
Impact of Recent Change in the National Institute of Standards and Technology Standard for ^{18}F on the Relative Response of ^{68}Ge -Based Mock Syringe Dose Calibrator Standards

Brian E. Zimmerman, Denis E. Bergeron, and Jeffrey T. Cessna

Physical Measurement Laboratory, National Institute of Standards and Technology, Gaithersburg, Maryland

As a result of a recent change in the National Institute of Standards and Technology (NIST) activity standard for ^{18}F , we have determined new relative response ratios for a ^{68}Ge solid epoxy mock syringe source used in activity calibrators as a long-lived substitute for ^{18}F . New standardized solutions of each radionuclide were used to determine the response ratios while maintaining traceability to national standards. This work updates our previously published data from 2010. **Methods:** Following our previously published methodology, solution-filled mock syringe sources, identical in geometry to the solid ^{68}Ge epoxy calibration source currently on the market, were prepared using NIST-calibrated solutions of $^{68}\text{GeCl}_4$ and ^{18}F -FDG and directly compared in several models of activity calibrators to determine empirically the relative response ratios for these 2 radionuclides. **Results:** The new relative response ratios measured in this study reflect the change in ^{18}F activity measurements that arise from the recent -4% change in the NIST activity standard. The results allow the ^{68}Ge activity of the mock syringe source to be expressed in terms of equivalent ^{18}F activity, with a relative combined standard uncertainty of about 0.8% for the activity calibrators used in this study. **Conclusion:** This work revises our previously derived relative response ratios for ^{18}F and ^{68}Ge by -3.7% , allowing users of the commercial mock syringe surrogate source to calibrate their activity calibrators in a way that is consistent with the recent change in the NIST ^{18}F standard.

Key Words: ^{18}F ; calibration; ^{68}Ge ; PET; standards

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In 2 recent publications (1,2), the National Institute of Standards and Technology (NIST) reported on a change of -4% in its radioactivity standard for the positron emitter ^{18}F . In addition to the obvious change that this brings in directly measuring this radionuclide in activity calibrators, it has an influence on the relative response ratio that allows ^{68}Ge -based solid mock syringe sources to be used as long-lived calibration artifacts for ^{18}F in those instruments.

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For correspondence or reprints contact: Brian E. Zimmerman, National Institute of Standards and Technology, 100 Bureau Dr., Stop 8462, Gaithersburg, MD 20899-8462.
E-mail: bez@nist.gov
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In 2010, we published a procedure for calibrating a specific commercially available mock syringe source for ^{68}Ge content (3). At the same time, we provided data on the relative response between that source and the equivalent activity of ^{18}F when measured in several models of activity calibrators. Although it would have been possible to merely take the magnitude of the published change in the ^{18}F standard and recalculate a new relative response ratio, a rigorous approach involving new direct measurements of sources containing calibrated ^{68}Ge and ^{18}F solutions gives more confidence in the results. Direct measurement of the ratios has 2 distinct advantages: direct traceability back to the NIST primary standards for the 2 radionuclides is maintained, and a proper uncertainty assessment for the new ratios is possible. We have undertaken a new series of measurements to experimentally determine the new ^{18}F -to- ^{68}Ge relative response ratios for the 6-mL (filling volume nominally 3 mL) epoxy-based ^{68}Ge mock syringe source to reflect the change in the NIST ^{18}F standard.

MATERIALS AND METHODS

Source Preparation

The procedure for preparing and measuring the sources used in this study was similar to that used in the experiments reported by Zimmerman and Cessna (3). Because of the length of time that had passed since those experiments were performed, the activity concentrations of the original solutions were too low and it was necessary to prepare a new set of ^{68}Ge sources from a new standardized solution.

All gravimetric transfers described in this paper were performed using polyethylene aspiration-type pycnometers. The amount of solution transferred during the preparation of any source was determined by mass difference using a calibrated microbalance.

