Protocol # 846.04 – 00xx
Cryptophane Expt
25 June 2010

Protocol for

Proposed Experiments on Thermodynamics of Radon (^{222}Rn) Binding to Cryptophone in Water

(in collaboration with Dept. Chemistry, Univ. of Pennsylvania)

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Group Leader	----------
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MATERIALS

- radon-in-water generator consisting of a 10-mL dose vial (with Al-lined rubber septum) that contains three polytethylene encapsulated ^{226}Ra solutions and completely filled with pure water
    - The three polyethylene capsules are completely sealed and contain a total ^{226}Ra activity of 21.7 kBq (as of 25 June 2010). They were prepared in 2001 using material from obsolete SRM 4961 (prepared in 1947).

- small (2 mL) reaction vessels (vials with Al-lined rubber septums) containing milligram quantities of cryptophane (provided by UPenn in the seled vessels)

- gas-tight syringes with needles for water and solution transfers

- pure water, de-ionized and distilled

- 20-mL LS vials, filled with commercial scintillation fluid (low flash point, environmentally safe), either Ready Safe (Beckman Coulter), UltimaGold (PerkinElmer), or Optiphase HiSafe 3 (PerkinElmer), or Instagel (PerkinElmer).

- 500 mL polypropylene waste bottle, partially filled with activated charcoal, for water rinses of syringes

- wheeled cart and trays for transporting containers (reaction vessels and LS viaaaaals between rooms)

- personal safety items (absorbent paper covering all work surfaces, gloves, lab coats, safety glasses)

- dosimetry (film badges , finger rings, survey meters, pocket dosimeters for UPenn visitors)

FACILITIES (in Bldg 245)

- Room B157 – primarily
    - counter tops for handling containers
        - (LS vials and reaction vessels prior to use)
    - fume hood – generator location and used for all syringe transfers

- Room B152 – balances for weighing aliquants transferred into reaction vessels
    - and solution samples transferred into LS vials

- Room C109 – LS counters

PROCEDURE

A schematic outlining the overall experimental procedure is attached as Figure 1. Steps include:

1. The radon-in-water generator is loaded inside a fume hood. That is, the vial is filled with water with the Ra capsules inside, and the vial is sealed with the septum. The hood surface is covered with absorbent material. The generator is kept within the hood throughout all of the steps that follow.
2. All procedures are performed with safety glasses, protective clothing (lab coats) and disposable gloves.
3. Monitoring is performed with badging, finger rings, and portable monitors.
4. No shielding is required.
5. ^{222}Rn accumulates in the generator (from decay of the ^{226}Ra in the capsules) for a period of several weeks to reach at least 90 % of radioactive equilibrium. No ^{226}Ra is present in the generator's water volume because the polyethylene capsule only allows diffusion of

^{222}Rn. Assuming an emanation fraction of 0.6 and 90 % equilibrium, the maximum ^{222}Rn activity available in the generator is 12 kBq. With a total volume of 11.7 mL, the maximum concentration that will be transferred by syringe to the reaction vessels is approximately 1 kBq/mL

