
QUARTERLY REVIEW

FALL 1993

MICHIGAN MEMORIAL PHOENIX PROJECT
THE UNIVERSITY OF MICHIGAN
NUCLEAR REACTOR LABORATORY
FORD NUCLEAR REACTOR
PHOENIX MEMORIAL LABORATORY



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Sixth Year, No. 18

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The Nuclear Reactor Laboratory Quarterly Review is published and distributed to University of Michigan faculty and staff members to inform them of the unique research capabilities of the Nuclear Reactor Laboratory and to make them aware of the types of research in progress.

The Michigan Memorial Phoenix Project was founded on May 1, 1948, as a memorial to the 585 University of Michigan alumni, students, faculty, and staff members who died in World War II. The Project's charter is to explore ways and means by which atomic energy can be a beneficent influence in the life of man. Research support and services provided by the Nuclear Reactor Laboratory and a research grant program are the means by which the Project fulfills its charter.

NEW RESEARCH

FORD NUCLEAR REACTOR

University of Michigan

Chemistry

Suzanne Argentine, a doctoral candidate in the Chemistry Department, irradiated neodymium in a toluene solution with the objective of conducting optical spectroscopic studies and EPR studies of metal-centered fullerenes. Ms. Argentine's advisor is Professor A. H. Francis, and the title of her thesis is Spectroscopy of the Fullerenes.

Nuclear Engineering

Jeff Martin, a doctoral candidate whose advisor is Professor David K. Wehe, utilized the reactor to produce a number of medium energy gamma ray sources for his research work. 200 to 500 microcurie amounts of sodium-24 from sodium chloride and antimony-124 from pure metallic antimony were produced. 1,369 keV, 1,691 keV and 2,754 keV gamma rays from the radioactive sodium and antimony are used to measure the resolution, efficiency and point-spread-function of gamma ray imaging in a Compton gamma camera. Mr. Martin also produced sodium-24

by an alternate method, a neutron-alpha reaction with aluminum-27. He irradiated pure molybdenum metal to produce molybdenum-99 with its 1,405 keV gamma ray and pure zinc metal to produce zinc-65 with its 1,115 keV gamma. The title of Mr. Martin's thesis is Ring Compton Camera for Imaging Medium Energy Gamma Rays.

Ayman Hawari irradiated small quantities of rhodium to produce approximately 300 microcuries of radioactive rhodium-104. His objective was to investigate the decay scheme of rhodium-104 as part of his PhD work. He further irradiated an iron-cobalt alloy to measure the neutron capture cross section of iron with respect to cobalt. Five microcuries of both iron and cobalt were produced. Mr. Hawari's advisor is Professor Ronald F. Fleming.

Ram Venkataraman, a doctoral candidate working for Professor Ronald F. Fleming, irradiated an ingot of indium in a special spent fuel storage rack designed as a gamma irradiation facility. His objective is to measure the production of gamma ray induced radioactivity by the reaction, In-115 (gamma, gamma') In-115m.

Physics

Doctor Scott L. Nutter and Professor Gregory Tarle are irradiating integrated circuit chips planned for use in the Superconducting Super Collider. Several irradiations are being conducted with neutron doses ranging from 1×10^{12} to 1×10^{13} n/cm² to determine radiation resistance.

Russell B. Makidon irradiated small quantities of nickel to measure the activity build-up of positron emitting cobalt-58 with time. The work is related to the Physics Department's interest in positron research. Mr. Makidon was an undergraduate working for Professor David Gidley. His BS thesis title was Measurement of Flux Curve and Activity Build-up of Cobalt-58.

New Mexico Institute of Mining and Technology

Professor Matthew Heizler, Department of Geology, irradiates geological samples in the reactor for $^{40}\text{Ar}/^{39}\text{Ar}$ age dating of rocks. The reactor is utilized to convert some of the ^{40}K to ^{39}Ar by an (n,p) reaction. Samples are returned to Professor Heizler for argon content measurement by vacuum or laser extraction and mass spectrometry.

