AN ABSTRACT OF THE THESIS OF

David S. Pratt for the degree of Master of Science in Radiation Health presented on July 1, 1993.

Title: Applications of Beta Spectroscopy

Abstract approved: Signature redacted for privacy.

Jack F. Higginbotham

Two applications of beta spectrometry using active gamma-ray discrimination are described. The beta spectra of smears taken at a nuclear power plant at the end of a 300 day shutdown were characterized. The average beta energy at "contact" (1.5 cm) was between 450 and 550 keV for locations influenced by activation products and slightly higher for locations with a high percentage of fission products. The spectrum hardened about 300 keV at the normal working distance of 30.5 cm. The possibility of pure beta emitters in the spectrum was investigated by comparing a composite beta spectrum consisting of nuclides identified by gamma-ray spectrometry to the beta spectrum from the smear. There was no indication of significant amounts of pure beta emitters in the spectra.

The second application of beta spectroscopy was developed, in conjunction with liquid scintillation counting, to measure the presence and radioactivity level of calcium-45 in apple juice prior to administration to human subjects. Although the calcium-45 activities

determined by beta spectroscopy and liquid scintillation counting consistently agreed, they both differed by about 20% when compared to the calcium-45 activity expected in the solution to be administered. However, even in view of this difference, the analytical technique developed during this study verified that the calcium-45 activity was well within the range required for use in a human blood absorption investigation. By comparing the beta spectrum from radioactivity in the apple juice to a known calcium-45 beta spectrum, the technique also confirmed that calcium-45 was the only radionuclide present in the apple juice.

Applications of Beta Spectroscopy

by

David S. Pratt

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Working full time while pursuing a graduate degree was not only difficult for me but affected many other people. There is no doubt in my mind that without their cooperation and assistance I would have never finished.

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CONTRIBUTION OF AUTHORS

Two manuscripts were generated from this study. Multiple authors contributed to these manuscripts.

"In-plant Beta Spectroscopy" included contributions from Dr. Jack F. Higginbotham, OSU Nuclear Engineering, and Dr. Wayne Lei, Portland General Electric. Dr. Higginbotham served as a consultant during the beta spectroscopy measurements, reviewed and edited the manuscript, and interfaced with the publisher. Dr. Lei provided the PGE feather analysis and smear variability data, in addition to providing the smears from the Trojan plant for analysis at OSU. He also reviewed and edited the manuscript.

The second manuscript, "Calcium-45 Quality Assurance in a Blood Absorption Study," includes contributions from Professor Arthur G. Johnson and Dr. Jack F. Higginbotham, both members of the OSU Nuclear Engineering Department. This manuscript was reviewed and edited by both Professor Johnson and Dr. Higginbotham.

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INTRODUCTION

This study describes two different practical applications of beta spectroscopy. In one application, smears containing removable radioactive contamination from various location at a nuclear power plant were analyzed. The radioactivity on these smears emitted both beta particles and gamma-rays. Because mixed beta/gamma-ray emitters are found in nuclear power plants, a beta spectrometer with active gamma-ray discrimination is a potentially useful instrument to assess the in-plant beta spectrum. In another application, a quality assurance program was established for Kaiser-Permanente Center for Health Research. This program was required by the regulatory agency to use a pure beta emitter in a blood absorption study. Before these applications are discussed, the concept of the beta decay process and beta spectroscopy must be understood.

The beta decay process has been well described by Wang, Willis, and Loveland.[1] For this study, the term beta will refer to only the negatively charged beta. A beta-minus particle and an antineutrino are emitted from the nucleus of an unstable neutron rich nuclide during the decay event in which a neutron is converted to a

proton. At this time, a negligible amount of excess energy from this conversion is transferred to the recoil nucleus; the remainder is shared between the beta particle and the antineutrino in the form of kinetic energy. This sharing of energy between the beta particle and the antineutrino is not proportional. The beta particle can receive all the excess energy and the antineutrino none. Or conversely, the beta particle can receive none and the antineutrino receive all of the excess energy. In most beta-minus decay events, the beta particle is emitted with approximately one third of the maximum energy and the antineutrino is emitted with the remainder. Nevertheless, beta particles are emitted with energy over the entire range from zero to the maximum energy.

Beta spectroscopy is the measurement of the distribution of these beta particle intensities as a function of energy. Beta spectroscopy is, however, hampered by gamma-rays that are often emitted concurrently with beta particles during the decay process. These gamma-rays, along with the beta particles, interact with the spectroscopy detector and distort the energy spectrum.

One beta spectroscopy method described by D.E. Martz, et al.,[2] involved taking two measurements, the

first with the detector bare measuring both beta and gamma energies, and the second with the detector shielded to attenuate the maximum energy of the beta particles. By subtracting the second spectrum from the first, the beta spectrum was obtained. Although potentially useful, this method is not desirable for three reasons. First, it required two separate measurements. Second, gain shifts could distort the spectrum. Third, the gamma field must have remained constant for both measurements.

Real time discrimination of the gamma-ray is more desirable because it does not have these limitations. Real time discrimination requires identifying whether the output signal from the detector originated from a beta particle or gamma-ray. A beta spectrometer with this capability was developed at Kansas State University[3] (KSU) and was used in this study.

The KSU detector accomplished the discrimination of beta particles from gamma-rays by joining two detectors utilizing different detection properties to form a single unit. The first detector, used for gating purposes, was a thin gas flow proportional counter with P-10 argon/methane gas. Due to the low density of the gas and the small volume of the detector, a gamma-ray was unlikely to ionize the gas during its passage through the gas chamber. However due to its charge, the beta

particle was likely to ionize the gas in the chamber. This ionization caused a signal to be sent from the detector. This signal was amplified, converted to a 5 volt logic pulse in a single channel analyzer, and sent to a linear gate stretcher.

The second detector was a BC-400 plastic scintillator that detected both beta and gamma interactions. The light output of the scintillator from these interactions was converted to an electrical signal in a photomultiplier tube and was then amplified. The signal was delayed in order to arrive at the linear gate stretcher after the signal from the gas flow detector.

The linear gate stretcher receives the input signals from both the BC-400 scintillator and the gas flow detector. In this instrument, the gas flow detector signal was lengthened in time duration to greater than the duration of the BC-400 scintillator signal. This permitted the linear gate stretcher to have three modes of operation: COINCIDENCE; ANTICOINCIDENCE; and NORMAL.

In the COINCIDENCE mode, the signal from the BC-400 plastic scintillator was allowed to pass only if there was also a simultaneous signal from the gas flow detector. This would occur when the detectors sensed a beta particle.

In the ANTICOINCIDENCE mode, the signal from the BC-400 detector was allowed to pass only if there was no signal from the gas flow detector. This occurred when a gamma-ray passed through the gas chamber without interacting and deposited its energy in the BC-400 scintillator.

The NORMAL mode of operation eliminated the gating function of the gas flow detector. All signals that originated in the BC-400 scintillator from either beta particles or gamma-rays passed through the linear gate stretcher to the multichannel analyzer.

The multichannel analyzer segregated the BC-400 signals by pulse height into channels. The energy distribution was thus derived.

This study consists of two applications of beta particle spectrometry which were conducted at the Oregon State University Radiation Center using the KSU detector. The first manuscript, "In-plant Beta Spectroscopy," was published in Radiation Protection Management and the second manuscript, "Calcium-45 Quality Assurance in a Blood Absorption Study," will be submitted to the Journal of Nuclear Medicine Technology.

