

THE USE OF ELEMENT RATIOS TO ELIMINATE ANALYTICAL BIAS IN COLD NEUTRON PROMPT GAMMA-RAY ACTIVATION ANALYSIS

R. L. PAUL

*Department of Commerce, Technology Administration, National Institute of Standards and Technology,
Analytical Chemistry Division, Gaithersburg, MD 20899 (USA)*

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Analytical bias due to neutron scattering and absorption in cold neutron prompt gamma-ray activation analysis (CNPAA) is largely eliminated for homogeneous samples when element ratios are measured. Application of sensitivity ratios (measured relative to titanium) to the multielement analysis of the Allende meteorite increases both the speed and accuracy of the measurement. Greater measurement accuracy is achieved for some samples when ratios of element concentrations are reported. Problems are encountered when applying the ratio method to measurement of elements which deviate from $1/v$ behavior, and when gamma-ray attenuation or sample heterogeneity are significant.

Elements sensitivities ($\text{cps} \cdot \text{mg}^{-1}$) in neutron capture prompt γ -ray activation analysis (PGAA) are dependent upon the geometry and composition of the sample. If the sample matrix contains large concentrations of neutron absorbing nuclides, self shielding occurs, resulting in a decrease in element sensitivities. The presence of large concentrations of neutron scattering nuclides, most prominently hydrogen (σ_s bound = 80 barns), may increase or decrease sensitivities, depending on the geometry of the sample.^{1–4} When long wavelength “cold” neutrons are used in the analysis, the effects of neutron absorption and scattering on sensitivities increase dramatically,⁴ because of higher neutron cross sections (relative to thermal neutrons). Furthermore, scattering of cold neutrons within a warm target results in an increase in the average neutron energy, and a significant decrease in element sensitivities.^{4,5} Sensitivities for elements in hydrogenous targets have been observed to decrease with both increasing H content and target thickness.

In order to obtain accurate analyses by CNPAA, the effects of neutron self shielding and scattering on element sensitivities must be dealt with. One approach is to correct the data for these effects. Count rates may be corrected for self shielding by using well-known absorption laws.⁶ Alternatively, element sensitivities may be calibrated as a function of matrix neutron absorbing and scattering power. The second method is time consuming, and the first is valid only if the target composition, density

and appropriate absorption and scattering cross-sections (σ_a) are known, and if the sample size and shape are well-defined. A more reliable approach to the problem has been to employ standards with a matrix and geometry which closely matches that of the sample being analyzed. This approach is also time consuming since new standards must be prepared each time a new sample matrix is analyzed.

The ratio method

An alternate approach to the elimination of analytical bias in PGAA is to measure sensitivity ratios rather than absolute sensitivities. This approach is analogous to the method of k_0 factors which has proven of great value with activation products, but has barely begun to be applied explicitly to PGAA.⁷⁻⁹ For a specific set of experimental conditions, the net peak area A_x of γ -ray of energy E_γ from element x in the spectrum of a homogeneous sample is given by:

$$A_{x,E_\gamma} = \left[\frac{N_0 m_x}{M_x} \right] \varepsilon(E_{\gamma,x}) \theta_x \Gamma_{x,E_\gamma} t_i \Phi_{th} \left[g \sigma_{0,x} + I'_0 \frac{\Phi_{epi}}{\Phi_{th}} \right] \quad (1)$$

where N_0 is Avogadro's number, m_x is the mass of the element x , M_x is the atomic weight, $\varepsilon(E_{\gamma,x})$ is the counting efficiency, θ_x is the abundance of the capturing isotope, Γ_x is the γ -yield in photons per unit capture, t_i is the irradiation (counting) time, Φ_{th} is the thermal neutron flux, g is the Wescott constant, $\sigma_{0,x}$ is the thermal capture cross section ($2200 \text{ m} \cdot \text{s}^{-1}$ neutrons), I'_0 is the infinitely dilute resonance integral:

$$I'_0 = \int_{\mu kt}^{\infty} \sigma(E) dE/E \quad (2)$$

and Φ_{epi} is the epithermal neutron flux.¹⁰ In the case of the cold neutron PGAA instrument at NIST, only neutrons with wavelengths greater than 4 \AA are present in the beam, and the epithermal flux is negligible. If we normalize the peak area of element x to the corresponding expression for the peak area of a monitor element s , we obtain the following:

$$k_{x,s} \equiv \frac{A_x/m_x}{A_s/m_s} = \frac{\theta_x \Gamma_x \sigma_{0,x} \varepsilon_{\gamma,x} / M_x}{\theta_s \Gamma_s \sigma_{0,s} \varepsilon_{\gamma,s} / M_s} \quad (3)$$