Open University, Great Britain

Geological samples were irradiated for $^{40}\text{Ar}/^{39}\text{Ar}$ age dating by Professor Simon Kelley of the Department of Earth Sciences. The Open University was one of the first users in the Earth Sciences community from Europe to use the Ford Nuclear Reactor; they are now receiving enquiries from other workers in both the UK and in Europe as to the availability of the reactor. The Open University extracts the argon from the geological samples with a laser microprobe run by Professor Kelley and two post-doctoral researchers. Collaborative projects are currently in progress with other universities in the UK, Turkey, and Australia. Three post-doctoral researchers, six academics, and four graduate students have either analyzed samples at the Open University or used data generated by the laser microprobe facility.

University of Manchester

Geological samples were irradiated for $^{40}\text{Ar}/^{39}\text{Ar}$ age dating by Professor John M. Saxton of the Geology Department.

Duke Power

Neutron attenuation measurements were conducted on a large number of zirconium diboride (ZrB_2) coupons. The objective of the measurements was to utilize the attenuation measurements to accurately determine the boron-10 loading, called the areal density, in the coupons. These coupons will be used as calibration standards for neutron radiographs of borated aluminum sheets that will be utilized in nuclear power reactor spent fuel storage racks. The measurements were performed at the facility's A beamport.

NEUTRON ACTIVATION ANALYSIS

University of Michigan

Geological Sciences

Gerald Dickens, a doctoral candidate in marine geochemistry is investigating manganese fluctuations in deep sea sediments in order to better understand the origin of manganese deposits. Manganese is an important industrial element principally used in steel alloys. The primary supply of the world's manganese currently comes from large stratiforms of manganese ores. A future economic source also may be oceanic manganese crusts. A series of recent studies concerning the accumulation of either of

these deposits through time has increasingly led to the concept of "oxygen minimum zone manganese redirection." Simply stated, sources of deep sea reducible manganese dissolve upon entering oxygen minimum zones (OMZ) and are subsequently redirected by advective/diffusive processes to become concentrated in distal, more oxygenated environments. If correct, this concept has fundamental ramifications towards both economic geology and paleoceanography (i.e., the marine deposition of manganese should be governed by and related to the evolution of oceanic OMZs). A critical piece of supporting evidence for this important theory has been lacking: documentation that marine sediments deposited at a time coincident with, but away from, stratiform manganese ores and manganese crusts are dramatically depleted in reducible manganese. Mr. Dickens is using neutron activation analysis to determine manganese concentrations in marine sediments and to demonstrate that diminished deposition of reducible manganese does indeed occur contemporaneously with the accumulation of manganese in distal, more oxygenated marine environments. Mr. Dickens' advisor is Professor Robert Owen.

Michigan State University

Neutron activation analysis was used as a means of identifying source materials for archaeologically derived pottery, specifically in terms of understanding socially determined patterns of procurement and paste preparation. A master's thesis that recently resulted from work performed at the Nuclear Reactor Laboratory made an important contribution to understanding these issues for the time period A.D. 800 through 1500 in the upper Great Lakes. This work is under the direction of Professor William Lovis of the Anthropology Department.

Professor Thomas A. Vogel of the Geological Sciences Department is utilizing neutron activation analysis on geological samples to obtain information about the exact location and nature of the Cenozoic North America - Eurasia Plate boundary in Northeast Asia. Of specific interest are basaltic samples used to determine block geometries in Eastern Russia. The basalt is characterized by high alkali and moderate to low niobium concentrations and has a composition characteristic of within-plate volcanism.

National Institute of Standards and Technology

Neutron activation analysis was performed for Dr. William C. Cunningham to test and verify a new method for determining sodium concentrations in biological materials. The method was tested at several facilities around the country.

University of Colorado

Geological samples are being irradiated for neutron activation analysis for Professor John Drexler of the Geology Department.

COBALT-60 IRRADIATOR

Central Michigan University

Douglas Beyer of the Dow Chemical Company is working with Professor Robert Howell by irradiating 3-3 dichloropentane and studying the radiation products of this chlorinated polymer.

Michigan State University

Xianda Zhao, a research assistant working for Professor Thomas C. Voice in the Civil and Environmental Engineering Department, is gamma sterilizing biofilm-coated granular-activated-carbon (GAC). He is testing a pilot scale fluidized bed system used to treat ground water contaminated with three milligrams of toluene per liter of water. The GAC is used as the carrier media for microbial growth. The biofilm-coated GAC is taken from the system each month and sterilized. The sterilized GAC is used in an adsorption isotherm experiment to determine the remaining adsorption capacity.