The first manuscript, discussed in chapter 2, characterized the beta spectra from smears taken at various locations throughout the Trojan nuclear power

plant during a refueling and maintenance outage. The contribution of beta particles to worker's radiation skin dose can be significant during these outages due to the numerous piping systems, pumps, and valves that are open during maintenance. These beta particles originate from the many different nuclides that constitute activated corrosion products and fission products.[4] Knowing the characteristics of the beta spectra permits better dose estimations and the effectiveness of protective clothing on reducing personnel exposures. The beta spectrometer with active gamma-ray discrimination has the potential to be a useful instrument to characterize the in-plant beta spectra because of the mixed beta/gamma-ray emitters that are found in nuclear power plants.

The second manuscript, discussed in chapter three, describes the use of the beta spectrometer to quantitatively and qualitatively identify calcium-45 in an apple juice solution intended for human consumption as part of a scientific study. Calcium-45, which is a pure beta emitter, was selected for use in a blood absorption study by a health research center. Regulatory requirements mandated a quality assurance program to ensure that the material contained only calcium-45, and to confirm that the radioactivity agreed with the radioactivity value provided by the supplier. As part of

the quality assurance effort, the beta spectrum from the radionuclide in the apple juice was compared to the beta spectrum from calcium-45 obtained from a commercial vendor. Although active gamma-ray discrimination was not required for this application, the spectrometer still had the potential to satisfy the specific quality assurance requirements.

Chapter 2
IN-PLANT BETA SPECTROSCOPY

IN-PLANT BETA SPECTROSCOPY

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2.1 ABSTRACT:

Beta particle energy spectra of smears taken from various locations in the Trojan nuclear power plant at the end of a 300-day refueling and maintainance outage were measured using the feather analysis technique and beta particle spectroscopy with active gamma ray discrimination.

The average beta energy at "contact" (1.5 cm) was between 450 and 550 keV for locations influenced by activation products and slightly higher for locations with a high percentage of fission products. The spectrum hardens about 300 keV at the normal working distance of 30.5 cm.

A composite beta spectrum of nuclides determined to be present in the fuel transfer canal smear based on gamma spectroscopy was compared to the beta spectrum of the fuel transfer canal smear.

2.2 INTRODUCTION:

Increasing regulatory interest has been shown in the selection of the appropriate beta correction factors (BCF) for thermoluminescent detector (TLD) systems used in general personnel dosimetry at power plants. Further interest exists in the effect(s) of placing the TLD under protective clothing. In order to properly address these issues, health physicists must determine the energy distribution of the beta particles which contribute personnel exposure. The purpose of this paper is to characterize a typical in-plant beta particle energy spectra.

An assessment of the in-plant beta spectra was performed by the Radiation Protection Department[5] of Portland General Electric's (PGE) Trojan power reactor facility. Smears, taken from various plant locations and systems at the end of a 300 day refueling and maintenance outage, were analyzed using the classical "Feather" analysis. In addition, PGE contracted with Oregon State University's (OSU) Radiation Center to provide additional spectral analysis of these smears. The results of these analyses are presented.

2.3 SAMPLE LOCATION AND PREPARATION:

Standard cloth disk smear samples (approximately 4.5 cm diameter) were collected from sampling points selected to span the expected variability in radionuclide mix that might exist in-plant. Two samples were collected at each of the following locations: (1) spent fuel pool (SFP) lip at the 93 foot elevation of the fuel building; (2) on the inside lip of the fuel transfer canal in the fuel building; and (3) on a work table inside a HEPA ventilated, free-standing tent used to decontaminate equipment and components from steam generator maintenance and repair work. Also collected for analysis were a one liter sample of reactor coolant system (RCS) water filtered through a 0.22 μm Millipore filter, a smear of a SFP light fixture, and a snippet of a Clean Waste (CW) Filtration System bag filter. When possible, two samples were taken at each location to assess the effect, if any, of sampling variability versus variability in concentration.

The samples were mounted on stainless steel planchets to approximate the backscatter that might occur under field conditions, such as contamination on piping. The planchets were covered with clear plastic film (~1.0 mg/cm^2) to contain any loose contamination.

2.4 ANALYTICAL EQUIPMENT AND METHOD:

The "Feather" analysis was conducted at Trojan using an Eberline Model HP-210T tungsten-shielded "pancake" type GM tube detector with a thin (1.7 mg/cm^2), 4.45 cm diameter window protected by a stainless steel screen (79% open). Signals from one minute counts were processed into an Eberline Model MS-2 scaler. Two counting geometries were assessed. In the first, an Eberline Model SH-4A sample holder was utilized to maintain a sample to detector distance of approximately 1.5 cm. This source to detector distance allowed the insertion of standard aluminum absorber disks. In the second geometry, the sample was positioned 30.5 cm (12") from the detector to approximate a representative worker-to-source distance when individuals are working with or near contaminated equipment or components. Only the two samples with the highest activity were measured in this geometry. A plywood "jig" allowed the placement of absorbers close to the detector face in order to determine the effective spectrum at that distance.

In addition to the "Feather" analysis, all samples with the exception of the bag filter from the clean waste filtration system were analyzed at OSU using a spectrometer developed by J. F. Higginbotham and G. G.

Simons at Kansas State University.[3] The spectrometer consists of a gas flow proportional detector, a BC-400 plastic scintillator, and Nuclear Instrument Modules (NIM) as shown in Figure 2.1. The gas flow proportional detector, which is insensitive to gamma-ray interactions, functions as a gating detector to control the signal from the scintillator.

The spectrometer has three modes of operation. In the COINCIDENCE (beta) operational mode, the output of the scintillation detector is routed to a multichannel analyzer (MCA) only when the gas flow detector has sensed the passage of a beta particle. In the ANTICOINCIDENCE (gamma) operational mode, scintillation detector output is channeled to the MCA only when there is no signal from the gas flow proportional detector. This occurs when a gamma-ray passes through the chamber without interacting with the gas. The third operational mode, NORMAL (combined), bypasses the gating function of the gas flow detector so that pulses from both beta and gamma interactions in the scintillator are sent to the MCA.

Coincidence timing of the spectrometer gating function was performed using a technique described by W. R. Leo.[6] This required the installation of additional identical delay amplifiers immediately upstream of the linear gate stretcher. As shown in Figure 2.2, the

Figure 2.1

Block diagram of the OSU beta-particle spectrometer with active gamma-ray discrimination.