6. A 10-mL gas-tight syringe is used to transfer aliquant of the accumulated radon solution into a set of 20 or less reaction vessels that contain the cryptophane (preloaded at UPenn and brought to NIST) . The transfers are effected by inserting the syringe needle through the generator's septum to fill the syringe, and a portion of the drawn solution is placed in a reaction vessel by inserting the needle through the vessels septum and dispensing. One vessel after another in the set receives the aliquant from the same syringe filling.
7. The radon-in water solution is never open to air so there is no release (or insignificantly trivial amounts) of radon to the air.
8. The transfers are performed inside the hood. Any accidental releases such as from mishandling of the syringe would release the radon into the hood's exhaust.
9. The expected binding reactions are near instantaneous, and the reaction vessels are immediately sampled.
10. Sampling is performed with a 2-mL gas tight syringe. The syringe needle is inserted through the reaction vessel's septum and the solution drawn up.
11. The solution in the syringe is dispensing below the surface of the scintillation cocktail in the LS vial. The needle is inserted into the bottom of the vial. Insufficient time elapses for any radon escape to occur before the LS vial is capped.. Two or three LS vials (for measurement replication) are filled from each syringe sampling.
12. The sampling syringe is rinsed twice with pure water between each reaction vessel sampled.
13. The rinse water is injected ito the large volume waste bottle that contains activated charcoal. Most of the small residual amounts of radon in the rinse water is expected to be absorbed onto the charcoal. Any further small releases will be taken up in the hood exhaust. All ^{222}Rn daughter products (in equilibrium) will be contained in the bottle.
14. The was bottle is capped at the conclusion of all work and is stored to decay down to background levels (< 30 days).
15. The filled LS vials are transferred for counting in the LS counters located in Room C109. The procedures used for counting are covered in other protocols.
16. On conclusion of this work, the generator is dismantled. The capsules are removed with long-handled tweezers and returned to their capped storage vials for long-term storage.
17. All materials and protective coverings in the hood will be surveyed for contamination by short-lived radon daughters before disposal.

GENERAL HAZARDS AND GENERAL HAZARDS MITIGATION

- The main general hazard involves the use of the radioactive ^{226}Ra source, that is radon-in-water generator, and the resulting ^{222}Rn solutions. However, of all the procedures performed by the Radioactivity Group with an "'opened radioactive solution" (see Protocol # 846.04-0032 for definition), this protocol is the least hazardous. The ^{226}Ra source is always completely contained within the polyethylene encapsulation, and the ^{222}Rn solutions are always continued in either the generator, gas-tight syringes, reaction vessels , or LS vials, all of which are sealed. The solutions are never open to the air for potential inhalation of radon or its daughter products.

- The procedures in this protocol are only performed in the presence of two persons, one of whom is designated as the person actually handling the radioactive materials. The presence of the other individual ensures safety.

- Handling of radioactive sources (generator, syringes, reaction vessels, LS vials) is to be done only by persons having been adequately trained and who have demonstrated their competence to the SRM coordinator.

- All persons handling these radioactive sources 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 the radon solutions that may result in the unintentional or accidental release of radioactivity include:
    1. spills resulting from mishandling of a syringe, highly improbable and only used inside the hood

    2. dropping, breaking, or cracking of a one of the containers (generator, reaction vessels, LS vials), also highly improbable and are only handled inside the hood, except for the counting of the subsequently loaded LS vials

- In each of the above cases, the radioactive solutions are handled throughout all operations inside the hood and all surfaces are covered with a sufficient quantity of absorbent paper to completely contain the total volume of the radioactive liquid. Secondly, all transfers of contained solutions are done in a secure way.

- Personnel exposure occurs with solutions when the radiations are shielded by glass containers.

- Typical handling times:

  Less than one minute to extract the radon solution from the generator by syringe.

  Less than five minutes for filling the set of reaction vessels.

  Less than 10 minutes to sample the reaction vessels and load the LS vials.

  Less than five minutes to insert the LS vials in the counter.

- Inventory control:

  All sealed sources are labeled as to nuclide, activity, reference date & researcher responsible.

  Sources used for this ongoing work are stored in B157.

  All sources, excepting the ^{226}Ra capsules, will deacy to background levels in less than 30 days.

- The materials used for this protocol may include chemical hazards, although the risks are minimal involving only water, cryptophane (a class of organic supramolecular compounds synthesized for research purposes primarily for molecular encapsulation of gases), and commercial scintillation fluids.

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

- The procedures covered here incorporate handling of sharps (syringe needles), possibly contaminated with radioactive material. The sharps are always handled with care and with gloved hands and with additional wadded absorbent paper for protection when possible.

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.

ATTACHMENT: schematic overview of the protocol