Gelman Sciences Incorporated

Thomas Dodson and Todd Borton have utilized the cobalt-60 irradiator to sterilize various experimental medical devices such as microfunnels and filters. The effects of radiation on these devices is also being observed.

FEATURE ARTICLE

NEUTRON DEPTH PROFILING

Introduction

Neutron depth profiling (NDP) is a nondestructive technique for the measurement of isotopic concentrations as a function of depth. Using neutrons to probe the first few micrometers of solid surfaces, NDP has become an important tool in the nondestructive analyses for several technologically important elements. Neutron depth profiling was introduced in 1972 at the National Institute for Standards and Technology (NIST) for the determination of implantation boron distributions in wafers. Researchers subsequently advanced the technique and greatly expanded the applications. Over one hundred articles have been published describing the use of NDP in research applications. The widespread use of NDP is limited by the availability of intense neutron sources--nuclear research reactors. Table 1 lists the NDP facilities around the world, including some under construction.

TABLE 1

List of NDP Facilities Found Around The World

Institution	Location	Flux (n/cm ² /sec)
1) National Institute of Standards and Technology	Gaithersburg, MD, U.S.A	1.2 x 10 ⁹
2) Institut Max von Laue-Paul Langevin	Grenoble, France	4 x 10 ⁸ 1 x 10 ⁹
3) Brookhaven National Lab	Upton, NY, U.S.A.	2.3 x 10 ⁸
4) Czechoslovakia Academy of Sciences	Rez near Prague, Czechoslovakia	1.4 x 10 ⁸
5) Chalk River National Lab	Chalk River, Ontario, Canada	3 x 10 ⁷
6) Hahn-Meitner-Institut	Berlin, Germany	3 x 10 ⁷
7) University of Texas-Austin	Austin, TX, U.S.A.	>10 ⁷
8) Texas A&M University	College Station, TX, U.S.A.	1.4 x 10 ⁷
9) University of Michigan	Ann Arbor, MI, U.S.A.	1.4 x 10 ⁷
10) North Carolina State University	Raleigh, NC, U.S.A.	10 ⁷
11) Joint Institute for Nuclear Research	Dubna, USSR	10 ⁶
12) Musashi Institute of Technology	Kawasaki, Japan	10 ⁶
13) FMBR reactor facility	Brunswick, Germany	(?)

Fundamentals of the Technique

A number of light elements such as He, Li, Be, and B have an isotope that, upon capturing a thermal neutron, undergoes an exoergic charged particle reaction (see Table 2). The reaction produces either a proton or an alpha particle, depending upon the isotope, and a recoiling nucleus. Each particle emitted has a very well known kinetic energy defined by the nuclear disintegration energy (Q-value) of the reaction. This monoenergetic particle also serves to identify the parent element.

To determine a concentration profile, a well-collimated beam of low energy neutrons is used to uniformly illuminate a sample volume. While most neutrons pass through the sample without interacting, areas containing elements such as those listed in Table 2 act as isotropic sources of monoenergetic charged particles. The particles travel outward, essentially in straight paths, and lose energy primarily through interactions with the sea of electrons that fill the matrix. The difference between the initial particle energy and its residual energy upon emerging from the sample surface is directly related to its depth of origin. Because thermalized neutrons carry relatively little momentum, the reaction center of mass is coincident with the site of the parent atom. The sample chamber is evacuated to avoid the loss of additional energy as the particle travels from the sample surface to the detector.

TABLE 2

List of Isotopes that are Useful for NDP Determinations

Summary of Neutron Depth Profiling (NDP) Reactions

Elem	Reaction	% Abundance or (atoms/mCi)*	Energy of Emitted Particles (keV)	Cross Section (barns)	Detection Limit (atoms/cm ²)#
He	³ He(n,p) ³ H	0.00014	572	5333	1.5 x 10 ¹²
Li	⁶ Li(n,α) ³ H	7.5	2055	940	9.0 x 10 ¹²
Be*	⁷ Be(n,p) ⁷ Li	(2.5 x 10 ¹⁴)	1438	48000	1.7 x 10 ¹¹
B	¹⁰ B(n,α) ⁷ Li	19.9	1472	3837	2.1 x 10 ¹²
N	¹⁴ N(n,p) ¹⁴ C	99.6	584	1.83	4.5 x 10 ¹⁴
O	¹⁷ O(n,α) ¹⁴ C	0.038	1413	0.24	3.5 x 10 ¹⁶

