Collé role in the ¹³³Ba incident of 2 May 2018

Report as of 4pm, 9 May 2018 Covering the period 26 April through 9 May 2018

Thursday 26 April 2018 morning

Laureano-Perez and I prepared 46 ampoules of ¹³³Ba for an SRM production run. All of the work was done in B157, excepting mass measurements of the master solution bottle on the Jupiter balance in B152. The production steps for filling the ampoules consisted of:

- 1. Nine empty ampoules were pre-weighed on the balance in B152.
- 2. Preweighing the master solution bottle in B152.
- 3. Adding the Ba⁺² / HCl carrier solution to the bottle in B157.
- 4. Weighing the bottle again in B152 to determine added carrier mass.
- 5. Opening the vial of ¹³³Ba stock solution (RS# 10-0113) using our standard protocol for SRM production (SE 38). This was done inside a plastic bag contained in a fume hood.
- 6. Aspirating pycnometer was used to gravimetrically transfer the solution from the vial to the master solution bottle. The pycnometer mass measurements were made on the balance in room B152
- 7. The capped master solution bottle was carried into B152 for final mass measurement on the Jupiter., and returned to B152 for dispensing.
- 8. Using the dispenser, the master solution was recycled for a few cycles back into the master solution bottle. The dispenser was located in the rear hood of B157
- 9. The master solution was dispensed into 46 ampoules, each containing 5 mL of solution, using the dispenser.
- 10. The 9 pre-weighed ampoules, now containing the ¹³³Ba solution were reweighed on the balance in B152. These ampoules were # 5, 10, 15, 20, 25, 30, 35, 40, 45 in filling order.
- 11. The ampoules were sealed using our manual sealer in the front hood of B157.

N.B. The ampoules were our old "NIST 1" standard ampoules (c. 1976). They were sealed with tip seals on cut-down 75 mm height ampoules, not by removal of excess glass with forceps. We had hoped to use our new Biosciences automatic sealer that has a fixed torch flame, but were unable to do so because the revised ampoule sealing SOP (846.04) was not yet approved. Although the majority of the tip seals were better than what we would have gotten with sealing with excess glass removal, the seals were somewhat inconsistent because of the absence of a fixed torch flame. It is not clear if the cutting down of the ampoules played any role in having a weak seal. At present, it is my belief that the problem may be a result of slight concave seals from insufficient molten glass from variable positioning of the flame.

12. The seals on all 46 ampoules were visually inspected for defects. Seven ampoules (# 17, 21, 28, 29, 37, 38, 42) were suspect. Each was tested for leaks by inversion. Only one leaked, though the appearance of the other six (ballooning seal, asymmetric seal collapsed glass) seemed to justify transfers. The solution in the 7 ampoules were transferred to new ampoules and resealed.

N.B. The transfer and resealing of 7 ampoules was an exceedingly pessimistic assessment since some of the seals were clearly better than others we have used in an SRM production.

- 13. All 46 of the ampoules were tested for leaks by inversion. None leaked. All 46 ampoules were swipe tested, along with swipes taken of the work areas and tools in B157 and the balance in B152. None of the smears indicated any contamination.
- 14. Both Lizbeth and I used personal contamination monitors (PCMs) on exiting the work areas with no indication of any personal contamination. I used the hand and foot monitor (per the recent directive), not the half-body monitor.

N.B. The 9 sealed ampoules that had determined masses were intended to be measured in both in chamber "A" (to get an approximate activity for the SRM solution based on an old calibration) and in auto chamber "B" (to establish a calibration factor for that chamber. The experimental plan was to also measure the other 37 ampoules in auto chamber B to examine solution homogeneity.

Monday 30 April 2018 morning

In preparation for transporting the ampoules to B47 for ion chamber measurements, I went to B157 (where ampoules were stored in racks) and put the 9 ampoules with determined masses into plastic tubes with caps. This operation was performed on a cart, and consisted of picking up each ampoule, inverting the ampoule (to remove condensation in the ampoule stem), inserting the ampoule into a tube, capping the tube, and placing the tube into a tube rack. Each ampoule was identified by number with a label on top of the tube. The rack was moved from the cart to the counter top.

On leaving the lab, I monitored my gloved hands with portable meter and surveyed myself with the PCM (hand and foot monitor) in hallway. No personal contamination was detected.