At this point we make several assumptions. At neutron energies less than 5 meV, for nearly all elements, the neutron capture cross section (σ) is inversely proportional to

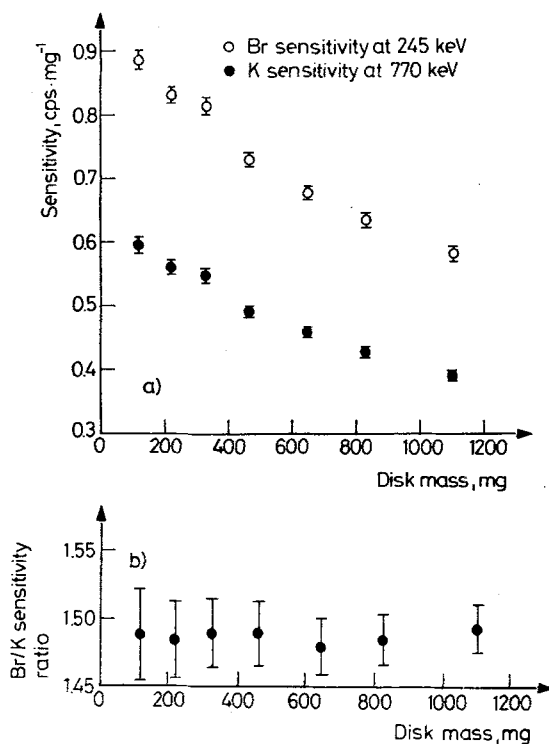


Fig. 1. K and Br CNPGAA sensitivity vs. sample mass (a) and Br/K CNPGAA sensitivity ratio vs. sample mass for cylindrical disk targets (b) prepared from KBr/urea mixtures. Uncertainties are 1σ based on counting statistics, variation in neutron flux, and sample positioning

velocity (v), hence $\sigma = \sigma_0 \cdot v_0 / v$, where v_0 is the standard velocity of a slow neutron ($2200 \text{ m} \cdot \text{s}^{-1}$). We may therefore assume the ratio of capture cross sections, σ_x / σ_s , to be unaffected by matrix or geometry of the sample. We also assume that for a specific experimental setup, the shape of the γ -ray efficiency vs. energy curve is independent of sample composition and is constant over the volume of the sample (γ -self absorption is negligible). Thus $k_{x,s}$ simply becomes the ratio of the experimentally measured sensitivity for element x to that of element s , and is independent of sample geometry or composition.

Equation (3) has been tested by measuring capture rates of cold neutrons by K and Br in a series of 13-mm disks of different thickness made from a homogeneous mixture of stoichiometric KBr with urea.⁸ Although increasing the target thickness from 1 to 6 mm causes K and Br sensitivities to drop by almost 35% (Fig. 1a), the Br/K sensitivity ratio remains constant (Fig. 1b).

Applications

Determination and verification of relative sensitivities

Interference-free sensitivities for many elements have been measured using the cold neutron PGAA instrument located in the Cold Neutron Research Facility (CNRF) at NIST. The instrument is described in detail elsewhere.^{11,12} A sensitivity for a given element at a specific γ -ray energy is usually obtained from measurement of a standard consisting of a single element or a simple compound. In order to minimize the effects of neutron scattering and self shielding, a thin foil or cylindrical disk of nonhydrogenous composition is used as a standard whenever possible. Since sensitivity also depends upon the strength of the neutron flux and the experimental setup (i.e., target-to-detector distance, neutron and γ -ray collimation), sensitivities are normalized to the count rate of the 1381 keV γ -ray peak given by a titanium foil of comparable geometry to the standard being measured.

