FINAL REPORT

(Covering second year of a 3-year grant,* one no cost extension, and additional research to meet project goals)

DOE/ER/75878--T1

STUDY OF NEUTRON FOCUSING AT THE TEXAS COLD NEUTRON SOURCE

DE FG03-93ER75878

Bernard W. Wehring and Kenan Ünlü

Nuclear Engineering Teaching Laboratory

The University of Texas at Austin

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DISCLAIMER

I. INTRODUCTION AND EXECUTIVE SUMMARY

Funds were received from DOE for two years of a three-year DOE Nuclear Engineering Research Grant, "Study of Neutron Focusing at the Texas Cold Neutron Source." The goals of this three-year study were to do the following:

1. design a neutron focusing system for use with the Texas Cold Neutron Source (TCNS) to produce an intense beam of cold neutrons appropriate for prompt gamma activation analysis (PGAA),
2. orchestrate the construction of the focusing system, integrate it into the TCNS neutron guide complex, and measure its performance,
3. design, setup, and test a cold-neutron PGAA system which utilizes the guided focused cold neutron beam.

The first year of the 3-year grant was funded under DE FG02-92ER75711. The second year was funded under DE FG03-93ER75878. This was done because DOE moved the administration of the grant from the Chicago Field Office to the San Francisco Field Office (now the Oakland Operations Office). The third year was not funded at all because the Department of Energy Nuclear Engineering Education Research Program was terminated. Congress was not willing to continue to appropriate funds for this program unless DOE put it in their budget, and DOE was not willing to do so. Since DOE would not transfer equipment money from one field office to another, I was given two no-cost extensions under the first year grant number and a one-year no-cost extension under the second year grant number to carry equipment money over for the purchase of one big item the design of which was the focus of the research.

During the first year of the DOE grant, a new procedure was developed and used to design a focusing converging guide consisting of truncated rectangular cone sections. Detailed calculations were performed using a 3-D Monte Carlo code which we wrote to trace neutrons through the curved guide of the TCNS into the proposed converging guide. Using realistic reflectivities for Ni-Ti supermirrors, we obtained gains of 3 to 5 for 4 different converging guide geometries.

During the second year of the DOE grant, the subject of this final report, Ovonic Synthetic Materials Company was contracted to build a converging neutron guide focusing system to our specifications. Considerable time and effort were spent working with Ovonics on selecting the materials for the converging neutron guide system. The major portion of the research on the design of a cold-neutron prompt gamma activation analysis (PGAA) system was also completed during the second year.

At the beginning of the third year of the grant, a converging neutron guide focusing system had been ordered, and a cold-neutron PGAA system had been designed. Since DOE did not fund the third year, there was no money to purchase the required equipment for the cold-neutron PGAA system and no money to perform tests of either the converging neutron guide or the cold-neutron PGAA system. The research already accomplished would have little value without testing the systems which we had designed. Thus we continued the project at a pace we could sustain with internal funding.

Over the last 2 1/2 years, the converging neutron guide was delivered, installed, and tested. The equipment for the cold-neutron PGAA system was purchased, and the cold-neutron PGAA system was installed. Recently, the initial tests of the cold-neutron PGAA system were completed. The results of these measurements using the converging neutron guide and the cold-neutron PGAA system established that our research was an unqualified success. The sensitivities and detection limits obtained by us demonstrate that our PGAA system is competitive with the National Institute of Standards and Technology (NIST) cold-neutron PGAA system at the NIST 20-MW reactor.

Two graduate students were supported by the DOE grant. During the first year both students were admitted to candidacy for the doctoral degree at The University of Texas at Austin. Since that time, both students have graduated with Ph.D. degrees.

II. BACKGROUND

A. Texas Cold Neutron Source And Curved Neutron Guide

The low energy tail of any thermal (Maxwell-Boltzmann) neutron distribution consists of cold neutrons, but only as a small fraction of the distribution. The intensity of the cold neutrons can be increased by moderating the thermal neutrons in a material held at cryogenic temperatures. The speed distribution of the cold neutrons coming from the moderator has been approximated by a modified Maxwell-Boltzmann distribution, with a neutron temperature somewhat higher than the temperature of the cold moderator material. [(1)]

A cold neutron source was constructed and is operated by the Nuclear Engineering Teaching Laboratory (NETL) at The University of Texas at Austin (UT).[(2-6)] The Texas Cold Neutron Source (TCNS) was installed in a radial beam port which pierces the graphite reflector of the NETL 1-MW TRIGA reactor. An organic compound mesitylene (1,3,5-trimethylbenzene) is used as the moderating material and is contained in an aluminum right circular cylindrical chamber (7.6-cm diam x 2-cm thick), located about 18 cm from the nearest fuel element. Lead, 7.6-cm thick, shields this chamber from core gamma rays. The mesitylene is cooled by a 3-m long (1.9-cm o.d.) neon thermosyphon with a slope of 2.5°. Gaseous neon is fed to the thermosyphon from a room temperature 12-lt reservoir through a small tube. Cooling is provided by a 2-stage helium expansion cryorefrigerator, whose "cold head" is attached to the upper end of the thermosyphon. This connection is contained in a vacuum box outside the biological shield of the UT-TRIGA reactor. All cryogenic components are held under vacuum (3×10^{-6} torr when the moderator is cooled) to avoid convective heating.