A stock solution containing 150 MBq of $^{68}\text{GeCl}_4$ in 2 mL of $0.5 \text{ mol}\cdot\text{L}^{-1}$ HCl (International Isotopes Idaho, Inc.) was diluted to a total volume of about 18 mL with a carrier solution containing nominally $45 \mu\text{g}$ each of nonradioactive Ge^{+4} and Ga^{+3} per gram of solution in $0.5 \text{ mol}\cdot\text{L}^{-1}$ HCl. Five grams of the solution were then gravimetrically transferred to each of 3 NIST-standard 5-mL glass ampoules.

The solution from one of the ampoules was then used to make a second, diluted solution that was used to make up the ^{68}Ge mock syringe solution masters. The entire 5 g of ^{68}Ge solution were gravimetrically transferred to a 22-mL glass liquid scintillation vial containing 10 mL of a freshly prepared carrier solution containing nominally $65 \mu\text{g}$ each of nonradioactive Ge^{+4} and Ga^{+3} per gram of solution in $0.5 \text{ mol}\cdot\text{L}^{-1}$ HCl. The difference in ion concentrations between the carrier solutions used in the first and second dilutions is a result of variability in the preparation procedure and is not expected to affect the stability of the ^{68}Ge solutions.

After agitating to ensure complete mixing, 3 g of the solution were gravimetrically transferred to each of 3 empty mock syringe cylinders (RadQual, LLC). Between dispensing the first and second mock syringes, 5 mL of the solution was transferred into a NIST standard 5-mL glass ampoule to calibrate the activity concentration of the solution. The ampoule was measured in NIST “4 π ” γ ionization chamber “A” (IC “A”) 40 times, in 4 groups of 10 measurements, alternating with 5 groups of 10 measurements of ^{226}Ra reference source RRS1000. The total activity of the ^{68}Ge solution was determined using the calibration coefficient (K value) for IC “A” that was measured during the original ^{68}Ge standardization in 2008 (4). The ^{68}Ge activity concentration of the solution was calculated by dividing the total ^{68}Ge activity in the ampoule by the transferred solution mass. The resulting activity concentration value was used along with the solution masses contained in each mock syringe to calculate the total ^{68}Ge activity in each syringe.

The ^{18}F mock syringes were prepared in the same manner as for ^{68}Ge , except without the initial dilution step. Approximately 3 mL of ^{18}F -FDG with a nominal activity of 560 MBq was obtained from a commercial radiopharmacy (IBA Molecular) and transferred to a 22-mL glass liquid scintillation vial containing 10 mL of distilled water. After agitation, 3 g of the solution were gravimetrically transferred to each of 3 empty mock syringe cylinders. Between dispensing the first and second mock syringes, 5 mL of the solution were transferred into a NIST standard 5-mL glass ampoule so that the activity concentration of the solution could be determined.

The ampoule was measured in the IC “A” using the same procedure as for the ^{68}Ge ampoule, except that the total activity of the ^{18}F solution was determined using the K value for IC “A” that was determined in 2013 and which is based on the revised ^{18}F standard (1). The ^{18}F activity concentration in each ampoule was calculated by dividing the total activity in the ampoule by the transferred solution mass.

Activity Calibrator Measurements

All the sources described above were measured in the activity calibrators maintained at NIST: a NIST-maintained Vinten 671 ionization chamber (hereafter denoted VIC), Capintec CRC-12, Capintec CRC-15R, Capintec CRC-25PET, Capintec 1.8-ATM (the 1.8-atm chamber from Capintec is the basis of their CRC-PC dose calibrator), and AtomLab AL-500. The primary purpose for measuring the ampoules was to provide a consistency check for both the IC “A” measurements and the activity calibrator setting data that were reported previously (1,2). For each VIC measurement, the ampoule was placed in the ampoule holder, and the current was directly read from a Keithley 6517 electrometer using a LabVIEW interface; 100 readings were acquired at 2-s intervals. The currents were corrected for background and decay corrected to the reference time before being averaged. The sources were remeasured 10 d later using the same counting protocol.

