curium-243	4329	40 kBq	nitrate in dilute HNO_3	liquid
Plutonium-239	4330B	20 kBq	nitrate in dilute HNO_3	liquid
Americium-243	4332D	1 MBq	nitrate in dilute HNO_3	liquid
Plutonium-242	4334H	30 kBq	nitrate in dilute HNO_3	liquid
Lead-210	4337	4 MBq	chloride/$BiCl_3$/$PbCl_2$ in dilute HCl	liquid
Plutonium-240	4338A	40 kBq	nitrate in dilute HNO_3	liquid
Radium-228	4339B	2 MBq	chloride/$BaCl_2$ in dilute HCl	liquid
Plutonium-241	4340B	1 MBq	nitrate in dilute HNO_3	liquid
Neptunium-237	4341	50 kBq	nitrate in dilute HNO_3	liquid
Thorium-230	4342A	30 kBq	nitrate in dilute HNO_3	liquid
Europium-152	4370C	15 MBq	chloride in dilute HCl	liquid
Cobalt-60	4915F	20 MBq	chloride/ $CoCl_2$ in dilute HCl	liquid
Strontium-90	4919I	2 MBq	chloride/YCl_3/$SrCl_2$ in dilute HCl	liquid
Iron-55	4929f	3 MBq	chloride in dilute HCl	liquid
Radium-226	4967A	2 MBq	chloride/$BaCl_2$ in dilute HCl	liquid