The TCNS was constructed with two horizontal neutron channels (8.9 x 4.5 cm) to extract cold neutrons coming from the moderator chamber. One of them, aligned with the center line of the beam port, has been left empty to have a straight view of the moderator chamber. The "empty channel" is now blocked and is not used. The other channel runs divergent and exits the biological shield 11.5 cm away from the beam port center line. It contains an inner 2-m-long neutron guide section. The neutron guide is continued with two 2-m-long sections outside the beam port. The locations of the Texas Cold Neutron Source and the three 2-m long neutron guide sections are shown in Fig. 1.

All neutron guide sections are curved to a 300-m radius and divided into three vertical channels (5 x 0.45 cm) by 0.1-cm-thick walls. This array provides blocking of the straight-path background components streaming through the guide. The curved neutron guide, with all reflecting surfaces coated by a 1000-Å ^{58}Ni layer, utilizes total reflection to transport neutrons without the normal $1/r^2$ intensity loss. The critical angle for total reflection of neutrons from ^{58}Ni is 0.12° per Å. The characteristic wavelength of the curved neutron guide is 2.7 Å, which corresponds to a neutron energy of 11 meV. Each section of the curved neutron guide is evacuated during operation of the TCNS, the two external sections by an oil free diaphragm vacuum pump.

The two external neutron guide sections are mounted on optical translation stages on top of optical laboratory jacks which are fixed to an aluminum "I" beam. After the curved guides were geometrically aligned, a fine-tune adjustment was achieved by neutron intensity measurements using a ^3He detector. These measurements were performed at low reactor power, adjusting the front and back end positions of each section. Neutron measurements were made for two cases, one with the moderator chamber empty and one with the chamber filled with mesitylene at about 30 K.

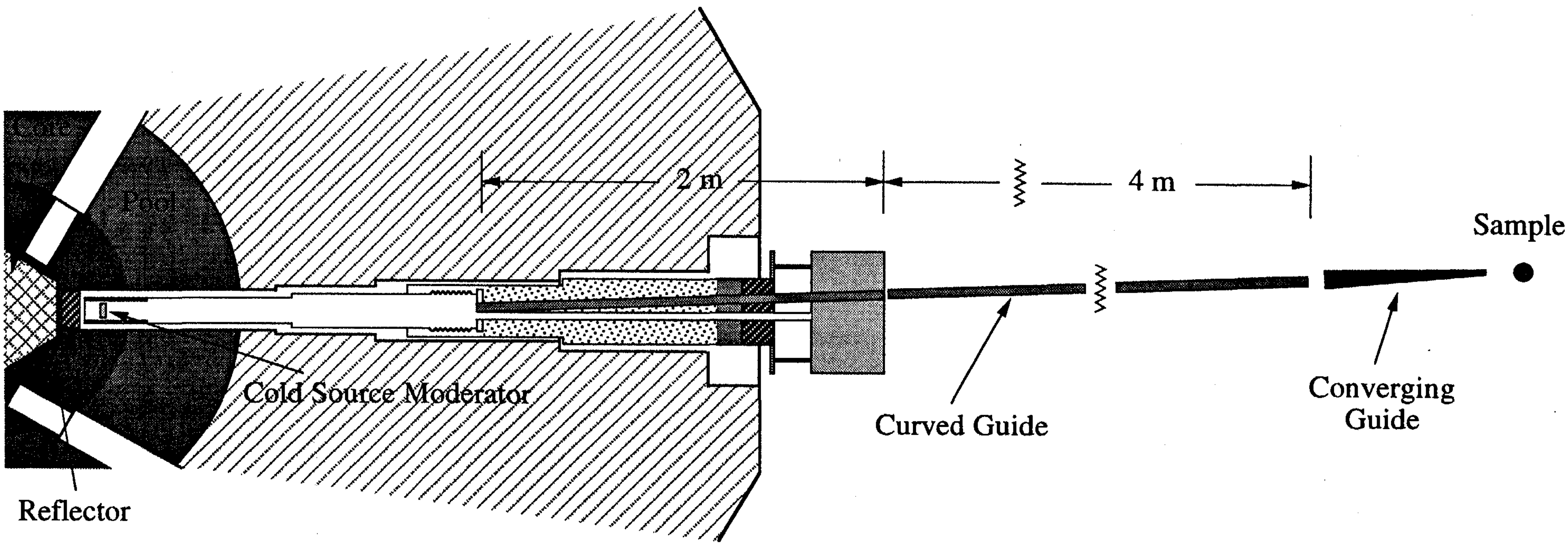

Fig. 1. Cross sectional view of the Texas Cold Neutron Source in the piercing beam port of the UT-TRIGA research reactor showing the location of the 6-m long curved neutron guide and the 80-cm long converging neutron guide.

B. Neutron Focusing (First Year Grant)

After researching all the methods for neutron focusing, we decided to design a converging neutron guide for use as a focusing system. Furthermore, we chose to investigate multisection converging guides where each section is a truncated rectangular cone. The design procedure developed was to optimize the neutron flux at the prompt gamma activation analysis (PGAA) sample point of the converging guide by varying the two slant angles for each truncated rectangular cone section as the sections were added one at a time. The resulting set of slant angles is a good approximation to the set that would be obtained using a global optimization since only a small fraction of the neutrons suffer more than one reflection in the converging guide.