Monday 30 April 2018 afternoon.

Ryan and Lizbeth (I was not present) retrieved the 9 ampoules in plastic tubes from B157 and took them to B47 for auto chamber B measurements. It is my recollection that near 3 pm that afternoon, Lizbeth and I unloaded the ampoules from chamber B holders and returned the

ampoules to the plastic tubes. We then carried the ampoules in the tube rack inside a cardboard box back to B157 and set the rack on the counter top. Before exiting the basement and on leaving B47, I surveyed myself in the PCM (hand and foot monitor) and the half-body PCM in hallway. No personal contamination was detected. On exiting B157, I again surveyed myself on the hand and foot PCM. No personal contamination was detected.

We returned shortly thereafter to B47 to see what was going on with Ryan. I again exited and surveyed myself on both PCMs outside B47. I went up to B157 (to fetch I think papers or for some reason) and left immediately. On surveying myself for the fourth time in a matter of minutes, I set off the half-body alarm. I checked myself on the hand and foot PCM and was "clean". I went back to the half-body PCM and it alarmed again. I then checked myself thoroughly with the PCM's hand-held monitor and could find nothing. On a third measure, I was "clean". I did not notify GSRD that the alarm went off, but left a note (and perhaps people will want to make a big deal about it) — however given that (i) we have been instructed to remeasure ourselves when an alarm goes off, which I did; (ii) all previous PCM monitoring on both floors did not indicate personal contamination; and (iii) I could not detect anything with the held-held PCM. This was my conscious and personal decision.

Tuesday 1 May 2018 morning

I went into B157 to place the remaining 37 ampoules into plastic tubes for transport to B47 for ion chamber measurements. An identical procedure to that done previously was performed; viz., picking up each ampoule from ampoule rack, inverting it, placing it in a plastic tube, capping the tube, and placing the tube into a tube rack. The rack was placed on the counter top next to the rack that was filled on Monday.

On leaving the lab, I monitored my gloved hands with portable meter and surveyed myself with the PCM (hand and foot monitor) in hallway. No personal contamination was detected.

Wednesday 2 May 2018 morning

Fitzgerald, Lizbeth, and I went into B157 to retrieve the two racks of ampoules (in plastic tubes) to carry them to B47. We all surveyed ourselves on the PCMs on exiting. No personal contamination was detected.

With Ryan hanging around and giving Lizbeth instructions on how to run chamber A software, Lizbeth began to make the chamber A measurements on the first batch of 9 ampoules (those having determined masses). I began to load the auto chamber B holders with the other batch of 37 ampoules.

We all stopped a little before 11 am to go to a Division seminar at the reactor building. Ryan and I exited first. I surveyed myself with the half body PCM (on someone's recommendation that it would be more sensitive). No personal contamination was detected.

After the seminar, Lizbeth and I returned to B47. I continued loading the ampoules into auto chamber B holders; Lizbeth continued making chamber A measurements. When I finished loading the ampoules, I left to return to my office. Lizbeth continued with the measurements. No personal contamination was detected with half-body PCM.

Wednesday 2 May 2018 afternoon

At approximately 1 pm, Lizbeth called me at my office, asking me to come down. On arriving at B47, Lizbeth said she detected a small drop of liquid on her glove while handling ampoule # 45 when inserting it into the chamber holder. She had placed the ampoule back into the plastic tube. I took the tube into B46, and opened it inside a plastic bag in the fume hood. I inverted the ampoule (#45) over wadded paper and with a survey meter confirmed that the ampoule seal was leaking. This ampoule had been measured previously in chamber A that morning.

Lizbeth notified GSRD, and we were soon joined by Fitzgerald and John Zometsky (GSRD). Lizbeth surveyed herself (particularly gloves and hands) with portable meter and in the PCM. No personal contamination was detected on her. Zometsky using a portable NaI detector found contamination on Lizbeth's hand, which they then proceeded to decontaminate. I will leave the retelling of the saga of Lizbeth's clean-up for others more personally involved. Ryan and I with Zometsky began to survey other parts of the room and secure contaminated articles in bags, and decontaminate the tweezers, etc.