The accuracy of individual sensitivity measurements for some elements has been assessed by determination of sensitivity ratios in mixtures. Table 1 gives standards used for measurement of sensitivities for eight elements (H, K, Cr, Mn, Fe, Ni, Cu and Gd) and Ti. Sensitivity ratios for these elements, and for Co, relative to titanium were

Table 1
Standards used for measurement of element sensitivities

Element	Standard
H	<i>tris</i> (hydroxymethylamino)methane disk, 67 mg
K	KIO ₃ disk, 493.6 mg
Ti	Ti foil, 147.5 mg
Cr	Cr "chunk", 370.1 mg
Mn	Mn "chunk", 180.6 mg
Fe	Fe foil, 251.5 mg
Ni	Ni foil, 101.7 mg
Cu	Cu foil, 257.7 mg
Gd	0.153 mg Gd (deposited from solution) on Whatman filter paper

determined from analyses of solution and powder mixtures. Solution mixtures were prepared by combining milliliter amounts of NIST Standard Reference Material (SRM) spectroscopic solutions of titanium and another element (see Table 2) and evaporating 500–1000 μ l of the mixture on a 0.03 mm thick sheet of FEP teflon. The teflon sheet was folded into a bag prior to analysis. Powder mixtures were prepared by combining

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Table 2
Spectroscopic solution mixtures

NIST SRM	Element	Concentration, mg/ml	ml used	ml Ti solution used*
3141a	K, in 1% HNO ₃	10.00	2	1
3112a	Cr, in 10% HNO ₃	10.00	2	1
3132	Mn, in 10% HNO ₃	10.00	1	1
3126a	Fe, in 10% HNO ₃	10.00	5	1
3113	Co, in 10% HNO ₃	10.00	1	1
3136	Ni, in 10% HNO ₃	10.00	2	1
3114	Cu, in 10% HNO ₃	10.00	2	1
3118	Gd, in 10% HCl	0.00196**	1	1

*SRM 3162, 10.00 mg/ml Ti, in 10% HNO₃ and 2% HF.

**SRM solution diluted with deionized H₂O.

Table 3
Powder mixtures

Element	Powder	mg	mg Ti powder
H	Urea	231.4	78.5
Mn	MnO ₂	75.7	53.8
Fe	Fe	147.2	42.0
		204.8	41.7
Co	Co	101.8	104.5
		105.6	115.8
Cu	Cu	107.3	38.9

milligram amounts of a high purity titanium powder with a second powder in a Spex 800 Mixer Mill (see Table 3), then transferring the mixture to a teflon bag.

Relative sensitivities from ratio measurements are compared with ratios calculated using individually measured sensitivities in Table 4. In all cases, ratios obtained from solution mixtures are in agreement with those obtained from analysis of powder mixtures. When metal foils or thin disks of pure materials were used as standards, ratios calculated from individual sensitivity measurements are in agreement with those measured from mixtures. Discrepancies are seen only for Mn and Cr, where standards of suitable geometry and composition could not be obtained. Sensitivities for Mn and Cr, measured from analyses of small "chunks" of metal, are apparently low. Using equations given by FLEMING⁶ for neutron self shielding for a sphere (assuming the standards to be roughly spherical) and estimating cold neutron capture cross sections to

Table 4
Sensitivities for selected elements relative to Ti (1381 keV peak)

Element (Gamma-energy, keV)	Solution mixture*	Powder mixture*	Measured individually**
H (2223)	—	2.40 ± 0.01	2.36 ± 0.12
K (770)	0.320 ± 0.006	—	0.321 ± 0.005
Cr (835)	0.352 ± 0.006	—	0.310 ± 0.005
Mn (212)	1.47 ± 0.03	1.44 ± 0.02	1.19 ± 0.02
Fe (352)	0.122 ± 0.005	0.126 ± 0.001 0.125 ± 0.001	0.125 ± 0.002
Co (556)	1.64 ± 0.03	1.66 ± 0.02 1.66 ± 0.02	—
Ni (465)	0.305 ± 0.005	—	0.302 ± 0.004
Cu (278)	0.398 ± 0.007	0.398 ± 0.005	0.398 ± 0.006 0.401 ± 0.006
Gd (182)	1630 ± 30		1640 ± 20

*Uncertainties are 1σ , based on counting statistics.

**Ratios calculated using sensitivities, determined from measurement of standards listed in Table 1. Uncertainties are 1σ , based on counting statistics, sample positioning, and neutron flux variation.

be 2–3 times thermal values, it was determined that spheres of radii 1–2 mm for Mn and 2–3 mm for Cr would undergo sufficient self shielding to account for the low sensitivities. These dimensions correspond roughly to those of the standards used.