Several analytical calculations for neutron guides were found in the literature, but these calculations were done in two dimensions (2 D) with a neutron source that is spatially uniform and isotropic in 2 D (i.e., dn/dø constant where ø is the polar angle in 2 dimensions). The 2-D treatments could not be used for designing the converging guide because neutron reflections from all four walls (top, bottom, and two sides) of the converging guide are important. Also, the spatial distribution of neutrons entering the converging guide from the curved guide is not uniform, and the angular distribution is not 2-D isotropic. Thus, we wrote our own general 3-D Monte Carlo code to transport neutrons through neutron guides with rectangular cross sections.

In order to achieve an increase in neutron flux, the critical angle for neutron reflection from the surfaces of the converging guide must be greater than the critical angle for ^{58}Ni. This dictates coating the converging guide reflecting surfaces with NiC-Ti supermirror layers having an effective critical angle of 0.3° for 1-Å neutrons. (7) Recent advances in supermirror technology have produced reflectivities greater than 95% for angles of neutron reflection up to this effective critical angle. (8)

A total of eight different converging guide systems were designed and analyzed using our 3-D Monte Carlo neutron-guide code.[9, 10] Four different geometries were considered, and two different neutron sources were used. A generic source was taken to be a uniform isotropic (3 D) source of 4-Å neutrons located at the entrance of the curved guide. A specific source, "the TCNS source," was generated having the energy dependence calculated for neutrons leaving the moderator chamber in the general direction of the guide and biased by calculated angular and spatial distributions.

A more detailed description of the research done during the first year grant is given in "Final Report, A Study of Neutron Focusing at the Texas Cold Neutron Source" -- DE-FG02-92ER75711. Two no-cost extensions were given by DOE under the first year grant to carry equipment money over to cover a portion of the cost of the focusing system.

III. CONVERGING NEUTRON GUIDE (SECOND YEAR GRANT AND BEYOND)

Based on our calculated performance results and financial considerations, the four-section 80-cm long converging guide focusing system was selected for use with the TCNS. Ovonic Synthetic Materials Company,[11] Troy, Michigan, the only company in the U.S. capable of doing NiC-Ti supermirrors coating, was contracted to build the converging neutron guide focusing system to our design specifications. Supermirror layers of NiC-Ti were deposited by Ovonics on 20-cm-long single crystals of silicon. These plates (a total of 16) were glued together in the correct orientation to form the converging guide. Figure 2 shows the Si plates drawn to scale where the axial dimension has been compressed a factor of 5 compared to the transverse dimensions. The curved-guide cross section is shown for comparison. The assembly is gas tight with entrance and exit windows made of high purity aluminum to allow helium to flow through the assembly displacing air in the converging guide.