Michael Spady and Manny Mejias (GSRD people) arrived shortly thereafter. I am not certain if Christopher Young was there as well. My recollection may be a bit fuzzy at this time because of all of the activity of individuals. At this point in time, I largely stood around as GSRD took over. GSRD assumed control of the situation in terms of making the survey measurements by smears and by confiscating contaminated articles

Ryan and I continued to assist GSRD in isolating and surveying with meter and smears contaminated and potentially contaminated articles. Ryan and I also tried to decontaminate a few articles (mainly tweezers) under GSRD direction. GSRD detected that the ampoule rack for the 37 ampoules still in chamber B holders was contaminated. They secured it. The entire room and counter tops was surveyed by GSRD. The GSRD personnel eventually declared that B47 was sufficiently contained, and I requested to take the leaking ampoule #45 (in plastic tube in plastic bag in box) and the other 8 ampoules (in tubes) to B157 to get the activity out of B47. like the plastic tubes and rack. GSRD released the ampoules and I took then to B157. On exiting B47, I surveyed my gloved hands with a portable meter and used both the half body PCM and hand and foot PCM. No contamination was found. I also re-surveyed myself on both 2nd floor PCMs on leaving B157, on returning to B47.

In discussions with Fitzgerald and consultation with GRSD, we decided that the ampoules in auto chamber B could be measured overnight – given that the ampoules were secure within the holders, and that this data might give us additional information on the situation. I was told the next day that they were measured.

I departed B47 at about 4:30pm. On exiting, I surveyed myself on both the half-body monitor and the hand and foot monitor.

Thursday 3 May 2018 morning

Fitzgerald and I, accompanied by Chris Young and John Zometsky, began to evaluate the contamination in B47 and decontaminate the room. Largely, Zometsky took smears and Young counted them while Ryan and I processed the room and materials. Many, many smears were taken. Of which GSRD has the data. I personally tried to decontaminate the tweezers (used to insert the ampoule into chamber holders) repeatedly, and although the levels decreased, they never were fully clean. evaluate B47. Lizbeth joined us after a while. Ryan, Lizbeth and I We completely stripped the protective coverings off the counter top where the ampoule changes took place and placed it in rad waste bags. GSRD did the smear surveys as we proceeded.

Several times throughout the clean-up process, I surveyed myself with both PCMs in the hallway. No personal contamination was ever detected.

The biggest task was unloading the ampoules from the auto chamber B holders. The task was divided as follows to minimize the spread of further contamination:

- (i) I removed the holders and opened them, exposing the ampoule. I never touched the ampoules at this point.
- (ii) Lizbeth removed the ampoule from each holder with tweezers and placed the ampoule in a plastic tub. Lizbeth never touched the holders.
- (iii) John Zometsky individually smeared the inside cup of each holder and the outside of the holders.
- (iv) Chris Young counted the smears.
- (v) I put the holders back together and returned them to the chamber's sample changer. At the end of this process, we detected contamination on the end of the tweezers used by Lizbeth, well above what it had been when we started. I tried to decontaminate it repeatedly until it appeared to be at background levels. GSRD subsequently told me that they were still contaminated at very low levels.

The capped plastic tubs of ampoules removed from the holders was taken to B157.

Personal monitoring on exiting B47 and again on exiting B157 was performed with both the half-body and hand & foot PCMs. No contamination was detected.

N.B. I was subsequently informed that one of the holders (I believe it was that holding ampoule #16) was seriously contaminated inside, suggesting that at least one other ampoule seal had

failed. It had by then also become apparent that the outside of all holders was at least slightly contaminated, based on smear counting. Ryan informed me that he made "background" measurements on all of the emptied holders, and detected nothing above background level.

Thursday 3 May 2018 late afternoon

I had returned to B47 around 2:30 pm to see if I could be of any further assistance and to learn of new developments and findings. I largely just stood around and really didn't touch or handle anything that was potentially contaminated.

On exiting the area with Lizbeth, I set off the alarm on the half-body PCM, indicating contamination on the front chest area. Lizbeth just in front of me was clear. We were immediately joined by Chris and John (GSRD). I re-surveyed myself three times in the PCM, and it alarmed each time. I surveyed myself in the hand& foot monitor, and no personal contamination was detected with that. Chris used the hand-held wand for the half-body PCM and located the contamination area as on the front of my shirt near a mid-level button — not on the lab coat. Chris tried to remove the contamination several times with a lint roller, unsuccessfully. I removed my shirt and Chris surveyed my skin with the hand monitor, finding no contamination. I also surveyed myself, bare chested in the half-body PCM. The alarm did not go off. Although nothing was detected on my lab coat, GSRD retained it. They gave me a lab coat and I departed for the day.