Determination of absolute concentrations using relative sensitivities

Carefully measured sensitivity ratios may be used to facilitate multielement determinations in CNPGAA. If element sensitivities have been measured relative to a comparator element (in this case, titanium) then it is necessary to measure only the sample and a single standard containing the comparator element. The standard should be of comparable geometry to the sample, and the matrix should be of comparable neutron scattering and absorption power.

In a previous investigation, CNPGAA was applied to the determination of elemental concentrations in the Allende meteorite.¹² Allende powder was mixed with graphite and the mixture pressed into a cylindrical pellet, 13 mm diameter. The pellet was then analyzed along with separate standards for each element analyzed. The results for selected elements are given in Table 5, along with literature values.

Concentrations of these same elements have also been calculated using experimentally verified sensitivity ratios, and count rates given by the Allende pellet

Table 5
Selected elements determined in the Allende Meteorite

Element (concentration)	Concentrations determined		Literature value
	Using individual standards*	Using ratios**	
H ($\mu\text{g/g}$)	150 ± 30	150 ± 30	$190, 180^{13}$ $110, 150^{14}$
K ($\mu\text{g/g}$)	390 ± 80	400 ± 80	300 ± 80^{15}
Cr (%)	0.46 ± 0.01	0.37 ± 0.01	0.36 ± 0.01^{15}
Mn (%)	0.165 ± 0.005	0.133 ± 0.004	0.15 ± 0.01^{15}
Fe (%)	22.4 ± 0.3	22.9 ± 0.4	23.57 ± 0.08^{15}
Co ($\mu\text{g/g}$)	-	650 ± 20	600 ± 100^{15}
Ni ($\mu\text{g/g}$)	1.60 ± 0.03	1.33 ± 0.03	1.42 ± 0.02^{15}

*Same standards as listed in Table 1, except Ni (Ni powder/graphite disk: 269 mg Ni, 553 mg graphite). Uncertainties are 1σ based on counting statistics, background subtraction, sample positioning, and neutron flux variation.

**Calculated using average of ratios measured in solution and powder mixtures.

Uncertainties are 1σ based on ratio uncertainties, sample statistics, and background subtraction.

and a titanium foil of comparable diameter. Neutron self shielding and scattering in both the pellet and the foil were minimal. Elemental concentrations calculated in this manner are given in Table 5.

For most elements, concentrations calculated from sensitivity ratios are comparable with literature values. Comparable or better agreement with literature values was obtained when sensitivity ratios were used instead of individual standards. The results indicate that the use of experimentally verified sensitivity ratios may serve to improve both the speed and accuracy of multielement determinations.

Expressing concentrations as ratios

In some instances it is difficult to accurately determine absolute concentrations by CNPGAA. Nevertheless, if the relative sensitivities of two elements present in the sample are well known, the ratio of the concentrations of these elements may be accurately determined. Furthermore, if the concentration of one of these elements is already known, the concentration of the other element may be determined. This approach, which has been commonly used in CNPGAA analyses at NIST, is especially applicable in cases where the sample analyzed is larger than the neutron beam (hence the mass of sample analyzed is unknown) and where sensitivities are not easily

corrected for matrix effects (such as highly hydrogenous matrices). A few examples of these measurements follow.

In order to study the effects of hydrogen embrittlement in titanium alloy compressor blades, hydrogen was measured in several blades (of dimension 9 cm × 25 cm) from a turbine engine.¹⁶ Measurements were made by irradiating each blade at numerous locations. The mass of sample from each analysis was not known (the blade was larger than the neutron beam), hence measurements were reported as hydrogen/titanium ratios. In a similar investigation, CNPGAA was used to determine hydrogen in 32 × 10 × 0.34 cm³ titanium alloy panels prepared as hydrogen standards for neutron tomography measurements.¹⁷ Hydrogen/titanium concentration ratios were measured in numerous locations on each panel. Since the titanium content of the alloy was known, the ratio measurements were readily converted to absolute hydrogen concentrations. The results were in agreement with hydrogen concentrations measured using a LECO* inert gas fusion analyzer.