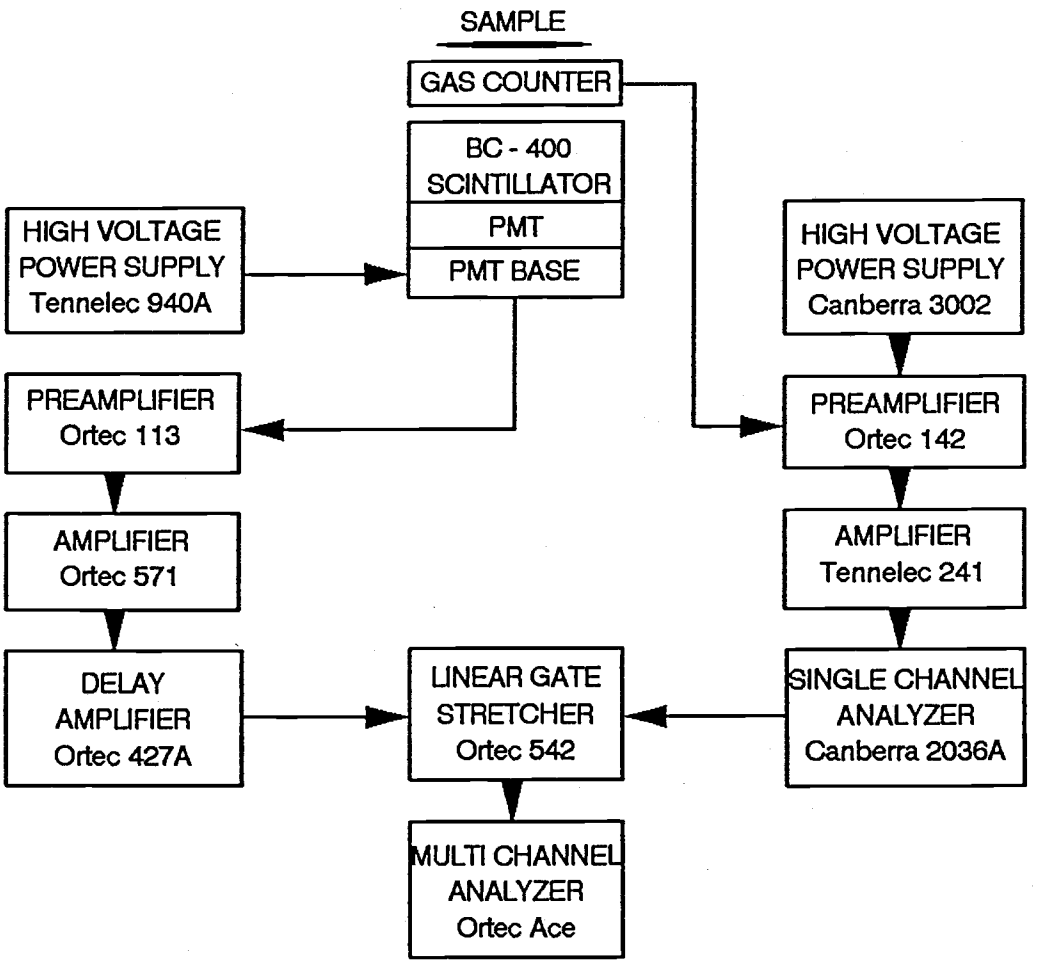
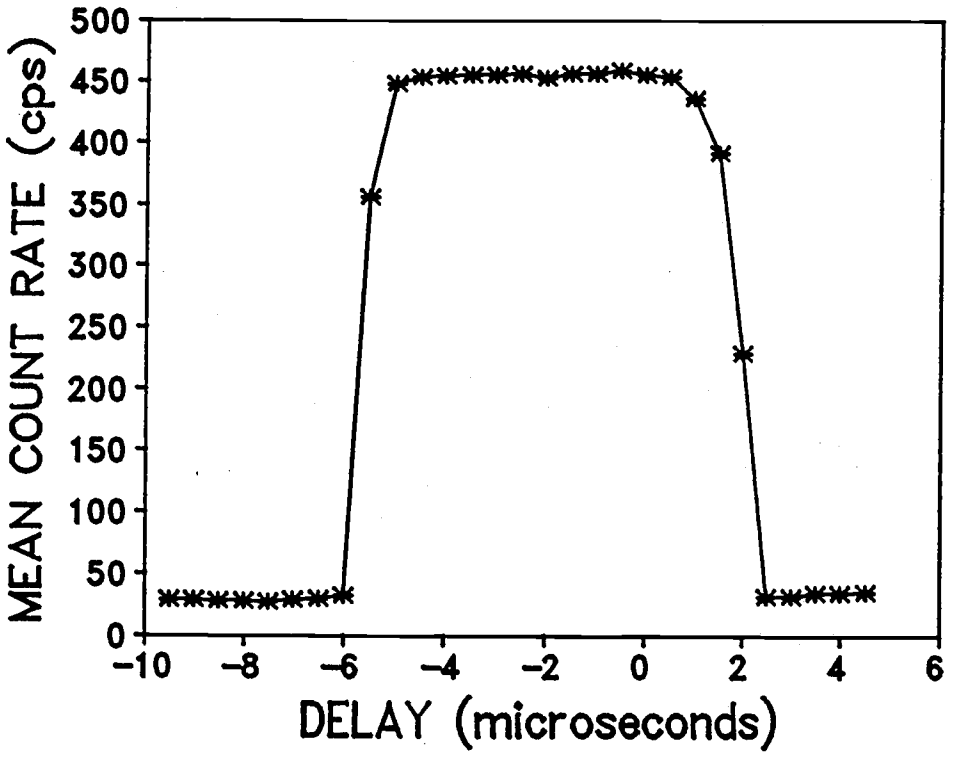


Figure 2.2

Coincidence timing of the OSU spectrometer gating function.



coincidence plateau ranged between -4.5 to +0.5 μs. For the OSU spectral measurements, the scintillator signal was delayed to a point near the center of the plateau at -1.75 μs.

The energy calibration of the spectrometer was performed by measuring the spectra of strontium-90/yttrium-90, cobalt-60, and cesium-137 sources (Figures 2.3, 2.4, and 2.5). These sources were prepared by depositing a known quantity of the radionuclide on a standard cloth smear mounted on a stainless steel planchet, drying the smear under a heat lamp, and covering the planchet with a thin plastic film. Smears standards containing ruthenium-106 and cerium-144 were also prepared. The endpoint channel numbers corresponding to the maximum beta particle energy for the cobalt-60 (318 keV), cesium-137 (514 keV), strontium-90 (546 keV), and yttrium-90 (2281 keV) were calculated using a linearization technique developed by Cramer, et al.[7] In this technique, a linearization variable, $Y_{(x)}$, is related to the number of counts in channel x, $N_{(x)}$ by the expression:

$$Y_{(x)} = \left[\frac{N_{(x)}}{X^k}\right]^{1/2}$$

Figure 2.3

Energy distribution for the beta particle (coincidence), gamma-ray (anticoincidence), and combined (normal) response of the OSU spectrometer to Sr/Y-90.

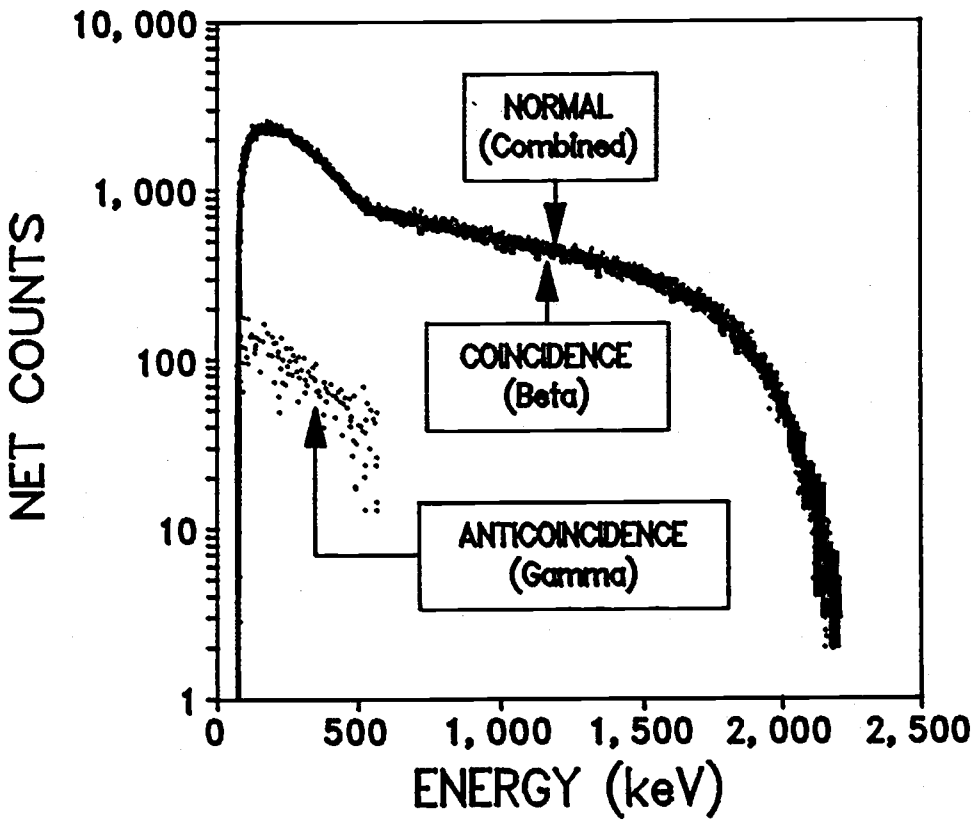


Figure 2.4

Energy distribution for the beta particle (coincidence), gamma-ray (anticoincidence), and combined (normal) response of the OSU spectrometer to Co-60.