For the Capintec and AtomLab chambers, the calibration curve method (5) was used with the ampoule in the bottom of the appropriate dipper from each chamber; the mock syringes were measured hanging in the syringe holder position of the dipper. For the VIC, the mock syringes were measured using the vial holder and the ampoules were measured using the ampoule holder.

Monte Carlo Calculations

To investigate possible measurement effects due to differences in composition and density between the epoxy and the ^{68}Ge solution, as well as to provide a check on the magnitude of the ^{18}F -to- ^{68}Ge relative response ratio, simulations of the VIC with the liquid- and ^{68}Ge epoxy-filled mock syringes were performed using the PENCYL user code from the 2011 version of the PENELOPE (6) Monte Carlo package. The dimensions and materials of the VIC were obtained from the manufacturer’s technical drawings, and a CT image of the chamber was acquired on a Philips Gemini TF 16 PET/CT scanner. Cross-section

data for all of the materials, with the exception of the pressurized nitrogen in the VIC, came directly from each code package’s materials database. The cross sections for the pressurized nitrogen were calculated using the PENELOPE PENDBASE materials program, assuming molecular nitrogen at standard temperature and a pressure of 1.01 MPa (10 atm). Similarly, the cross sections for the epoxy were calculated using the composition provided by the manufacturer (John Gilbert, Emerson & Cuming, oral communication, 2008) and PENDBASE. The positron spectra for ^{68}Ga and ^{18}F were calculated using SPEBETA (7) and the output reformatted so as to be compatible with the PENCYL input data file format.

Each simulation was run for a minimum of 5×10^6 primary (positron or photon) events. The cutoff energies for photons and electrons (positrons) were 1 keV and 100 eV, respectively.

All nuclear and atomic data used as input for the simulations, as well as for the analysis of experimental data, were taken from the Decay Data Evaluation Project evaluations (8–10).

RESULTS

Unless otherwise specified, uncertainties are given as combined standard ($k = 1$) uncertainties and are calculated as the quadratic sum of individual uncertainty components. Standard uncertainties on numerical values are expressed in parentheses as the uncertainty on the final digits.

The IC “A” measurements of the ^{68}Ge activity concentration for the solution used to make up the mock syringes gave a value of $2.824(13) \times 10^6 \text{ Bq}\cdot\text{g}^{-1}$ at the measurement reference time. The uncertainty is based on components due to: measurement repeatability (0.01% for 40 measurements), the IC “A” calibration coefficient (0.31%, due mostly to the uncertainty on the NIST ^{68}Ge standard), ^{226}Ra reference source ratios (0.05%), positioning of the ^{226}Ra reference source (0.10%), decay correction (0.01%), and a sample holder position correction (0.31%). Likewise, the activity concentration of the ^{18}F solution used to make up the mock syringes was found to be $7.948(29) \times 10^6 \text{ Bq}\cdot\text{g}^{-1}$ at the measurement reference time, with the following uncertainty components: measurement repeatability (0.01% for 40 measurements), the IC “A” calibration figure (0.35%, due mostly to the uncertainty on the NIST ^{18}F standard), positioning of the ^{226}Ra reference source (0.10%), and decay correction ($4 \times 10^{-4}\%$).

In addition to deriving the calibration figures for the VIC ($K_{\text{VIC},x,y}$, where x is either F18 or Ge68 for the 2 radionuclides, and y is either amp or MS for the ampoule or mock syringe geometries, respectively) for the ^{18}F and ^{68}Ge ampoules and the ^{68}Ge mock syringe to confirm our previously published data (2,3), a new $K_{\text{VIC},\text{F18},\text{MS}}$ was also determined for the ^{18}F mock syringe to reflect the change in the NIST ^{18}F activity standard. A comparison of the newly measured $K_{\text{VIC},x,y}$ values with those previously published is given in Table 1.