Element ratio measurements have also been used for characterization of C₆₀ fullerenes and related compounds (such as K₃C₆₀).^{12,18,19} Since these samples are often analyzed sealed inside a metal container, the exact sample geometry is unknown. Finally, in a recent study on the uptake of hydrogen by proton conductors, hydrogen/cerium ratios were measured in a series of Yb doped SrCeO₃ pellets.²⁰ The results were in agreement with hydrogen uptake of the pellets determined using a thermal balance.

Problems with ratio measurements

Non 1/v behavior

We have previously noted that scattering of cold neutrons within a room temperature target results in an upward shift in the average neutron energy, and a corresponding decrease in element sensitivities. Since neutron capture cross sections for most elements vary as 1/v at low energies, sensitivity ratios are generally unaffected by neutron scattering (Fig. 1b). Such may not be the case, however, when one or both of the elements measured possess an isotope whose neutron cross section exhibits a low energy resonance.

In a previous study, Gd sensitivities were observed to undergo the same relative decrease with neutron scattering power as those of 1/v elements (H, Br, K), while Sm sensitivities exhibited smaller decreases.⁴ This was attributed to the fact that Sm

*Certain commercial equipment, instruments, or materials are identified in this paper in order to specify the experimental procedures in adequate detail. Such identification does not imply recommendation by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

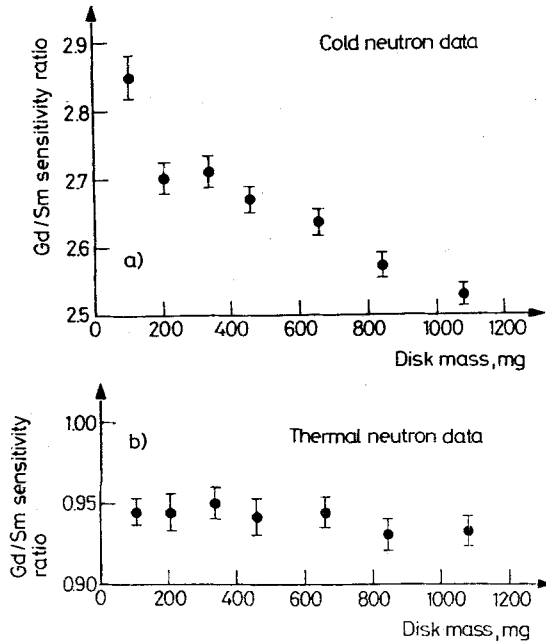


Fig. 2. Gd/Sm sensitivity ratio vs. sample mass for cylindrical disk targets prepared from Gd/Sm doped urea, measured by cold neutron prompt gamma analysis (a) and by thermal neutron prompt gamma analysis (b). Measurements were made using Gd and Sm gamma-rays at 182 and 333 keV. Uncertainties are 1σ based on counting statistics

possesses a low energy resonance at 0.09 eV. (Gd also exhibits a low energy resonance at 0.03 keV, both the magnitude of the resonance is much smaller than that of Sm.) Gd/Sm sensitivity ratios measured in Gd/Sm doped urea disks of different thickness are plotted versus sample mass in Fig. 2. Figure 2a gives ratios measured using the NIST cold neutron PGAA instrument, while ratios in Fig. 2b were measured using the U.Md-NIST thermal neutron PGAA spectrometer. While ratios measured by thermal neutron PGAA remain constant (samples and the neutron beam are both ~ 300 K, therefore no neutron warming occurs and no change in neutron capture cross sections), CNPGAA Gd/Sm ratios exhibit an 11% decrease over the range of sample sizes measured.

Preliminary studies indicate that cooling of the sample to liquid nitrogen temperatures may mitigate the effects of scattering-induced neutron warming in CNPGAA.⁵ The future installation of a cryogenic sample chamber may improve the accuracy of measurements involving elements with low energy resonances.