TABLE 2 (cont)

List of Isotopes that are Useful for NDP Determinations

Summary of Neutron Depth Profiling (NDP) Reactions

Elem	Reaction	% Abundance or (atoms/mCi)*	Energy of Emitted Particles (keV)	Cross Section (barns)	Detection Limit (atoms/cm ²)#
Na*	²³ Na(n,p) ²² Ne	(4.4 x 10 ¹⁵)	2247	103	2.3 x 10 ¹¹
S	³² S(n,α) ³⁰ Si	0.75	3081	411	6.0 x 10 ¹⁶
Cl	³⁵ Cl(n,p) ³⁵ S	75.8	598	17	1.7 x 10 ¹⁶
K	⁴⁰ K(n,p) ⁴⁰ Ar	0.012	2231	56	1.9 x 10 ¹⁵
Ni*	⁵⁹ Ni(n,α) ⁵⁶ Fe	(1.3 x 10 ²⁰)	4757	340	7.0 x 10 ¹⁴
Bi	²⁰⁹ Bi + n → ²¹⁰ Bi* → ²¹⁰ Po* α(5.3 MeV) ²⁰⁶ Pb			12.3	

* Radioactive species

Detection limit based on 0.1 counts per second, 0.1% detector solid angle, and a neutron intensity of 6 x 10⁸s⁻¹

The number of charged particle counts collected within a given energy band is directly proportional to the concentration of the nuclide located within that corresponding depth interval. The entire energy spectrum is displayed on a multichannel analyzer and provides a real time depth profile. The time required for an analysis can range from a few minutes to a few hours depending on the nuclide and the desired precision. A boron concentration of 1 x 10¹⁵ atoms per cm² will typically take about an hour to obtain 1 percent precision. Because the precision of the measurement is limited by counting statistics and since the background signal is almost negligible, a sample volume could be analyzed for tens of hours to steadily improve the definition of low concentration profiles.

Each material and reaction particle type has a characteristic stopping power, consequently the resolution and the depth of profiling varies between materials. A good illustration is the ¹⁰B(n,d)⁷Li reaction. The recoiling lithium mass loses energy at a higher rate than the lighter, more energetic alpha particle, allowing improved depth resolution; however, the alpha particles have greater range and consequently allow deeper profiles to be obtained (1 to 2 μm deeper). The full width at half maximum resolution (FWHM) in the profile obtained from the 1472 keV alpha of a boron reaction in silicon is typically 10 to 20 nanometers. On the other hand, protons in the same material from the ²²Na(n,p)²²Ne reaction are emitted at 2246 keV and give a resolution on the order of only a few hundred nanometers. This proton, though, can be used to probe 30 to 40 micrometers in depth.

Since the emission of particles is isotropic, the detector can be placed at an angle with respect to the sample surface to view different particle path lengths emitted from the same sample depth. In this way, for example, the depth resolution of boron in silicon can be improved from 20 nm to 7 nm. Minute profile variations in the first nanometer of a sample surface can often be identified by comparing differentiated spectra of known homogeneous standards with that of differentiated spectra of unknown samples.

Based on an article, Neutron Depth Profiling Techniques and Facilities", by R. Gregory Downing and George P. Lamaze, National Institute of Standards and Technology, Gaithersburg, MD, U.S.A.

NUCLEAR REACTOR

LABORATORY

Profile of Services

Neutron Irradiation Services

In-core, pneumatic tube, and beamport irradiations with high energy (fast) and low energy (thermal) neutrons. Thermal neutron range: 8×10^6 to 1.5×10^{13} n/cm²/sec.

Neutron Activation Analysis

Identification of trace quantities of sixty-two elements including most metals and rare earth elements utilizing a technique that is almost non-destructive and requires very small sample volumes.

Gamma Irradiation Services

Gamma irradiations utilizing a large cobalt-60 source to sterilize bone and cartilage for reconstructive surgery and to study radiation effects on materials.

Neutron Radiography

Radiographic imaging of low density materials such as plastic, oil, water, and gasoline contained in heavy materials and porous media that cannot be imaged with ordinary x-rays.