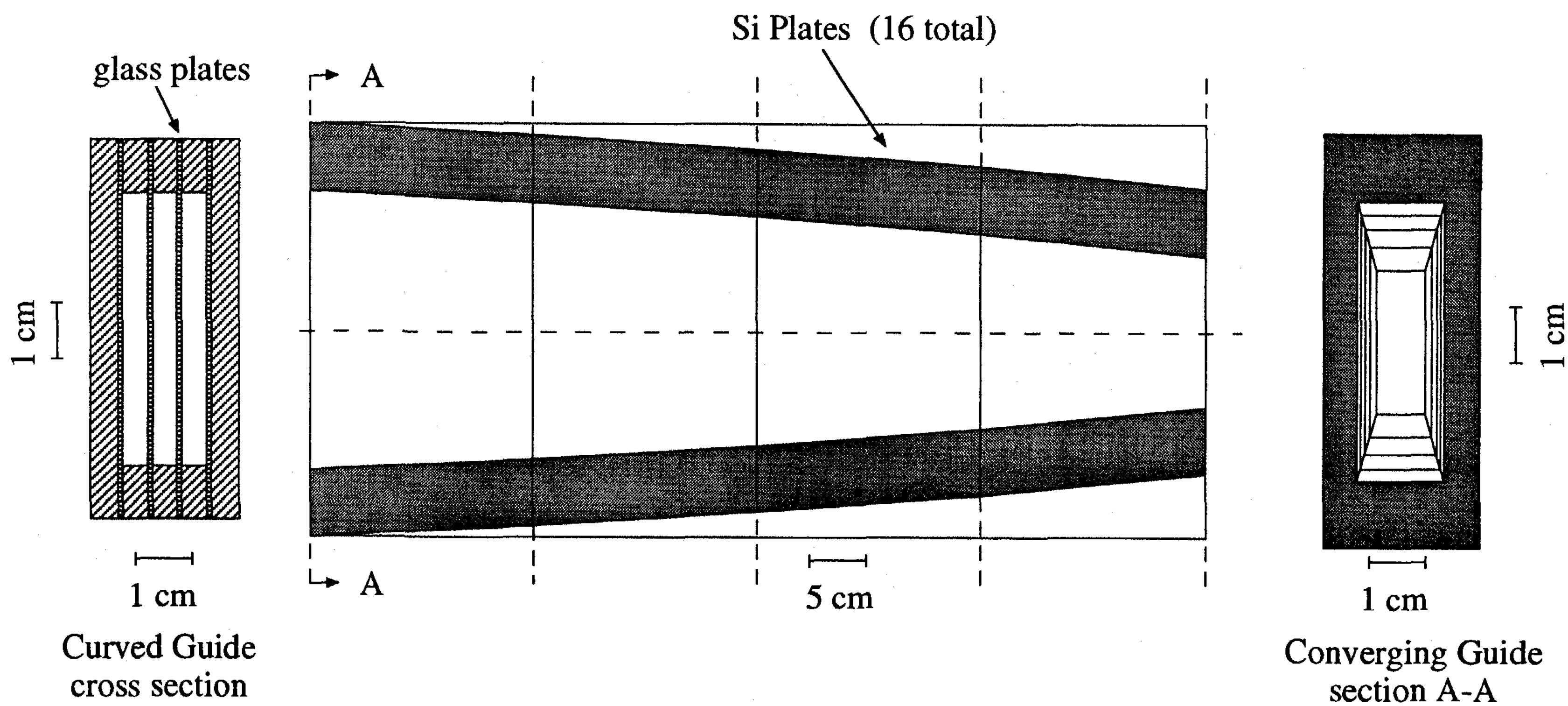

Fig. 2. The converging neutron guide (center and right panels). The NiC-Ti supermirror layers coat all reflecting surfaces of the 16 Si single-crystal plates. A cross section of the curved neutron guide is shown for comparison.

The converging neutron guide was mounted directly behind the third 2-m-long curved neutron guide section. Alignment screws (total of 10) provided by the manufacturer of the converging guide allowed setting the x-y positions of the front and back ends of the unit as a whole. The correct settings were determined by using a Thomson tube neutron radiography imaging system. A radiograph of the neutron beam at the PGAA sample position (24 cm from the exit of the converging guide) is shown in Fig. 3. along with the derived x and y intensity profile slices.

The neutron flux at the PGAA sample position was measured at low reactor power using a ^3He neutron detector that was nearly 100% efficient for detecting subthermal neutrons. The ^3He detector was covered by cadmium except for a 3 x 10 mm slit opening parallel to the axis of the detector. The results were extrapolated to 1-MW reactor power yielding the following values: $(9 \pm 2) \times 10^6$ n/cm^{2}s with no moderator in the chamber ("empty chamber operation") and $(1.5 \pm 0.2) \times 10^7$ n/cm^{2}s with mesitylene cooled to 30 K in the chamber ("cold moderator operation"). For the case of empty chamber operation, the curved neutron guide selects neutrons on the low-speed side of the room temperature neutron distribution. Using a calculated distribution for these neutrons gave a thermal equivalent flux for "empty chamber operation" of $(1.5 \pm 0.6) \times 10^7$ n/cm^{2}s. For the "cold moderator operation," the curved guide selects neutrons having an average speed of about 800 m/s yielding a thermal equivalent flux of $(4.6 \pm 0.7) \times 10^7$ n/cm^{2}s.

The gain of the converging neutron guide determined from the measured fluxes is 2.7 ± 0.2. This gain is the ratio of the neutron flux average over the 3 x 10 mm area at a distance of 24 cm from the exit of the converging guide (PGAA sample point) to the neutron flux average over the exit of the curved neutron guide. The gain calculated for a 1 cm^2 area at the sample point was 3.0. This was the quantity that was optimized in our design procedure.

Fig. 3.b. Radiograph of neutron beam at PGAA sample position showing y intensity profile.

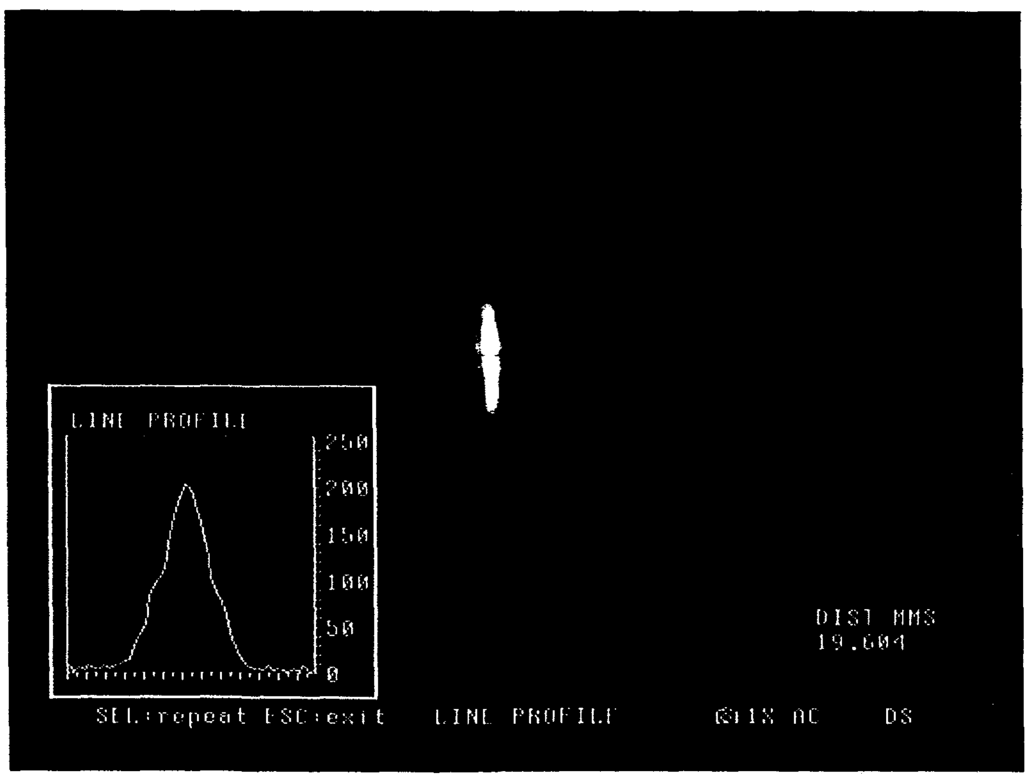

Fig. 3.a. Radiograph of neutron beam at PGAA sample position showing x intensity profile.