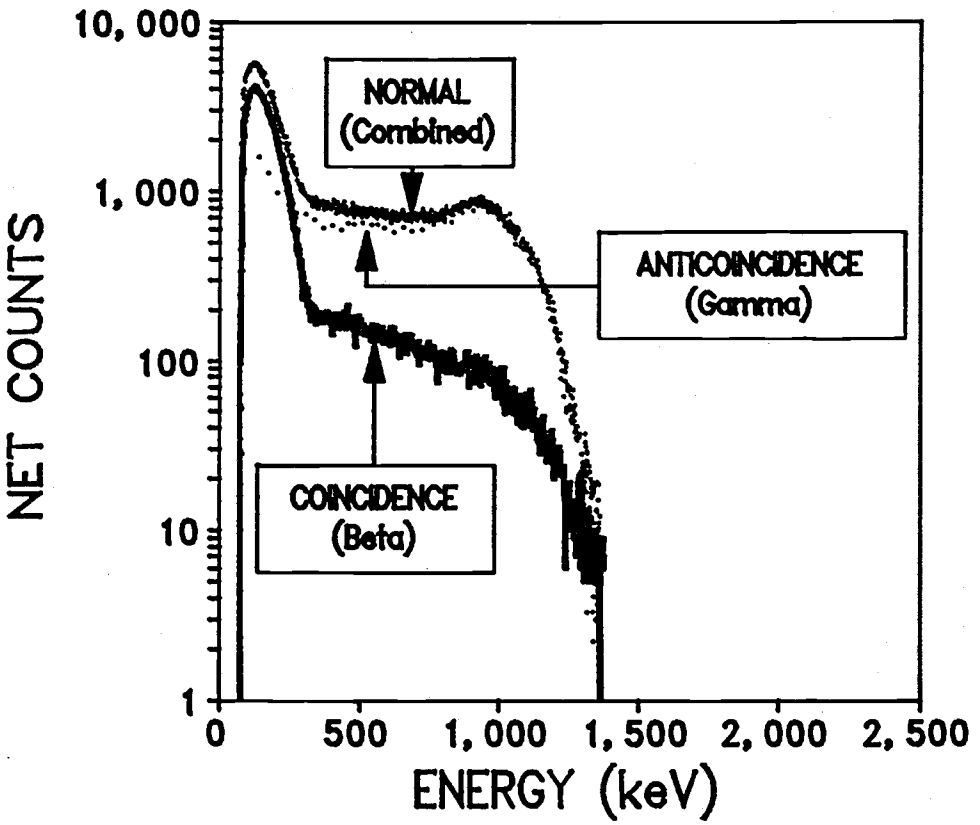
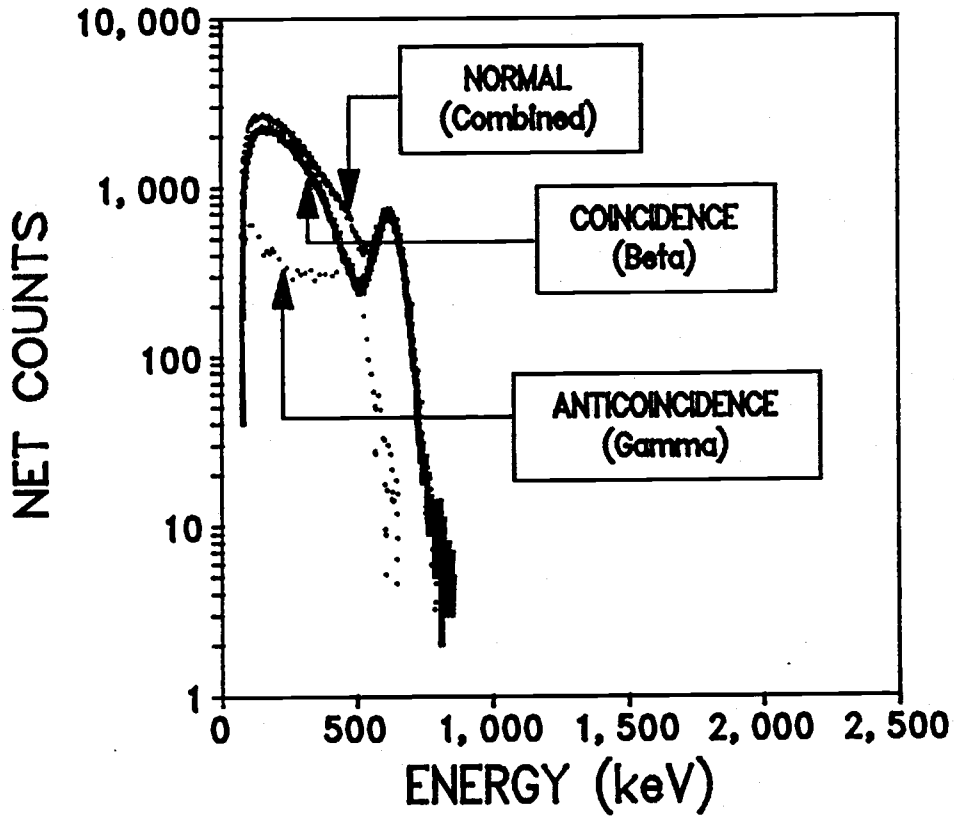


Figure 2.5

Energy distribution for the beta particle (coincidence), gamma-ray (anticoincidence), and combined (normal) response of the OSU spectrometer to Cs-137.



where $N_{(x)}$ = the counts in channel x

K is a constant = 1.4

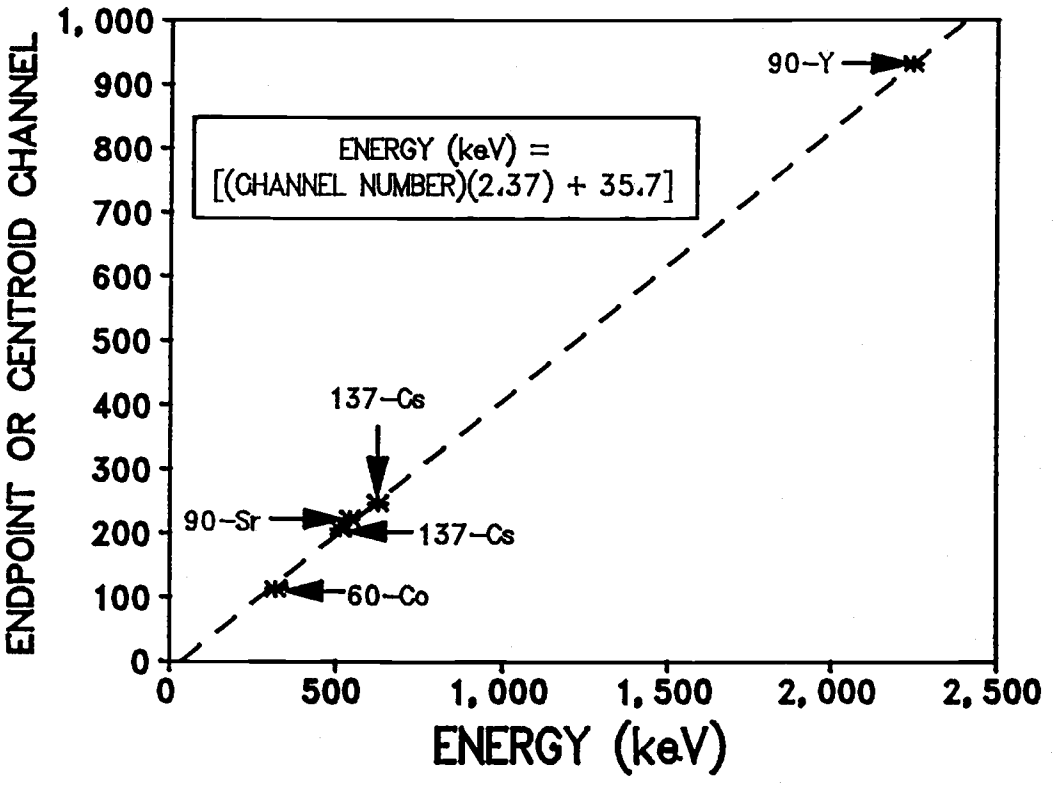
The intercept of the least squares fit of $Y_{(x)}$ versus x corresponded to the endpoint channel number of the beta distribution.

