NEUTRON SCATTERING BY HYDROGEN IN COLD NEUTRON PROMPT GAMMA-ACTIVATION ANALYSIS

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The effects of neutron scattering by hydrogen within targets for cold neutron prompt γ -ray activation analysis (CNPGAA) have been characterized. For most targets studied, the probability for neutron absorption, and hence CNPGAA sensitivities (counts \cdot s⁻¹ · mg⁻¹), decrease with increasing H content and with target thickness. Comparisons with results from thermal neutron PGAA indicate that the effects of cold neutron scattering differ from those of thermal neutron scattering. CNPGAA sensitivities for "1/v" nuclides show similar sensitivity decreases, while Sm sensitivities show smaller decreases.

Element sensitivities (counts $s^{-1} \cdot mg^{-1}$) in neutron capture prompt γ -ray activation analysis (PGAA) are dependent upon the average neutron fluence rate (Φ) within the volume of the sample. This average fluence rate depends not only on the incident fluence rate, but also on the target size and shape and the neutron absorbing and scattering power of the target matrix. If the matrix contains large concentrations of absorbing nuclides, self shielding occurs, resulting in a decrease in Φ and a corresponding decrease in element sensitivities. If the target composition, density and appropriate absorption cross-sections (σ_a) are known, and if the sample size and shape are well-defined (e.g., a sphere, a slab, or a cylinder), this self-shielding effect may be corrected for using well-known general absorption laws.¹

The effects of neutron scattering by H, a very strong scatterer with a bound-atom scattering cross-section (σ_b) of approximately 80 b, on thermal neutron PGAA (TNPGAA) element sensitivites have been studied previously.²⁻⁴ Element sensitivities vary as a function of the amount of scatterer and of the target size and shape. Monte Carlo simulations⁵⁻⁷ have shown that elastic neutron scattering i.e., scattering without change in the neutron energy, can account for changes in sensitivities. Elastic scattering affects absorption rates by altering the average distance traveled by the neutrons within the target. Depending upon the target size, shape, and scattering power, this scattering may either increase or decrease element sensitivities relative to sensitivities for pure absorbers. Experiments by MACKEY et al.^{3,4} have shown that while element sensitivities are enhanced in hydrogenous disk-shaped targets that are thinner than they are wide, spherical targets show little or no change for many elements in hydrogenous

matrices. Results of Monte Carlo simulations for similar targets compare well with experimental results.⁷

A recent innovation in PGAA has been the use of beams of cold neutrons.⁸⁻¹⁰ For cold neutrons, possessing wavelengths >4 Å, or energies of <0.005 eV, the value of σ_a for most elements is about two to three times greater than for thermal neutrons (average wavelength 1.8 Å or average energy of 0.005 eV). Larger values of σ_a result in higher cold neutron PGAA (CNPGAA) sensitivities (relative to thermal) for most elements, and therefore an increase in target self-shielding. The effects of neutron scattering on CNPGAA have not previously been characterized.

We have begun to characterize the effects of neutron scattering by H on element sensitivities in CNPGAA as a function of H concentration and of target geometry. Sensitivities have been measured for (1) a series of liquid targets of different H concentrations, contained in 1.2×1.2 cm² Teflon bags, (2) a series of disks of constant thickness and different H concentrations, (3) a series of disks of constant H concentration and different thicknesses, and (4) a series of spheres of constant hydrogen concentration and different diameters. CNPGAA sensitivities as a function of target size and hydrogen content are compared with TNPGAA element sensitivities for the same targets.

Experimental

Three sets of liquid samples were prepared in mixtures of H_2O and D_2O . The first set contained H_3BO_3 , the second CdCl₂ and KCl, and the third, KBr. Two stock solutions, one H_2O and one D_2O , were prepared for each set of liquid targets. Individual 400 µl portions ranging in H concentration from approximately 0.1 to 11.19% H were prepared by pipetting appropriate amounts of each of these two stock solutions into Teflon FEP bags which were then sealed. Compositions of the six stock solutions are given in Table 1. To minimize evaporative losses, samples were stored frozen until required and were allowed to thaw to room temperature prior to analysis. For each of the three sets, seven or eight different H concentrations were used and as many as ten individual portions.