Radiopharmaceutical Preparation

Production and distribution of large quantities of investigational drugs containing iodine-123, iodine-125, and iodine-131 to almost 250 hospitals and medical research institutions for diagnosis and therapy of adrenal gland cancer and adrenomedullary diseases.

Radiochemical Production

Preparation of bromine-82 labeled motor oil for use in engine oil economy research programs, and bromine-82 labeled toluene, argon-41, sodium-24, and lanthanum-140 for use in oil refinery flow tests.

Testing Programs

Accelerated neutron and gamma aging of reactor materials; fast neutron damage effects in reactor vessel steels; and quality assurance tests of irradiated materials including neutron attenuation properties, strength, gas evolution, radionuclide content, and changes in physical parameters.

Training

Neutron activation analysis and reactor operations laboratories for university students, advanced high school students, and electric utility engineers and reactor operators.

NUCLEAR REACTOR LABORATORY DIRECTORY

Hours of Operation:

Monday-Friday Sept. 1 - April 30 8:00 a.m. - 5:00 p.m.
May 1 - Aug. 31 7:30 a.m. - 4:00 p.m.

Facilities can be made available 24 hours a day, if required.

Tours: Monday-Friday 9:00 a.m. - 4:00 p.m.

Tours should be scheduled at least 48 hours in advance.

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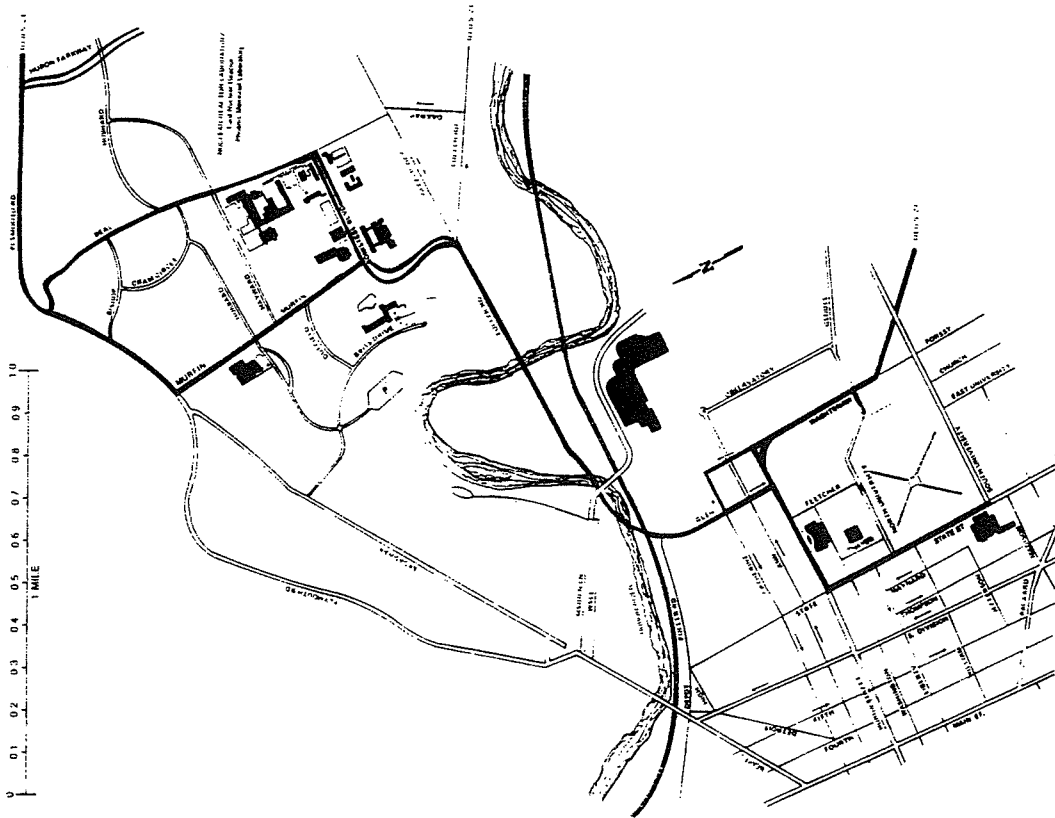
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Nuclear Reactor Laboratory Location
University of Michigan Central and North Campus

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