The 624 keV and 656 keV cesium-137 conversion electrons were also used as a calibration point. Because the peaks from these monoenergetic electrons were not resolved, a combination of the Gaussian distributions based on their isotropic yield was used to determine the centroid energy. The pulse height distribution for the conversion electron peak was fitted using a procedure described by Knoll.[8] A least squares fit of the log of the net counts in channel x-1 divided by the net counts in channel x+1 yielded a line whose slope, m, and intercept, b, could be related to the Gaussian centroid channel (x_o) by x_o = -b/m. A least squares fit of the channel number versus endpoint and centroid energy yielded the equation: Energy (keV) = [(Channel Number)*(2.37)+35.7]. The correlation coefficient for the fit was 0.9997. The calibration curve is shown in Figure 2.6.

The energy spectrum from eight samples was measured by counting for 3600 seconds in the NORMAL, COINCIDENCE, and ANTICOINCIDENCE modes. The detector to sample

Figure 2.6

OSU spectrometer calibration curve of endpoint or centroid channel number vs. energy for Co-60, Cs-137, and Sr/Y-90.



distance was approximately 1.5 cm. The smear of the spent fuel pool light fixture contained sufficient radioactivity to also analyze at a detector to sample distance of 30.5 cm.

The gamma-ray spectrum from each of the Trojan samples was also analyzed. This provided additional information on the sample's radionuclide constituents and their abundance.

2.5 ANALYTICAL RESULTS and DATA PRESENTATION:

The "Feather" analysis data are shown in Table 2.1 for the 1.5 cm detector to sample measurements and in Table 2.2 for the 30.5 cm measurements. The collected data were corrected for dead time using the nonparalyzable model given by Knoll[9]:

$n = m/1-m\tau$ where: n = true count rate (cpm)

m = recorded count rate (cpm)

τ = dead time (50 μsec per the manufacturer)

The data were also corrected for instrument background and the photon contribution during each count.

The sample variability, based on the absorber measurements for the three duplicate sample locations, is shown in Table 2.3. The coefficient of variation (the standard deviation divided by the mean) ranged from 9% to 43%. Because these samples were taken by a single radiation protection technician, the variability was probably due to uneven contamination at the sample location. The nuclide percentage of the total gamma activity for each of the samples, shown in Table 2.4, indicates very similar composition for each of the duplicate samples. The absence of a nuclide in the sample is indicated by "xxx" while "0.0" indicates that

TABLE 2.1

PGE "Feather" analysis (in corrected net counts) for increasing aluminum absorber thickness at various in-plant locations measured at 1.5 cm sample to detector distance

ALUMINUM ABSORBER THICKNESS	S/G WORK TABLE		FUEL TRANSFER CANAL		SPENT FUEL POOL LIP		RCS WATER FILTER	SFP LIGHT FIXTURE	CW BAG FILTER
mg/cm^2	#1 net counts	#2 net counts	#1 net counts	#2 net counts	#1 net counts	#2 net counts	net counts	net counts	net counts
0	16,916	9,016	10,677	8,900	19,127	21,649	2,856	198,379	114,132
2	13,602	7,893	8,362	7,245	15,330	17,379	2,071	117,074	105,198
7	11,577	6,701	7,084	6,348	13,188	15,096	1,687	163,343	100,047
13	8,632	5,161	5,324	4,509	10,421	11,838	1,329	145,591	94,198
29	5,773	3,387	3,321	3,021	6,855	8,005	1,028	120,512	82,533
46	5,044	2,953	2,752	2,519	5,472	6,852	846	110,540	77,950
79	4,656	2,817	2,265	2,049	4,603	5,759	706	103,081	73,474
118	4,298	2,545	1,997	1,833	4,090	5,008	635	96,321	68,105
124	3,990	2,377	1,826	1,674	3,766	4,792	642	92,319	65,531
168	3,720	2,143	1,639	1,431	3,262	4,201	629	86,115	61,066
224	3,187	1,833	1,249	1,296	2,639	3,275	481	73,854	51,185
264	2,863	1,631	1,241	1,113	2,410	2,911	440	67,183	47,923
345	2,399	1,389	962	907	1,789	2,269	339	55,900	39,656
413	1,878	1,129	698	679	1,435	1,746	271	44,931	31,982
483	1,604	912	628	600	1,222	1,465	232	36,636	27,103
556	1,473	889	561	515	1,095	1,301	213	33,551	24,301
618	1,320	763	537	492	942	1,100	210	28,613	21,652
679	1,155	646	454	414	787	938	165	23,980	18,355
842	902	481	354	340	583	678	131	16,458	13,968
1,005	733	444	342	275	468	585	129	12,201	10,682
1,275	627	358	275	212	386	452	99	8,542	8,640
1,423	607	328	267	230	383	405	96	8,125	8,150
1,530	589	353	255	210	409	407	91	7,930	7,828
4,469	473	253	241	205	364	306	86	6,626	6,824

TABLE 2.2

PGE "Feather" analysis (in corrected net counts) for increasing aluminum absorber thickness at various in-plant locations measured at 30.5 cm sample to detector distance

ALUMINUM ABSORBER THICKNESS mg/cm^2	SFP LIGHT FIXTURE net counts	CW BAG FILTER net counts
0	4,417	2,133
2	4,203	2,050
7	4,074	1,961
13	3,640	1,881
29	3,202	1,631
46	2,872	1,477
79	2,645	1,387
118	2,507	1,269
124	2,294	1,059
168	2,054	1,011
224	1,685	891
264	1,478	777
345	1,173	630
413	915	493
483	777	417
556	678	385
618	610	321
679	498	272
842	397	212
1,005	338	186
1,275	311	156
1,423	314	141
1,530	303	150
4,469	262	120

TABLE 2.3

PGE smear sample variability (in net counts) at various in-plant locations

SMEAR SAMPLE LOCATION	MEAN net counts	STANDARD DEVIATION net counts	COEFFICIENT OF VARIATION %
S/G WORK TABLE	12966	5586	43
FUEL TRANSFER CANAL	9789	1257	13
SPEND FUEL POOL LIP	20388	1783	9

TABLE 2.4

OSU nuclide percentage of total gamma activity for various nuclides at different in-plant locations