IV. COLD-NEUTRON PROMPT GAMMA ACTIVATION ANALYSIS SYSTEM

Prompt gamma activation analysis (PGAA) is based on detection of capture gamma rays emitted by target material while being irradiated by neutrons. All elements capture neutrons and nearly every neutron capture produces gamma rays that are potentially suitable for determining the amount of the capturing element. The probability of neutron capture, however, varies widely from isotope to isotope, and therefore analytical sensitivities (cps/mg) and detection limits (mg) do so also.

When reactor thermal neutron beams are utilized for neutron irradiation in PGAA, the high gamma-ray background at the sample limits the usefulness of this technique. Neutron beam filters help, but also reduce neutron intensity along with reducing gamma-ray background. Employing guided cold neutron beams, however, greatly reduces the gamma-ray background at the PGAA sample while maintaining a comparable neutron capture rate.(12) This is done by transporting cold neutrons far from the reactor in a straight neutron guide without significant loss of neutron intensity. For shorter distances, curved neutron guides attenuate line-of-sight background radiation while allowing the cold neutrons to pass through by total reflection from the guide walls.

A cold-neutron prompt gamma activation analysis (PGAA) system was designed taking into account the advantage of the low background provided by the TCNS and curved guide systems. (13-16) The following criteria were used in the design:

1. The structure and shielding materials for the cold-neutron PGAA facility were chosen to minimize the background contributions for elements to be detected in the samples to be studied. Accordingly, the use of materials containing hydrogen or boron was minimized.

2. The sample handling system was designed to be versatile to permit the study of a wide range of samples with quick and reproducible sample positioning and a minimum of material close to the sample.

3. The detection system was chosen to cover the wide range of capture gamma-ray energies (0.1 to 12 MeV) with good resolution over the entire energy range.

The cold-neutron PGAA system consists of a sample chamber, a gamma-ray spectrometer with data acquisition and processing electronics, detector shielding, and a beam stop. During operation, the TCNS focused cold-neutron beam irradiates the sample inside the sample chamber. This chamber consists of a 14.5 x 14.5 cm aluminum base plate with four 3-mm-diameter 16-cm-long Al rods attached to the corners of the base plate. The rods are held together at the top with an aluminum frame, to support a 5-mil thick fluoro-ethylene-propylene (FEP) bag. An aluminum sample frame is secured to the base plate and supports a web of FEP tread to hold the sample. The FEP bag is held to the base plate with an o-ring pressed by an adjustable aluminum frame. The base plate is provided with two tubes for purging the sample chamber with helium during operation, to eliminate the background due to neutron captures in air.

The cold-neutron PGAA gamma-ray spectrometer is a 25% efficient high-purity n-type germanium detector (GMX-25190-s ORTEC) in the DUET configuration with an offset port dewar (30 liters). This detector was selected in order to incorporate a Compton suppression system at a later date. Detector shielding is provided by a ^{6}LiF/poly (PNPI USA Corp., Houston, Texas) collar inside a close pack lead cave. The detector sees the sample through a 6.5-cm diameter opening in the lead cave, shielded with a ^{6}LiF/poly disk. For the current arrangement, the sample - detector distance is 26.5 cm. This distance can be reduced with a different lead cave configuration. Layers of ^{6}LiF/poly line the front face of the lead cave and the lateral sides of the sample chamber. The gadolinium converter plate of a neutron imaging system is used as a beam stop. A top view of the TCNS experimental facility containing the UT-PGAA system is shown in Fig. 4. Figure 5 is a photograph of the UT-PGAA system.