Disks of constant thickness with different H concentrations were prepared from mixtures of urea, graphite and KBr. Eleven different mixtures were prepared by combining various combinations of the three substances in a Spex 800 Mixer Mill.*

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.

Hydrogen concentrations in the mixtures ranged from 0% (graphite and KBr) to 5.88% (urea and KBr), while KBr concentration remained relatively constant $(11.75 \pm 0.09\%)$). Disks with thicknesses of 1 mm and 5 mm were prepared from each of the eleven mixtures, and 3 mm thick disks were prepared from six of the urea/graphite/KBr mixtures.

A series of disks of constant diameter (12.7 mm) and different thicknesses were prepared from each of the following: *tris*-hydroxymethyl aminomethane (9.15% H),

Table 1 Compositions of stock solutions								
Solution	Element	Compound	Stock A (H ₂ O), mg/ml	Stock B (D ₂ O), mg/ml				
1	В	H ₃ BO ₃	0.242	0.245				
2	Cd	CdCl ₂	0.104	0.99				
	Cl	KCI	11.66	11.58				
	K	KCl	11.04	11.05				
3	K	KBr	61.2	60.6				
	Br	KBr	125.1	123.9				

urea (6.71% (w/w) H), deuterourea (>98% of H replaced by D, 0.1% (w/w) H), a KBr/urea mixture (4.36% K, 8.90% Br and 5.78% H), a KBr/deuterourea mixture (4.50% K, 9.20% Br and 0.1% H), a Gd/Sm/graphite/urea mixture (0.00404% Gd, 0.00988% Sm, 6.15% (w/w) H) and a Gd/Sm/graphite/deuterourea mixture (0.00454% Gd, 0.0111% Sm, 0.1% (w/w) H). The KBr/urea and KBr/deuterourea mixtures were prepared by weighing approximately 0.8 g KBr and 5 g of urea or deuterourea into a mixing vial and agitating in the Mixer Mill. Mixtures containing Gd and Sm were prepared by first doping 5 g of spectroscopic grade graphite with microliter amounts of dilute solutions containing 1086 μ g/ml Gd and 1329 μ g/ml Sm, drying the graphite at 100 °C, then combining with 5 g of urea or deuterourea in the Mixer Mill. Disks of each material were formed using a commercial steel die and hydraulic press. For each material up to 8 disks were prepared, ranging in thickness from 1 to 8 mm. Spheres of *tris*-hydroxymethyl aminomethane were also analyzed; preparation of these spheres is described in detail elsewhere.⁴

Measurements were made at the NIST reactor at 15 MW using both the CNPGAA instrument located in the Cold Neutron Research Facility and the University of Maryland-NIST TNPGAA instrument. Both facilities have been described in detail elsewhere.^{8,9,11,12} Targets were sealed in bags of Teflon FEP and suspended between

Teflon PFA strings at an angle of 45° to the neutron beam. Gamma rays of energies up to 8 MeV were collected using detection systems consisting of a germanium detector connected to a 16K analog-to-digital converter coupled to a Nuclear Data (ND) acquisition interface module (AIM), connected in turn to an ND9900 workstation. Both of the germanium detectors provided 2.1 keV FWHM resolution for 1333 keV Co line and relative efficiency of 27% (compared to a 3"×3" NaI crystal). Irradiation and counting times were sufficient to obtain $\leq 1.5\%$ counting statistics for all elements.