On the basis of the measured activity concentration of the ^{68}Ge solution and the contained masses, the average $K_{\text{VIC},\text{Ge68},\text{amp}}$ from the 4 ^{68}Ge ampoules was $10.04(5) \text{ pA}\cdot\text{MBq}^{-1}$, where the uncertainty was calculated from the following components: measurement repeatability (average SD of the mean of 0.04% for 100 measurements), the uncertainty on the ^{68}Ge activity (0.45%, including decay correction to VIC measurement time), between-source reproducibility (0.07% for 4 sources), and background ($1.1 \times 10^{-3}\%$). The $K_{\text{VIC},\text{Ge68},\text{amp}}$ value determined in 2008 was $10.121(34) \text{ pA}\cdot\text{MBq}^{-1}$ but was measured in the VIC vial holder instead of the ampoule holder used in the present experiments. We chose to measure the $K_{\text{VIC},\text{Ge68},\text{amp}}$ in the ampoule holder in the present study because the

TABLE 1
Comparison of K_{VIC} Values for Ampoule and Mock Syringe Geometries for ^{18}F and ^{68}Ge

Nuclide, geometry	K_{VIC} (pA·MBq ⁻¹), present study	K_{VIC} (pA·MBq ⁻¹), previous work
^{68}Ge , ampoule	10.04(5)	10.04(3)*
^{68}Ge , mock syringe	10.00(5)	10.00(5)
^{18}F , ampoule	10.31(4)	10.36(4)
^{18}F , mock syringe	10.24(4)	10.29(9) [†]

*On the basis of the reported value of 10.12(3) pA·MBq⁻¹ and corrected for differences in measurement geometry.

[†]Adjusted unpublished value by -4% to reflect change in ^{18}F .

Data are as measured in present study and as reported in Fitzgerald et al. (1) (for ^{18}F) and Zimmerman and Cessna (3) (for ^{68}Ge , denoted SSIC in that reference). Value of K_{VIC} for ^{18}F mock syringe was previously determined during experiments reported in Zimmerman and Cessna but was not included in the paper. Uncertainties are quoted as combined standard ($k = 1$) uncertainties. Details of uncertainty analysis are given in text (for present studies) or in appropriate reference.

ampoule holder provides a much more reproducible geometry than the vial holder. The previous choice of geometry was a matter of convenience because it allowed both the ampoules and the mock syringes to be measured sequentially without switching holders, albeit conceding a small additional uncertainty in the ampoule measurement due to source positioning. A separate set of measurements performed in the present study revealed the magnitude of the effect of changing holders to be 0.79%. Using this value to make a correction to the previously measured $K_{VIC,Ge68,amp}$, we obtained a calibration coefficient of 10.041(34) pA·MBq⁻¹, which is in agreement with the result from the present study.

For the ^{68}Ge mock syringes, the average $K_{VIC,Ge68,MS}$ value was measured to be 10.00(5) pA·MBq⁻¹, where the uncertainty was calculated from components due to: measurement repeatability (average SD of the mean of 0.02% for 100 measurements), the uncertainty on the ^{68}Ge activity (0.45%, including decay correction to VIC measurement time), between-source reproducibility (0.10% for 4 sources), and background ($5.9 \times 10^{-3}\%$). As seen

in Table 1, this new measurement is identical to our previously reported value.

The measured $K_{VIC,F18,amp}$ for the ^{18}F ampoule was measured in this study to be 10.31(4) pA·MBq⁻¹, where the uncertainty was a standard uncertainty calculated from the quadratic combination of components due to: measurement repeatability (average of 0.02% for 100 measurements), the uncertainty on the ^{18}F activity (0.36%), ^{18}F half-life and decay correction ($1.1 \times 10^{-3}\%$), within-measurement repeatability (SD of the mean of 0.03% for 100 measurements), and background ($9 \times 10^{-4}\%$). This value is in agreement with the value reported by Fitzgerald et al. (1) to within their respective standard uncertainties.

For the mock syringe, $K_{VIC,F18,MS}$ was calculated from the present data to be 10.24(4) pA·MBq⁻¹, where the uncertainty components were calculated the same way as for the ampoule, with nearly identical magnitudes for each of the different components. This new $K_{VIC,F18,MS}$ is 5.3% higher than the (unpublished) value determined during the experiments performed by Zimmerman and Cessna (3). This observation is primarily a result of the -4.0% change in the NIST ^{18}F standard and improvements in the measurement protocols for the VIC since those original measurements were made.