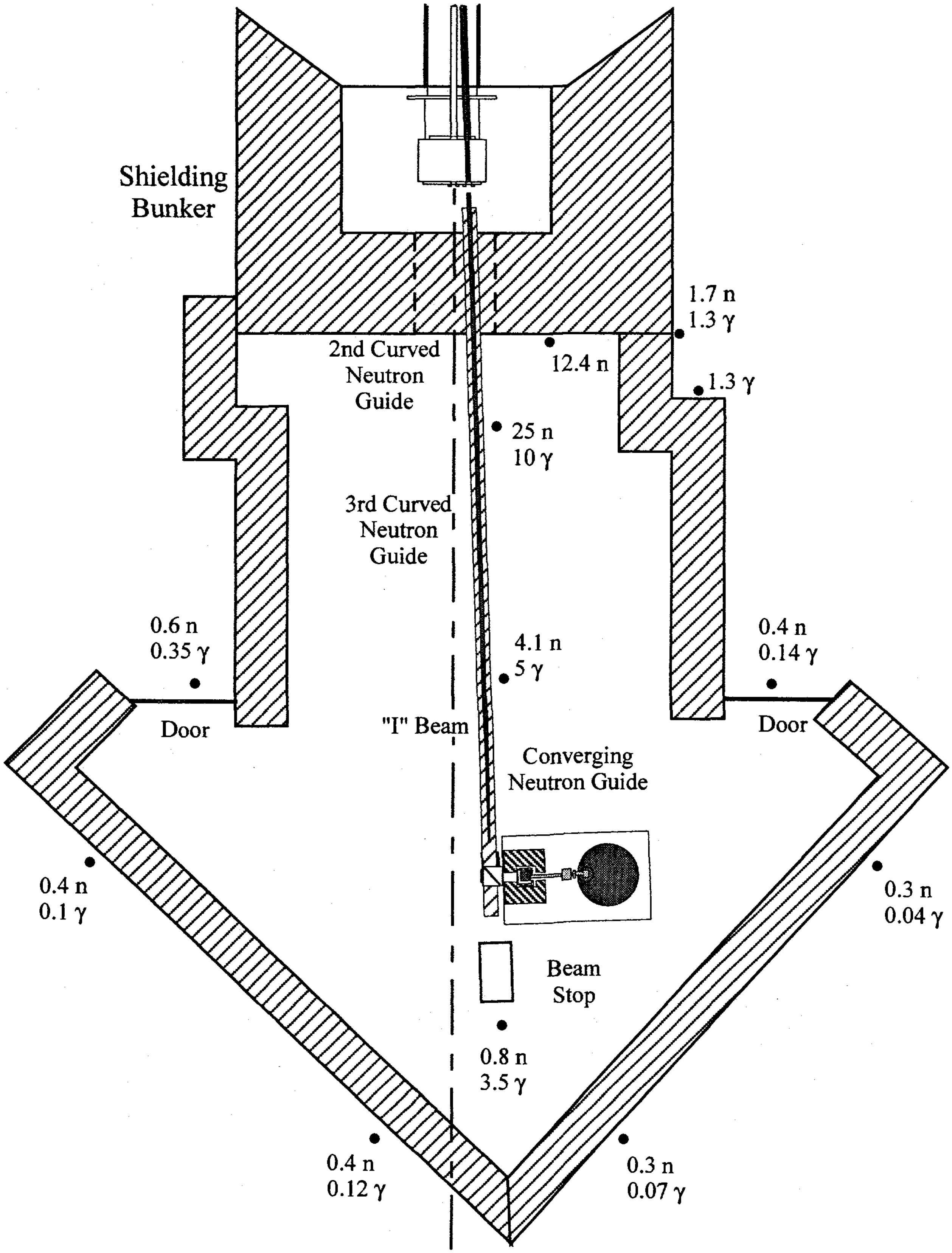

Fig. 4. Top view of the Texas Cold Neutron Source experimental facility. The curved neutron guide, the converging neutron guide, and the prompt gamma detection system are enclosed by a concrete shielding wall. The concrete bunker (top) shields radiation scattered by that portion of the curved neutron guide just outside the biological shield of the reactor. Line-of-sight radiation can not travel past this spot in the curved guide without intersecting a wall. Also shown are measured dose rates in mrem/hr.