Gamma-ray attenuation

When very thick samples or samples with large concentrations of high Z elements are analyzed by PGAA, the effects of γ -ray self attenuation may be significant. In this case the γ -ray efficiency vs. energy curve is no longer constant over the volume of the sample, and sensitivity ratios are no longer independent of sample geometry and composition. Since attenuation increases exponentially with decreasing γ -ray energy, the effect on ratios is greatest when the two γ -ray peaks being compared are of very different energies, particularly when one or both γ -rays are of low energy (< 500 keV). The best course of action is to reduce self absorption by reducing sample thickness, if possible. The effect may also be reduced if the γ -rays chosen for the two elements are of comparable energy, which is again not always possible. If the geometry and composition of the sample are known, self attenuation of γ -rays may be corrected using derived equations.²¹ Alternatively, if one of the elements present in the sample matrix emits a spectrum of γ -ray lines covering a range of energies, comparison of the relative intensities of these lines with those measured in a thin foil (where self attenuation is minimal) may allow for calibration of γ -attenuation as a function of energy. This approach has been used in the determination of hydrogen in titanium alloys.¹⁷ The attenuation of the hydrogen 2223 keV peak relative to the titanium 1381 keV peak in a titanium alloy plate was determined by comparing relative intensities of titanium lines in the sample with those measured in a thin titanium foil.

Attenuation may also be a problem when a semi-thick γ -ray collimator is used, and the sample is either larger than or not properly aligned with the collimator. To illustrate this, four titanium foils of comparable thickness and different diameter were measured, and the ratio of the count rates for the titanium 341 and 1381 keV peaks plotted vs. foil diameter (Fig. 3). The γ -ray signal was first collimated by a 2 cm aperture in copper, then the measurements were repeated using a copper collimator with a 5 cm aperture. Ratios decrease with increasing foil diameter, the amount of decrease being smaller with the larger collimator. The results are consistent with what would be expected if γ -rays in the larger foils were attenuated by the collimator. The fact that the ratios do not remain constant over the diameter of the collimator indicates that the samples may not have been properly aligned with the center of the collimator. The results indicate that more reliable ratio measurements are obtained when the sample size is small compared with the aperture of collimation of the γ -ray signal. For larger samples, replacement of the copper collimator with a more effective γ -ray absorbing material (lead or tungsten) will serve to reduce the effect of collimation on ratio measurements.

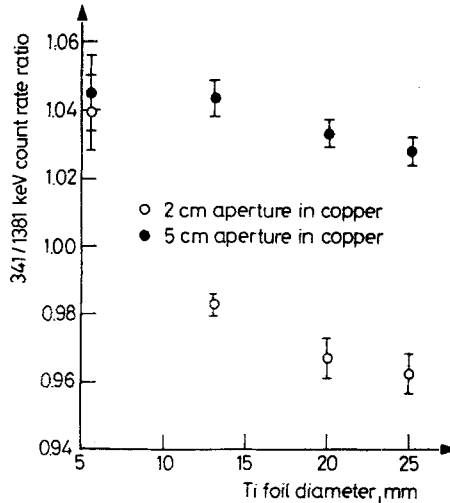


Fig. 3. Count ratio of 341 to 1381 keV peaks vs. Ti foil diameter. Gamma-ray signal was collimated with 2 cm and 5 cm copper collimators. Uncertainties are 1σ based on counting statistics

Heterogeneous samples

It should be emphasized that the ratio method is valid only when the sample being measured is homogeneous. If the sample matrix is grossly heterogeneous, the efficiency vs. energy curve may not be constant for all elements over the volume of the sample. Furthermore, it can no longer be assumed that neutron scattering and absorption affect sensitivities of all elements in the matrix equally. Accurate analyses of such samples by PGAA requires knowledge of the distribution of element concentrations over the volume of the sample. The homogeneity of a sample may be assessed by analyzing it in different orientations. For example, if significantly different ratios are measured in a sample which has been rotated 180° to its original orientation, heterogeneity may be a problem. In the future it may also be possible to measure lateral variations in composition in thin samples without loss of sensitivity by using a neutron lens, which focuses the beam to a small spot size (1 mm).²²

Conclusions

Element ratios have played a significant role in the analysis of homogeneous samples in cold neutron prompt γ -activation analysis. Further measurements are necessary to obtain accurate sensitivity ratios (relative to titanium) for all elements. Once these sensitivity ratios have been measured, and γ -ray efficiencies have been measured as a function of energy, then it will be possible to calculate k_0 factors which may be used by

researchers at other facilities. The effect of sample temperature upon sensitivity ratios in CNPGAA also requires further research. This is especially significant, since the replacement of the current D₂O cold source at NIST with a liquid hydrogen cold source will result in a neutron beam of lower average energy. This may lead to an even greater up shift in the energy of the neutron beam when neutrons are scattered within a room temperature target.

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