N.B. THIS EVENT WAS THE FIRST INDICATION OF ANY <u>DEFINITIVE</u> PERSONAL CONTAMINATION OF ME, DESPITE THE MANY, MANY PCM SURVEYS TAKEN OVER THE PREVIOUS 4 DAYS!

I was subsequently told that my shirt and lab coat was contaminated with ¹³³Ba at low Bq levels. It was also reported to me that the following morning GSRD surveyed my office and found only suspected contamination on the corner of one piece of paper on my desk. They confiscated the pad that contained the paper. GSRD also too the lab coat that I and used earlier in the week for some of the ¹³³Ba work. I was told that this coat was also slightly contaminated.

Friday 4 May 2018 morning I

It was reported to me that GSRD went into B157 around 4pm on Thursday to assess the state of the room for possible gross contamination. Lizbeth was there with Manny and John Zometsky. I was told "They checked the carts, racks, hoods, box and counter and no gross contamination was found. Manny believes that the contamination was contained to B47. They swiped the floor and will have a result for us tomorrow."

In the morning, Lizbeth and I entered B157 to completely strip and survey the room since it had yet been cleaned up since the SRM production. We first surveyed the room with hand held meters. We systematically surveyed and stripped all surfaces on carts, countertops, and fume hoods where the ¹³³Ba work had been performed. We took many smears and counted them on

a Biodex AtomLab Swipe Test Counter that we installed in the corridor outside the room. We never detected anything above background levels. Attached is the data sheet for the surveys. The floor was also completely wiped down with a Swiffer mop.

We requested that GSRD pick up the 7 bags of rad waste from the room (including that from the SRM production) and that they perform a complete survey of the room.

Personal monitoring on exiting B157 was performed with both the half-body and hand & foot PCMs. No contamination was detected.

Tuesday 8 May 2018 morning

Lizbeth and I went to B46 and B??, accompanied by Chris Young (GSRD), to clean the trace contamination on the ion chamber holders. Prior to washing the holders, we covered the area surround in the sink in B46 with paper. I did the washing, and was garbed in lab coat, sleeve protectors, gloves, and booties. Chris and Lizbeth assisted me. Chris brought the holders in bags to B46. Lizbeth handled providing me with paper towels for cleaning and receiving the cleaned holders. I washed them with detergent from a spray bottle and copious quantities of water, performing the washing of all parts three times, including swabbing the insides of the holder cups with a wetted paper towel. The chamber A holder stick was said to have about 15 Bq and its cup about 4 Bq. After cleaning and on checking both parts together, the contamination only decreased slightly. The holder cord was thought to maybe be the culprit in not lowering the level, so it was removed and disposed of. I then recleaned the entire holder, and on a re-survey in the HPGe detector well, no ¹³³Ba gamma lines were detected.

In all 14 of the chamber B holders were cleaned in the same way. Each had trace contaminations in the range of < 1 Bq to 4 Bq. As of 4pm on Tuesday, only two of the 14 were re-measured and both had no detectable, remaining 133 Ba contamination.

I surveyed myself several times throughout the cleaning process (when taking breaks and on leaving the area) on both PCMs. No personal contamination was detected. We concluded for the day at about 1:30 pm. I picked up all the paper surrounding sink for rad disposal before closing B46 for the day. GRSD (Sarah Yu) took smears around the sink area and floor.

Wednesday 9 May 2018 morning

Lizbeth, Chris Young (GSRD), and I continued decontaminating and assessing the auto chamber holders. Procedures were identical to that described for Tuesday, 8 May. Of the 14 holders cleaned on Tuesday, only three were not "cleared" to background levels. These three were recleaned and re-assayed, and subsequently cleared. The remaining 27 holders were cleaned. On assessment, 15 were clear. Two of those not cleared were re-cleaned and were subsequently cleared. Unfortunately, some of the holders were initially surveyed in batches of 5 holders. These holders were individually cleaned, but were re-assessed in a batch of 5. So, it is not apparent as

to which of the 5 in a batch are still not clear. Individual surveys of the holders are presently underway.