Taking the previous $K_{VIC,F18,MS}$ value of 10.72(9) pA·MBq⁻¹ that was determined during the experiments conducted in the 2010 study (3), but not included in the publication, and correcting for the change in the NIST standard, we obtained a value of 10.29(9) pA·MBq⁻¹, which is in agreement with the present determination. The ratios between the old and new $K_{VIC,F18}$ values for both the ampoule and the mock syringe geometries are also in accord within experimental uncertainties, providing additional internal consistency.

The dial settings for the ^{18}F and ^{68}Ge mock syringes determined in our activity calibrators are given in Table 2. The uncertainties on the dial settings are dominated by the uncertainty in the activity calibration for the radionuclide (0.36% and 0.45% for ^{18}F and ^{68}Ge , respectively), although much smaller components due to decay correction, the goodness of fit of the relationship between dial setting and apparent activity, and measurement repeatability were also considered. When compared with the data for the ^{68}Ge mock syringe presented in Table 3 of Zimmerman and Cessna (3), we found that the dial settings for the CRC-12 and CRC-35R were in agreement between the 2 studies. The data reported for

TABLE 2
Measured Dial Settings for ^{18}F and ^{68}Ge Mock Syringes in Several NIST-Maintained Activity Calibrators

Chamber	^{18}F			^{68}Ge		
	Previous dial setting	Dial setting	% activity	Previous dial setting	Dial setting	% activity
CRC-12	472(2)	460(4)	0.6	452(1)	453(3)	0.6
CRC-15R	—	447(3)	0.5	442(1)	436(3)	0.5
CRC-25PET	—	459(2)	0.4	—	451(2)	0.5
CRC-35R	—	452(2)	0.4	444(1)	447(5)	0.9
CRC-1.8 atm	—	496(2)	0.4	—	488(3)	0.5
AL-500	—	10.1(2)	2.0	—	10.1(1)	0.5

Uncertainties ($k = 1$) are given in parentheses and are expressed as uncertainty in last digit of dial setting. % activity column gives uncertainty in measured activity value arising from uncertainty in dial setting. Values for previous dial settings for ^{18}F and ^{68}Ge are from Cessna et al. (11) and Zimmerman and Cessna (3), respectively.

TABLE 3

Average Ratios (R_{avg}) of Response of Commercial Activity Calibrators to ^{18}F and ^{68}Ge in Mock Syringe Geometry at Appropriate ^{68}Ge Dial Setting (Table 2) for Each Specific Calibrator

Chamber	R_{avg}
CRC-12	1.015(8)
CRC-15R	1.022(7)
CRC-25PET	1.016(10)
CRC-35R	1.010(6)
CRC-1.8 atm	1.018(6)
AL-500	0.997(21)

Ratios were obtained by dividing activity readout from chamber for each ^{18}F -FDG source by NIST-calibrated activity for that source. Uncertainties on response ratios were calculated from SD on average measured ratio from 3 sources and average uncertainties on ^{68}Ge and ^{18}F activities in mock syringes.

the CRC-15R are in agreement within their respective expanded ($k = 2$) uncertainties.

The AtomLab 100, which was used in the previous studies of Zimmerman and Cessna (3), has been retired from use in our laboratory. The AL-500 chamber used in this work is relatively new for our laboratory, and we have no previous data against which to compare.

In the VIC, these new results give a ^{18}F -to- ^{68}Ge response ratio of 1.024(6) for the mock syringe geometry. For the Capintec and AtomLab chambers, the ^{18}F -to- ^{68}Ge relative response ratios were measured in the same way as done by Zimmerman and Cessna (3)—that is, by measuring the ^{18}F mock syringes at the appropriate dial setting that had been determined for the ^{68}Ge mock syringes (Table 2). The results of those measurements are also given in Table 3 and supplant the data presented in Table 4 of Zimmerman and Cessna (3).