Peak areas were determined using either the ND peak search program, PEAK, or one of several "SUM" codes written by R. LINDSTROM at NIST. Pulse pileup corrections were made using the ND program PILEUP. To monitor changes in neutron fluence rate throughout the course of the experiment, a Ti foil (12.7 mm diameter, 147.49 mg) was irradiated and counted at regular intervals. Results from both CNPGAA and TNPGAA were normalized to the average Ti count-rate measured with each system. Self absorption corrections were made using correction factors for disk-shaped targets irradiated at 45° to the neutron beam.⁷ Corrections were also made to account for the presence of H in the background. Neutron capture reactions in the surrounding support and shielding materials produce background radiation equivalent to approximately 1 mg H at the sample position for the TNPGAA system and to approximately 25 µg of H at the sample position for the CNPGAA system. It should be noted that background count-rates due to neutron capture in the surrounding support and shielding materials increase with the scattering power of the target for both thermal¹³ and cold neutron PGAA. Corrections to these data do not include the effects of background enhancement: the amount by which the background is enhanced is not yet well characterized for the CNPGAA system, but is very small (micrograms) in comparison with the amounts of hydrogen in the samples (milligrams).

Results

Element sensitivities as a function of hydrogen concentration

Cold and thermal neutron PGAA sensitivities for H, B, Cl, K, Br, and Cd have been measured in liquid targets ranging in H concentration from 0.1% (100% D_2O) to 11.19% (100% H_2O). Consistent with previous results,³ TNPGAA sensitivities increase linearly with increasing H concentration (see Fig. 1). For these relatively thin hydrogenous targets (approximately 2 mm), sensitivities are enhanced relative to measurements on non-hydrogenous targets. These trends are not observed for element sensitivities for B, K and Br remain constant within the uncertainties associated with counting statistics, while Cd and Cl sensitivities increase by 2–3%. CNPGAA



Fig. 1a. Cold neutron PGAA sensitivity for B in solutions of H₂O and D₂O. Sensitivities decrease with increasing H from 2% to 11% hydrogen

Fig. 1b. Thermal neutron PGAA sensitivity for B in solutions of H_2O and D_2O . Sensitivity increases linearly, y = 9.75x + 550, r = 0.986, or 1.77% enhancement per percent increase in H

sensitivities for all elements measured decrease with increasing H concentration from 2.7 to 11% H; Cd sensitivities decrease by approximately 0.9% per percent H, while the decrease for B, K and Br sensitivities is $1.6 \pm 0.1\%$ per percent H concentration (see Fig. 1).

Cold and thermal neutron PGAA sensitivities for Br and K were determined for three sets of solid urea/graphite/KBr disks of constant thickness (1,3 and 5 mm) ranging in H concentration from 0 to 5.88%. Sensitivity data are plotted as a function of optical thickness, τ (a dimensionless quantity), where $\tau = \Sigma_t \cdot t$ (macroscopic total cross section



Fig. 2a. Cold neutron PGAA sensitivities for K measured in 1 mm (O), 3 mm (Δ), and 5 mm (\Diamond) urea/graphite/KBr disks vs. optical thickness (Σ_t , t)

Fig. 2b. Thermal neutron PGAA sensitivities for K measured in 1 mm (O) 3 mm (Δ), and 5 mm (\Diamond) urea/graphite/KBr disks plotted vs. $\Sigma_t \cdot t$

multiplied by disk thickness). Values of Σ_t are estimated by

$$\Sigma_i = \Sigma N_i (\sigma_b + \sigma_a)_i,$$

where N_i is the number of atoms of element *i* per unit volume σ_b is the bound atom scattering cross section (used in place of σ_s , the scattering cross section), and σ_a is the absorption cross section, estimated to be two times the thermal value.¹⁴ At hydrogen concentrations greater than about 0.5% the values for the macroscopic total cross section are dominated by scattering by H; at lower H concentrations carbon contributes significantly to the total scattering. Sensitivity plots for K are shown in Figs 2a and 2b. Results for the series of 3 and 5 mm thick disks are similar to those for the liquids of constant size and different H content. CNPGAA sensitivities decrease with increasing H content and TNPGAA sensitivities increase with increasing H content. For targets with thicknesses of 1 mm, TNPGAA sensitivities show enhancements, as expected; however, CNPGAA sensitivities do not show the decreases observed with the targets of 3 and 5 mm thickness but show random fluctuations which are within counting statistics.