NUCLIDE	S/G WORK TABLE		FUEL TRANSFER CANAL		SPENT FUEL POOL LIP		RCS WATER FILTER	SFP LIGHT FIXTURE	CW BAG FILTER (c)
	#1 %	#2 %	#1 %	#2 %	#1 %	#2 %	%	%	%
Mn-54	2.0	2.1	1.7	2.5	2.6	2.9	10.6	14.2	6.0
Co-57	0.2	0.1	0.1	0.1	0.1	0.1	0.5	0.6	0.5
Co-58	5.0	5.3	3.2	5.4	5.0	4.7	22.1	21.6	20.6
Co-60	80.4	84.9	65.2	59.1	55.0	52.1	57.2	10.9	46.0
Zr-95	0.4	0.2	xxx	xxx	0.4	0.4	xxx	2.3	0.3
Nb-95	0.7	0.0	0.1	0.3	0.3	0.4	2.9	5.3	xxx
Ru-106	7.2	5.1	xxx	3.3	2.1	4.1	xxx	19.6	13.2
Ag-110m	xxx	xxx	2.2	2.1	2.1	2.2	0.3	0.5	6.2
Sn-113	xxx	xxx	xxx	xxx	0.2	xxx	0.8	1.0	0.5
Sb-125	1.0	0.6	1.5	1.4	2.0	1.8	1.4	6.2	3.0
Cs-134	xxx	xxx	4.7	4.3	4.9	5.4	xxx	0.6	0.2
Cs-137	xxx	xxx	14.0	12.9	15.6	16.1	0.4	1.9	0.6
Cd-144	3.0	1.6	7.1	7.7	8.9	8.9	3.4	13.7	2.9
Eu-154	0.2	0.2	xxx	0.4	0.5	0.8	0.4	1.2	xxx
Eu-155	xxx	xxx	0.4	0.3	0.2	0.2	xxx	0.3	xxx

(a) "xxx" indicates the absence of the nuclide in the sample.
(b) "0.0" indicates the nuclide was identified but the percentage of the total gamma activity is insignificant.
(c) Sample analyzed by PGE.

the nuclide was identified but the percentage of the total gamma activity was insignificant.

The average beta energy is shown in Table 2.5 for both the 1.5 and 30.5 cm source to detector distances. The average beta energy for each spectrum was calculated using the expression:

$$E_{avg} = \frac{\sum_{i=1}^{n} N_i E_i}{\sum_{i=1}^{n} N_i}$$

where N_i = net counts in channel i,
E_i = energy of channel i,
and n = number of channels in the spectrum.

With the exception of the spent fuel pool light fixture, the average beta energy for the samples measured at 1.5 cm ranged from 450 to 550 keV. The higher 679 keV average beta energy for the spent fuel pool light fixture was due to the low percentage of cobalt-60 in the spectrum. The spent fuel pool light fixture provided the only sample with sufficient activity to measure at 30.5 cm. The average beta energy at 30.5 cm was over 300 keV higher than the average beta energy at 1.5 cm. This hardening of the spectrum was due to the attenuation of the low energy beta particles by air.

TABLE 2.5

OSU average β energy per sample at various in-plant locations and sample to detector distances

SAMPLE LOCATION		AVERAGE β ENERGY	
		@ 1.5 cm SAMPLE TO DETECTOR DISTANCE keV	@ 30.5 cm SAMPLE TO DETECTOR DISTANCE keV
S/G WORK TABLE	#1	546 keV	(a)
	#2	552 keV	(a)
FUEL TRANSFER CANAL	#1	453 keV	(a)
	#2	470 keV	(a)
SPENT FUEL POOL LIP	#1	506 keV	(a)
	#2	516 keV	(a)
RCS WATER FILTER		546 keV	(a)
SFP LIGHT FIXTURE		679 keV	986 keV
CW BAG FILTER		(b)	(b)

(a) There was insufficient activity to measure at this distance.
(b) Sample was not sent to OSU for analysis.

The results of the Trojan "Feather" analysis are shown graphically in Figures 2.7, 2.8, and 2.9 for a typical sample: Fuel Transfer Canal smear #2. To illustrate intensity distribution, Figure 2.7 presents a semi-logarithmic plot of the net corrected counts in one minute of sufficient energy to pass through the absorber thickness versus the corresponding absorber cutoff energy[10] (keV). The effect of increasing absorber thickness on the sample spectrum intensity level is shown in Figure 2.8 as a semi-logarithmic plot of net corrected counts in one minute of all particles detected passing through the absorber thickness versus absorber thickness (mg/cm^2).

Figure 2.9 shows this same data plotted against the corresponding absorber cutoff energy (keV). This plot allows a crude visual estimate of the approximate number of components in the spectrum and where their beta end point energies might fall. A distinct low energy component resides in the 1 to 300 keV range. This is likely due to the significant presence of cobalt-60, 318 keV E_{max} beta. There are one or more components in the 400 to 1200 keV range that are difficult to identify qualitatively. The high energy end of the spectrum may contain yttrium-90 (beta E_{max} = 2270 keV). Although this plot allows a crude visual estimate of the approximate

Figure 2.7

Effective beta spectrum of net counts from beta particles of sufficient energy to penetrate the absorber thickness vs. equivalent absorber energy for the fuel transfer canal smear #2.

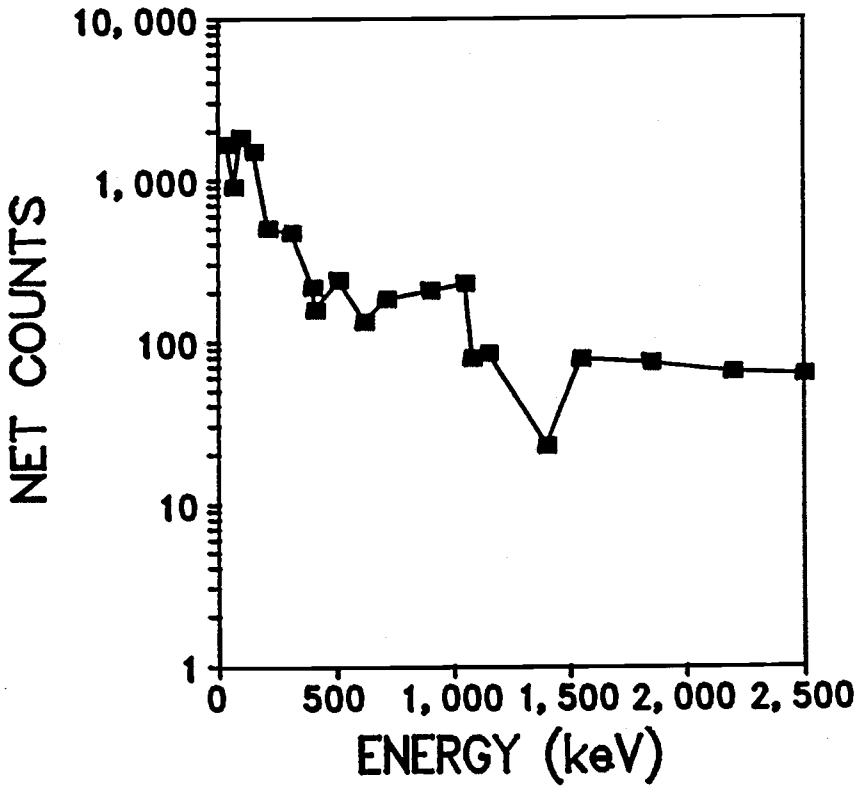


Figure 2.8

Transmission curve of net counts from all beta particles passing through the absorber vs. absorber thickness for the fuel transfer canal smear #2.