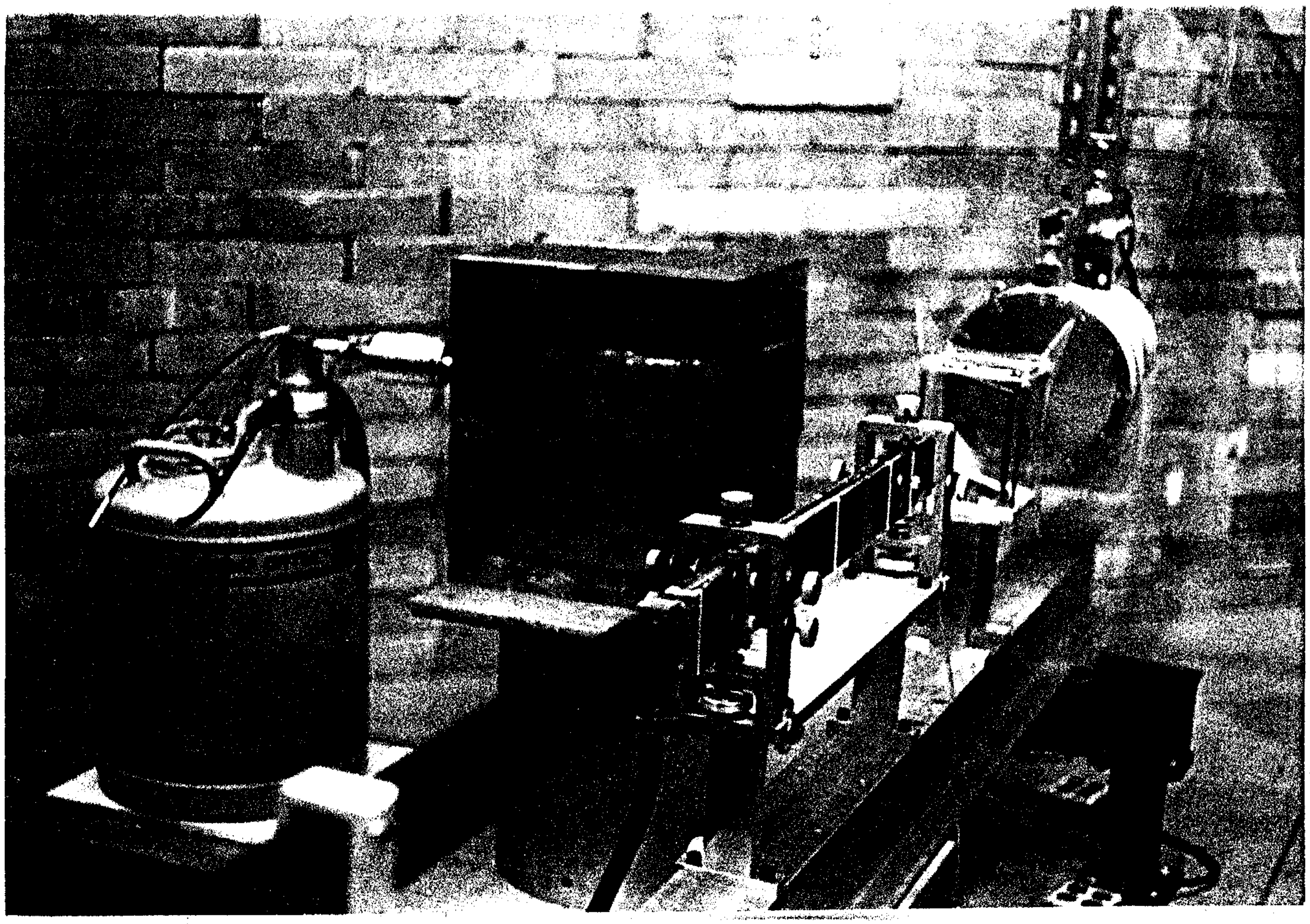

Fig. 5. Photograph of the PGAA system showing the converging neutron guide and a portion of the curved neutron guide (lower center), gamma-ray detector in lead cave (left center), neutron imaging tube (far right) and the FEP chamber (between converging guide and imaging tube). The PNPI (^{6}Li Poly) neutron shielding has not been installed.

The PGAA system was tested using the "empty chamber" TCNS operating mode with the close pack lead cave and a distance of 25.6 cm between the detector and the sample. The sample was a 660 nm thick borophosphosilicate glass coating on a silicon wafer positioned at 45° to the neutron beam axis. For this sample, the size of the beam, ~ 1 x 2.5 cm, determined the amount of sample material. A segment of one of the measured gamma-ray spectra is shown in Fig. 6. The resulting analytical sensitivities and calculated detection limits for boron and silicon are given in Table I. Four hours was used for "time" in the detection limit calculation.

Also shown in Table I are estimated sensitivities and detection limits for small samples where the size of the sample is smaller than the area of the neutron beam. Since the flux is not uniform across the area of the neutron beam, but peaks at the center of the beam, (Fig. 3) the "small sample" estimated values of sensitivity are larger and the detection limits are smaller. Using the measured fluxes, the performance for the "cold moderator" operating mode with an optimized system was also estimated and is given in Table I.

IV. STUDENT INVOLVEMENT

Dr. Jong-Youl Kim, supported by this DOE grant, was admitted to candidacy for the doctoral degree at The University of Texas at Austin during the first-year reporting period. The subject of his dissertation is the design and analysis of a focusing converging guide to be used with the TCNS. Dr. Greg Downing, a scientist at the National Institute of Standards and Technology, was a member of Dr. Kim's Ph.D. committee. Dr. Kim graduated in December 1993.

Dr. Carlos Rios-Martinez, supported by this DOE grant, was also admitted to candidacy for the doctoral degree during the first-year reporting period. The subject of his dissertation is the design, construction, and use of a PGAA facility using focused cold neutrons. Dr. Richard Lindstrom, a scientist at the National Institute of Standards