Sensitivities as a function of disk thickness

Cold and thermal neutron PGAA sensitivities have been measured for K, Br, Gd and Sm in KBr and Gd/Sm doped urea and deuterourea disks of different thicknesses. Hydrogen sensitivities were determined for all urea targets but were not determined for the deuterourea targets due to the uncertainty in the amount of H present. The concentration of H in the fresh deuterourea is not known and the material is hygroscopic. Though the KBr- and Gd/Sm-doped deuterourea targets were stored in a desiccator before counting, the H count rates for thin targets of pure and KBr-doped deuterourea were significantly higher than expected from a nominal H concentration of about 0.1%. Absorption of water or D–H exchange on the surface of the targets may explain these high count rates, with thin targets most affected because of the higher surface to volume ratio. Hydrogen count rates for all of the Gd/Sm-doped deuterourea targets were two to three times higher than expected. This may be due to retention of H₂O from the Gd and Sm solutions used to prepare the targets. The H count rates for deuterourea targets were 15 to 50 times lower than those for urea targets (6.67% H content) of similar thickness, indicating H/D ratios of 0.02 to 0.07 for the deuterourea.

Cold and thermal neutron PGAA sensitivities for Gd in these targets are plotted versus target thickness in Figs 3a and 3b, respectively. Similar results are obtained for K, Br, and Sm sensitivities in the same matrices. CNPGAA sensitivities for these elements in both the urea and deuterourea matrices decrease with increasing target thickness, with a greater slope for the more highly hydrogenous urea targets. Sensitivities determined in deuterourea matrices decrease linearly, while sensitivities for elements in urea matrices do not. Sensitivities for elements in the urea matrices, and for H in the *tris*-hydroxymethyl aminomethane decrease by as much as 40–50% (relative to the thinnest target) over the range of thicknesses measured (from about 0.6 to 8 mm), while those for elements in deuterourea targets exhibit only a 20 to 25% decrease. For the relatively thin targets (from about 0.6 to 2 mm), element sensitivities are higher in the urea matrix, whereas for targets ranging in thickness from approximately 2 to 8 mm, the deuterourea matrix yields higher sensitivity values (see Fig. 3a).

TNPGAA results are consistent with those reported previously for H, B and K sensitivities measured in disks of different thicknesses for several different matrices.³



- Fig. 3a. Cold neutron PGAA sensitivities for Gd measured in urea (●) and deuterourea (O) disks plotted vs. disk thickness
- Fig. 3b. Thermal neutron PGAA sensitivities for Gd measured in urea (•) and deuterourea (O) disks plotted vs. disk thickness



Fig. 4. Cold neutron PGAA sensitivities for H in disks (0) and spheres (O) of tris-hydroxymethyl aminomethane

Element sensitivities increase with increasing disk thickness for disks of 0.6 to 2 mm in thickness, and decrease with increasing disk thickness for disks ranging in thickness from 2 to 8 mm. Element sensitivities for deuterourea targets also show this trend, but to a lesser extent, exhibiting a 7.5% decrease in sensitivity as compared with an 16% decrease for urea (relative to the 2 mm target) over the same range of disk thickness (see fig. 3b). TNPGAA sensitivities for all elements are lower in the deuterourea targets than for the urea targets.

Sensitivities for spheres of *tris*-hydroxymethyl aminomethane were determined using CNPGAA and compared with previous results of TNPGAA.⁴ For TNPGAA, sensitivities remain constant within counting statistics for spheres ranging in diameter from about 5 mm to 12 mm. However, CNPGAA sensitivities decrease with increasing diameter. Hydrogen sensitivity for the largest sphere is 18% lower than that for the smallest (5 mm diameter) sphere. This decrease is not as sharp as the 40% sensitivity decrease observed for disks of the same material over the same range of mass (see Fig. 4).