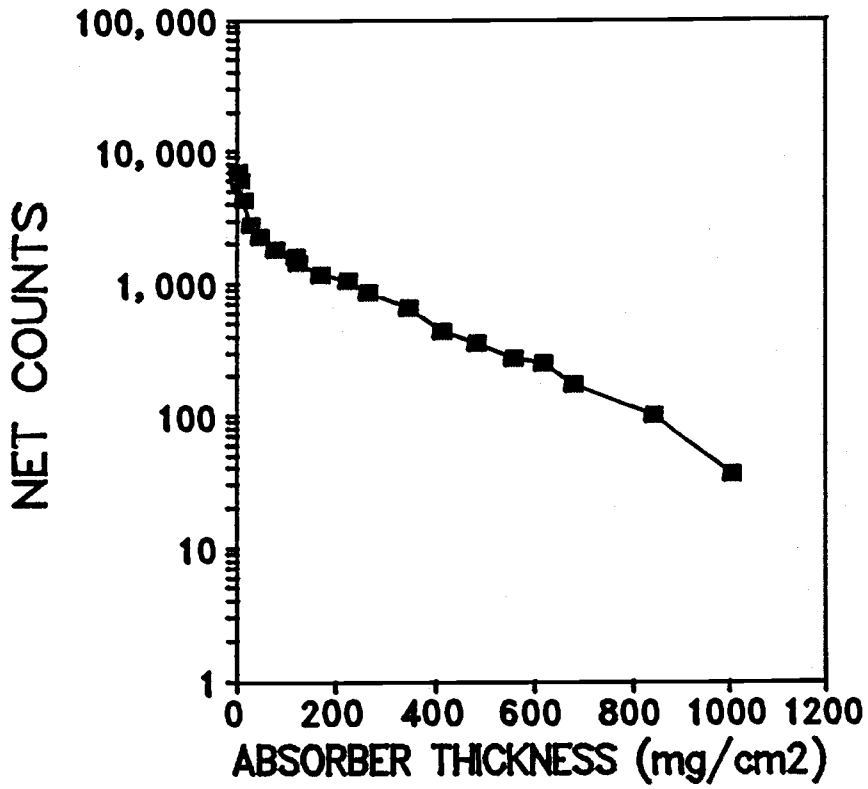
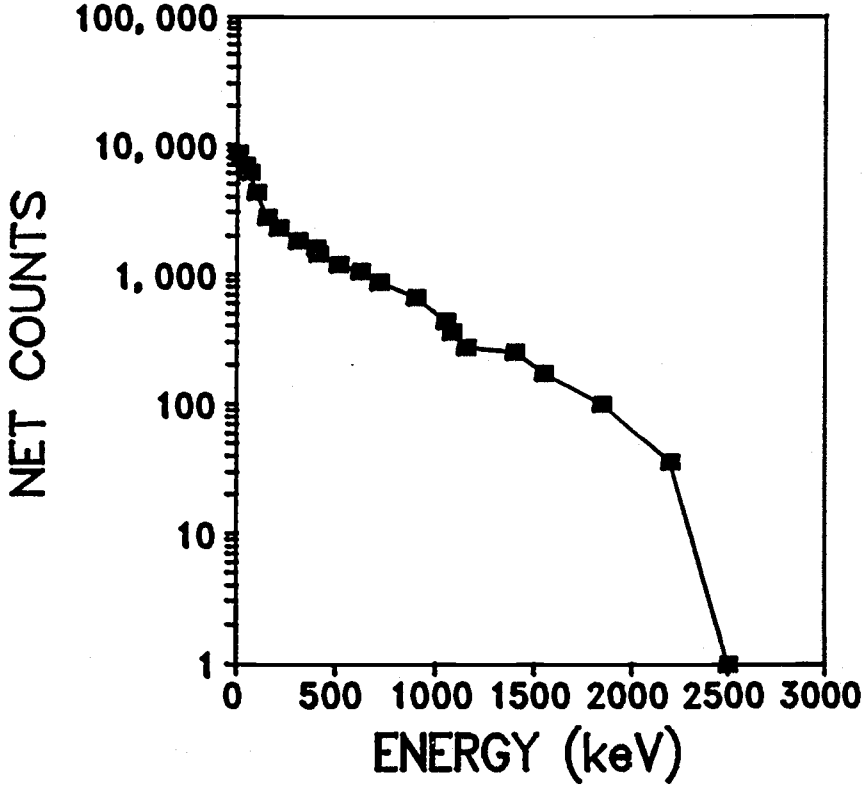


Figure 2.9

Transmission curve of net counts from all beta particles passing through the absorber vs. equivalent absorber energy for the fuel transfer canal smear #2.



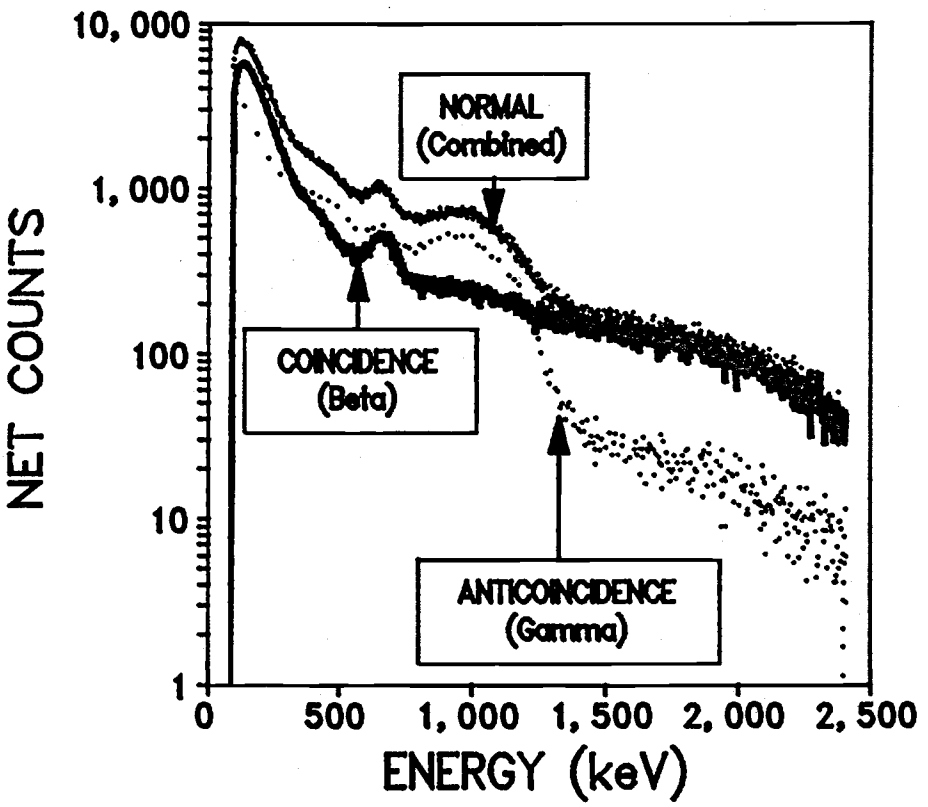
number of components in the spectrum and where their beta end point energies might fall, spectral analysis is a more sophisticated tool to identify the spectrum components.

Figure 2.10 shows the results of such a spectral analysis. The energy spectrum is presented for the Fuel Transfer Canal smear #2 in three modes: COINCIDENCE (beta); ANTICOINCIDENCE (gamma); and NORMAL (combined). Due to the discriminator setting, the spectra are lost below approximately 40 keV. The COINCIDENCE (beta) spectrum shows the components due to: (1) cobalt-60 (beta E_{max} = 318 keV); (2) cesium-137 (beta E_{max} = 514 keV); (3) cesium-137 monoenergetic conversion electrons (624 & 656 keV); and (4) a high energy combination of cerium/praseodymium-144 (2996 keV) and ruthenium/rhodium-106 (3540 keV). The ANTICOINCIDENCE (gamma) spectrum shows a peak at about 1000 keV due to Compton interactions of the 1173 and 1332 keV cobalt-60 gammas. After this peak, the ANTICOINCIDENCE (gamma) spectrum drops off rapidly. As expected, the NORMAL (combined) spectrum represents the sum of the COINCIDENCE (beta) and ANTICOINCIDENCE (gamma) spectra.