DISCUSSION

The data presented in this study revise our results previously published in 2010 (3). This revision was prompted by the recent change in the NIST standard for ^{18}F (1). To ensure consistency with previous data presented for ^{68}Ge mock syringes, we have repeated the measurements for the determination of dial settings and calibration coefficients for these sources and checked against previously determined dial settings for the NIST ampoule. In all cases, agreement between the 2010 data and the present experiments was achieved within the experimental uncertainties.

For ^{18}F , the same approach was used with the NIST ampoules to give confidence in the correctness of the ^{18}F mock syringe dial setting determinations. In 2010, using the previous NIST ^{18}F standard, we measured the relative response ratio of the mock syringes for the Capintec chambers to be 1.054(10). If the 2010 ^{18}F activities are adjusted by -4.0% , we obtain an average ratio of 1.012(10), which is in accord with the ratios recovered for all of the activity calibrators studied. This means that a mock syringe containing a certain amount of ^{18}F would give an activity value (at the ^{68}Ge setting) that now reads 1.2% high, as opposed to the previous value of 5.4%.

The measured relative response ratio of 1.024(6) obtained for the VIC for the mock syringes in this study is in agreement with the previously measured (and adjusted for the change in the ^{18}F standard) value of 1.029(10) for that chamber. With our improved model of the chamber and a larger number of events, the PENELOPE 2011 calculations predict a ratio of 1.017(8), with the uncertainty estimated from the uncertainty on the dose deposited in the ionization chamber gas as reported by the simulation and the uncertainties in the positron branching ratios and emission rate for the 1077 keV γ -ray. Within the respective uncertainties, agreement can be seen between the Monte Carlo prediction for the relative response, the previous (adjusted) measured value, and the ratio obtained from the present study, thereby providing a degree of confidence in the newly measured result.

The Monte Carlo results also provided verification that no attenuation correction is required when relating the activity in the liquid-filled mock syringe sources to the activity in the solid epoxy mock syringes. The ratio of calculated doses deposited in the ionization chamber gas for the solid mock syringe to the solution-filled mock syringe was 0.995(4) and is consistent with the results given in our previous work (3).

Information regarding the change in the NIST ^{18}F standards and the updated relative response ratios for the mock syringes in the Capintec and AtomLab chambers has already been transmitted to the calibration source and instrument manufacturers.

For most clinical applications, the change in the NIST ^{18}F standard will have a relatively minor impact on routine practice. The NIST ^{68}Ge standard has not changed, thus the calibrated value for ^{68}Ge activity in a traceable source is still valid. If the solid epoxy ^{68}Ge mock syringe source is used to calibrate an activity calibrator for ^{68}Ge , the user should expect to obtain the same calibration factors (dial settings) as they always have, but now the equivalent activity of ^{18}F is only 1.015–1.02 times the ^{68}Ge activity, instead of 1.054. Thus, the ^{18}F calibrated activity will shift to a lower value and should give dial settings close to those given in Table 2. If a clinical site obtains its ^{18}F from a commercial radiopharmacy that has not yet changed its calibration to match the new NIST standard, a difference between the measured value and activity on the label could be evident and should be investigated.

Because most calibrations for PET scanners are made relative to the activity reported by an activity calibrator, scanners should be recalibrated once the updated ^{18}F equivalent activity value is adopted in the radiopharmacy. Otherwise, a systematic 4% difference between the scanner-reported activities and the true injected activity will occur. This will also affect the calculation of standardized uptake values by 4%, since the relationship between the activity of ^{18}F used to calibrate the scanner and the calibration of new patient dosages is no longer the same.

CONCLUSION

In this work, we have performed a series of experiments to redetermine the relative response ratio between ^{68}Ge and ^{18}F for a commercially available solid ^{68}Ge mock syringe source. This -3.7% change was prompted by a recent revision of the NIST activity standard for ^{18}F . These new results allow the calibration of activity calibrators to once again be made using that source in a way that can be related back to NIST standards of activity.

DISCLOSURE

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No other potential conflict of interest relevant to this article was reported.

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