Discussion

Cold and thermal neutron scattering by hydrogen

The trends observed for CNPGAA sensitivity as a function of H content and target thickness are quite different from those for TNPGAA. TNPGAA element sensitivities for hydrogenous disks are enhanced relative to sensitivities in a non-hydrogenous matrix, whereas for CNPGAA, sensitivities for elements in most hydrogenous disks are lower than those in nonhydrogenous disks. The exception is for the very thin, 1 mm thick, hydrogenous targets. This implies that the effects of scattering for cold neutrons in disks differ from those for thermal neutrons.

The effects of neutron scattering by H on TNPGAA element sensitivities have been studied previously.^{3,4,7} Monte Carlo simulations using a monoenergetic model of neutron scattering within a target have shown that the trends observed for TNPGAA sensitivities can be explained by elastic scattering.⁷ A scattered neutron may be absorbed within the target, scattered out of the target, or further scattered within the target. The target size, shape, and scattering power determine whether the neutron is more likely to be absorbed or lost. Previous experimental work and Monte Carlo simulations have shown that as the target thickness approaches zero, sensitivities approach the value that would be obtained in the absence of scattering. Hence, sensitivities for urea and deuterourea targets should converge as the target thicknesses approach zero. This convergence is observed in the results from both cold and thermal neutron PGAA (see Figs 3a and 3b).

Results of previous TNPGAA experiments have shown that sensitivities for hydrogenous spheres do not vary with sample size or scattering cross section.⁴ Results of Monte Carlo simulations confirm that spherical targets are less affected by elastic scattering, and agreement between simulation and experiment provides additional evidence that the observed sensitivity enhancements for TNPGAA are the result of elastic scattering. However, the use of spherical targets does not eliminate any effects of inelastic scattering. The decrease observed for CNPGAA sensitivity for H in spheres indicate that the observed effects are not due to elastic scattering.

The differences between thermal and cold neutron scattering by H on element sensitivities are likely to be the result of a combination of two effects. First, the H scattering cross-section for cold neutrons is larger than for thermal neutrons, since the scattering cross section typically increases as the neutron energy decreases. Depending on the shape of the sample, this results in a decrease in the average number of neutrons within the target. Second, cold neutron scattering within a room temperature target is likely to result in more of a change in the average energy of the neutrons, than is thermal neutron scattering within a room temperature target. The thermal neutron distribution is roughly Maxwellian such that the neutrons are in thermal equilibrium with a room temperature target, and the effects of inelastic scattering are negligible. By contrast, cold neutrons are not in thermal equilibrium with a room temperature target. Thus, the consequence of scattering is an increase in the average energy of the cold neutron beam, decreasing the effective absorption cross-sections and thereby decreasing sensitivities.

Inelastic neutron scattering may be used to explain the trends observed in the CNPGAA sensitivity plots. Except in the 1 mm disks, sensitivities for elements in targets of constant thickness decrease with increasing hydrogen concentration (Figs 1a and 2a); i.e., as the concentration of scattering nuclides is increased, the number of neutron scattering interactions also increases, further increasing the average energy of the neutron beam. Because the number of scattering events increases with target thickness, sensitivities for elements in targets of constant H concentration decrease as sample thickness increases (Fig. 3a). It should be noted, however, that sensitivities for elements in thin (< 2 mm) urea disk matrices are identical to or perhaps greater than those in deuterourea disks of the same thickness (Fig. 3a). These results indicate that the effects of inelastic neutron scattering are not significant for very thin (1 mm) disk shaped samples, possibly because there are insufficient scattering interactions for a significant increase in average neutron energy to occur.