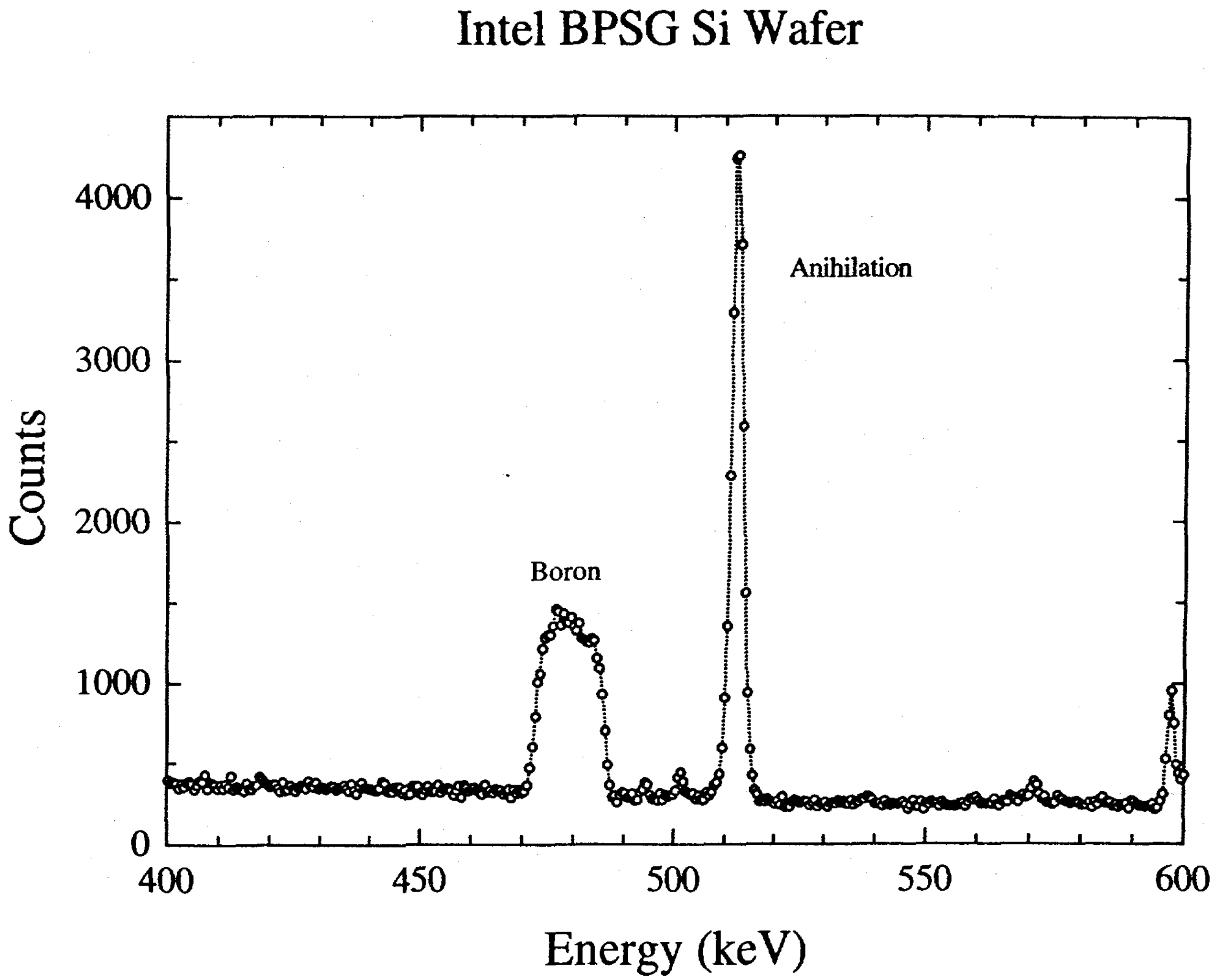

Fig. 6. A segment of the gamma-ray spectrum showing the boron-10 peak for a 3.8 μg sample. The results were obtained by the present, nonoptimized PGAA system with no cold moderator in the TCNS (empty chamber). In this situation, the curved neutron guide selects subthermal neutrons with an average speed of about 1500 m/s.

Table I. Performance of the cold-neutron PGAA

Boron Target Material	Sensitivity (cps/mg)	Detection Limit [a] (ng)
No cold moderator - present system		
Large sample (> 1.0 x 2.5 cm)	187	184
Small sample (0.5 x 0.5 cm)	470 [b]	74 [b]
Cold moderator - optimized system		
Large sample (> 1.0 x 2.5 cm)	600 [b]	57 [b]
Small sample (0.5 x 0.5 cm)	1,500 [b]	23 [b]
Silicon Target Material	**Sensitivity (cps/g)**	**Detection Limit [a] (mg)**
No cold moderator - present system		
Large sample (> 1.0 x 2.5 cm)	1.85	4.8
Small sample (0.5 x 0.5 cm)	4.6 [b]	1.9 [b]
Cold moderator - optimized system		
Large sample (> 1.0 x 2.5 cm)	5.9 [b]	1.5 [b]
Small sample (0.5 x 0.5 cm)	15 [b]	0.6 [b]

[a] $m = 3.29\sqrt{\frac{\text{Bkg Counting Rate}}{\text{Time}}} \Big/ \text{Sensitivity}$

[b] estimated values

and Technology, was a member of Dr. Rios-Martinez's Ph.D. committee. Dr. Rios-Martinez graduated in May 1995.

V. CONCLUSION

Through the use of cold neutrons, a curved neutron guide, and neutron focusing, the cold-neutron PGAA facility at The University of Texas at Austin has greatly improved detection sensitivities and lower detection limits, comparable to Instrumental Neutron Activation Analysis, allowing PGAA to be applied to many technical problems involving analytical chemistry. Proposed applications for the cold-neutron PGAA include: (a) determination of boron and hydrogen impurity levels in semiconductors materials; (b) determination of gadolinium concentrations in samples used for neutron capture therapy studies; (c) multielemental analysis of geological, archaeological, and environmental samples for determinations of major elements aluminum, sulfur, potassium, calcium, titanium, and iron, and minor elements hydrogen, boron, chlorine, vanadium, manganese, cobalt, cadmium, neodymium, samarium, and gadolinium; and (d) multielemental analysis of biological samples for the major and minor elements hydrogen, sodium, phosphorous, sulfur, and potassium, and trace elements boron and cadmium.

Funding from DOE allowed us to complete the first application of the Texas Cold Neutron Source (TCNS). Both the TCNS and the cold-neutron PGAA system are truely unique and inovative facilities. The TCNS is one of three reactor-based cold neutron sources with neutron guides in the U.S., and the cold-neutron PGAA system is one of two such facilities in the U.S. Some of the publications resulting from this research are given in the list of reference, viz., 9, 10, and 13-16.

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