Decontamination efforts on the remaining holders will continue next week, starting on Tuesday, 15 may 2018.

N.B. It is important to recognize that the contamination on all of these holders is at very low – few Bq – level.

FUTURE

Things to be addressed:

- 1. As noted, decontamination of the remaining holders will continue next week.
- 2. The only higher contaminated holder (at 340 Bq) will be decontaminated separately until brought down to a few-Bq level, and then cleaned to a background level.
- 3. The auto chamber will be surveyed more thoroughly, and decontaminated if needed.
- 4. The ¹³³Ba ampoules will be individually examined to try to more clearly identify the root cause of the failed seals.
- 5. Efforts will NOT be made to decontaminate the ampoules. Instead a procedure will be developed and a SOP with Hazard Review written for opening all of the ampoules and transferring the solution to prepare a new ¹³³Ba master solution and then follow the SE 38 production protocol again. This has been done in this laboratory previously twice by me.
- 6. The ampoules for the ¹³³Ba production run (and all future SRMs) will be sealed in 80 mm height ampoules by removal of excess glass with the manual sealer until the auto sealer is available and fully evaluated.

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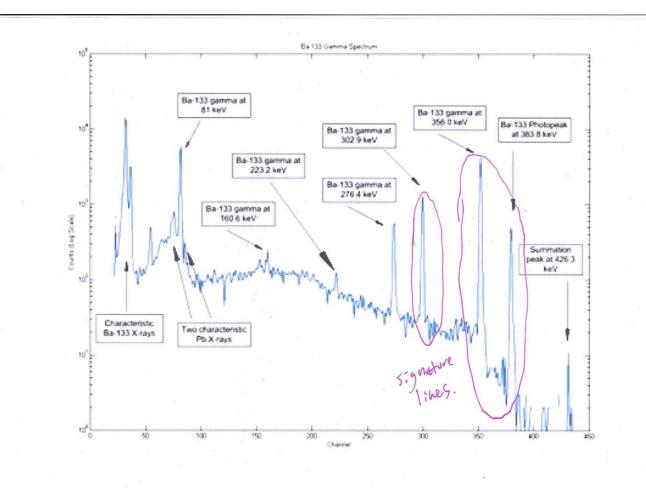
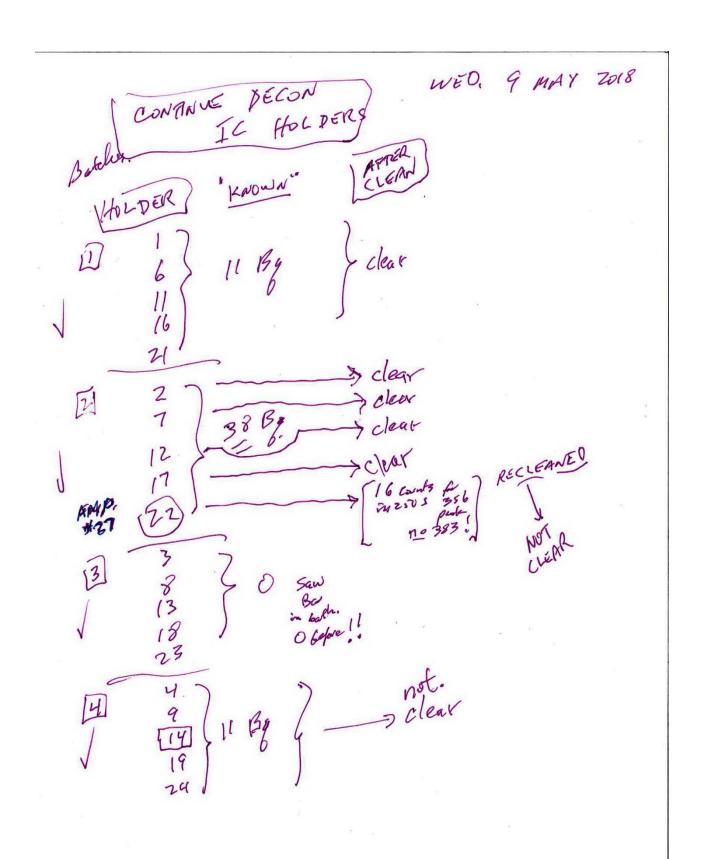


Figure 6.6: The ¹³³Ba gamma spectrum with select features labeled.



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