Sensitivities for "1/v" vs. "non-1-v" elements

The hypothesis that inelastic neutron scattering is responsible for decreasing element sensitivities in CNPGAA has been tested by comparing sensitivity decreases for "1/v" nuclides with those for "non-1/v" nuclides. Those nuclides for which the neutron absorption cross section is inversely proportional to neutron velocity (1/v) should yield the same relative decrease in sensitivity since changes in neutron energy affect the absorption rates by the same proportion. However, a change in the average neutron energy has a different effect on sensitivities for elements whose absorption cross sections include low energy neutron capture resonances. Absorption cross sections for H, K, and Br vary as 1/v, whereas nuclides of Sm and Gd possess resonances at about 0.09 eV and 0.03 eV respectively. In the energy range of interest, the Sm absorption cross section changes less rapidly with energy than that of a "1/v" nuclide.

Sensitivity decreases for elements measured in KBr- and Gd/Sm-doped urea disks of constant H concentration and increasing disk thickness are given in Table 2. The

Element	Sensitivity decrease (%) per unit optical thickness				
i dement	KBr Doped Urea	Gd/Sm Doped Urea			
Н	13.9 ± 0.9	13.3 ± 0.9			
K	13.6 ± 1.2	-			
Br	13.5 ± 1.4	-			
Gd	·	14.0 ± 0.8			
Sm	_	11.9 ± 0.7			

		Table 2				
Sensitivity	decreases,	relative to the	thinnest	target,	for	elements

measured in KBr- and Gd/Sm-doped urea disks as a function

decrease per unit optical thickness (relative to the thinnest target) is determined over the linear portion of the curve (values of $\Sigma_t \cdot t$ from 0.5 to 3.0). The observed decreases for H, K, Br and Gd agree within the stated uncertainties; however, the sensitivity decrease for Sm is found to differ significantly from those for the other elements. Although ¹⁵⁷Gd possesses a broad low energy resonance, the value of the radiative capture resonance integral (I_{γ}) 700 b, is small compared with its thermal value for σ_a of 254000 b. For ¹⁴⁹Sm, however, the value for I_{γ} of 3390 b is approximately one tenth of its thermal value for σ_a of 40140 b. The difference between the sensitivity decrease observed for

Sm and those observed for the other elements studied provides additional evidence that the observed CNPGAA sensitivity decreases are due, at least in part, to a scattering-induced increase in the average energy of the neutrons.

Conclusions

Element sensitivities in TNPGAA for hydrogenous disks that are thinner than they are wide are enhanced relative to non-hydrogenous targets. By contrast, CNPGAA element sensitivities decrease relative to non-hydrogenous targets, except for very thin disks. Although the observed sensitivity changes of CNPGAA are certainly the result of neutron scattering by H, the effects of scattering on CNPGAA sensitivities are quite different from those observed in TNPGAA.

It has been previously shown that sensitivity enhancements in thermal neutron PGAA can be quantitatively accounted for by a theoretical model of elastic neutron scattering.⁷ Results of the present study indicate that the CNPGAA sensitivity decreases that are observed with increasing hydrogen content and target thickness are due, at least in part, to an increase in the average energy of the neutrons. This has been demonstrated by comparison of sensitivities for 1/v and non-1/v nuclides as a function of scattering power of the matrix. Elements whose neutron absorption cross sections vary as 1/v show the same relative decreases with increasing target thickness and increasing H content, while sensitivities for Sm, an element with a non-1/v absorption cross section, show smaller decreases. Additional evidence is provided from the analysis of hydrogenous spheres. The use of spheres should nearly eliminate any effects of elastic scattering. However, CNPGAA sensitivities decrease with increasing sphere diameter indicating that elastic scattering is not entirely responsible for the observed trends.

Results of this investigation indicate that an understanding of the problems associated with neutron scattering is necessary to ensure that data from analyses of hydrogenous matrices performed using CNPGAA are accurately interpreted. Element sensitivities should be calibrated as a function of target geometry and neutron scattering power. The effect of neutron scattering by elements other than hydrogen (e.g., C) also should be investigated. Results of preliminary work indicate that the effects of inelastic neutron scattering may be reduced by controlling the target temperature. These preliminary data show that the effects of elastic scattering of thermal neutrons scattering within a thermal target also occur with cold neutrons scattering within a cold target.

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