The question of whether a pure beta emitter such as strontium/yttrium-90 exists in the beta spectrum can be addressed by comparing a composite spectrum based on

Figure 2.10

Energy distribution for the beta particle (coincidence), gamma-ray (anticoincidence), and combined (normal) response of the OSU spectrometer to the fuel transfer canal smear #2.



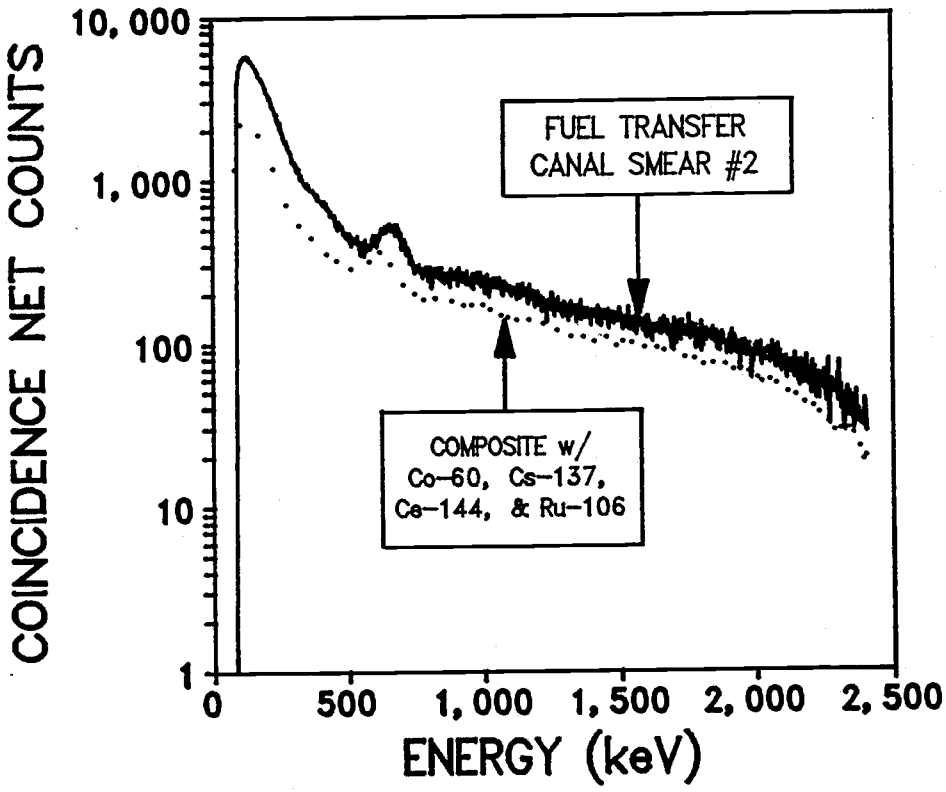
known beta sources to sample spectrum. The gamma analysis results yielded the major radionuclides on the fuel transfer canal smear #2. The beta spectra from prepared cobalt-60, cesium-137, cerium-144, and ruthenium-106 sources were combined in the ratio of their percentage of total activity as determined by the gamma analysis. The sample spectrum and the composite spectrum representing 83% of the total sample activity was plotted in Figure 2.11. The plot shows that the composite spectrum follows the sample spectrum consistently over the entire energy range. This leads to the conclusion that strontium-90 either is not present in the sample or is present in a negligible amount.

2.6 CONCLUSION:

The characteristics of in-plant beta spectra vary with location. At locations predominantly influenced by activation products, the average beta energy at "contact" (1.5 cm) is between 450 and 550 keV. Locations with a high percentage of fission products have a slightly higher average beta energy. The average beta energy hardens about 300 keV at the normal working distance of 30.5 cm. Sampling variability appeared to be due to concentration differences and not sampling technique.

Figure 2.11

Energy distribution for the beta particle (coincidence) response of the OSU spectrometer to the fuel transfer canal smear #2 and to a composite spectra of Co-60, Cs-137, Ce-144, and Ru-106 sources in the ratio of their gamma activity in the smear.



The "Feather" analysis, beta spectral analysis, gamma-ray analysis, and the construction of a composite spectrum from known standards provide complimentary information that characterizes the beta spectrum.

2.7 ACKNOWLEDGEMENTS:

The authors gratefully acknowledge the technical support of Terry Milham, Jerry Lane, and especially Terry Moore for his coordination of sampling and analysis.

Chapter 3
CALCIUM-45 QUALITY ASSURANCE IN A BLOOD ABSORPTION STUDY

Calcium-45 Quality Assurance in a Blood Absorption Study

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3.1 ABSTRACT:

As part of a calcium blood absorption study, an analytical technique was developed to measure the presence and radioactivity level of calcium-45 in apple juice prior to administering the solution to human subjects. The technique is based on beta spectroscopy with active gamma ray discrimination and liquid scintillation counting. Although the calcium-45 activities determined by beta spectroscopy and liquid scintillation counting consistently agreed, they both differed by about 20% when compared to the calcium-45 activity expected in the solution to be administered. However, even in view of this difference, the analytical technique developed during this study verified that the activity of the calcium-45 was well within the range required for use in the calcium absorption investigation. By comparing the beta spectrum from radioactivity in the apple juice to be administered to a known calcium-45 beta spectrum, the technique also confirmed that calcium-45 was the only radionuclide present in the apple juice.

3.2 INTRODUCTION:

A long-term study by a midwestern university is being conducted to identify predictors of osteoporosis, hip fractures, and other bone fractures in older women. A health research center in Portland, Oregon proposed to participate in a portion of the study involving calcium absorption into the blood. Calcium radiolabeled with calcium-45 was orally administered to human subjects in an apple juice carrier and a blood sample was then shipped to the midwestern university for subsequent analysis.

The regulatory agency overseeing this portion of the study required the establishment of a quality assurance program to verify that the calcium-45 activity was within 5% of the stated activity from the supplier (the midwestern university) and to ensure that no contaminate radionuclide was present. Oregon State University's Radiation Center was asked to perform this quality assurance test on the apple juice mixture (referred to as the "test solution") and to verify that the radionuclide was calcium-45. This resulted in the development of an analytical technique which used beta spectroscopy and liquid scintillation counting (LSC) to achieve the desired results.

3.3 MATERIALS AND METHODS:

A calcium-45 reference solution containing a specified amount of the radionuclide in 0.10 ml of calcium chloride was purchased from a commercial radioisotope vendor. The radioactive material was transferred from its original vial to a 100 ml flask using several rinses to ensure complete transfer. The reference material was then diluted to 100 ml total volume with 0.01N HCl. A one ml aliquot of this solution was transferred to another flask and diluted to 10 ml total volume with non-radioactive apple juice supplied by the health research center. A 0.10 ml aliquot was transferred from the 10 ml solution and placed into a liquid scintillation vial containing 10 ml of scintillation fluor.* A "blank" was also prepared by pipetting 0.10 ml of nonradioactive apple juice into 10 ml of fluor. Each vial was vigorously shaken before liquid scintillation counting.

The two vials prepared for liquid scintillation counting were analyzed for 0.2 minutes using a manual liquid scintillation counter.† The varible discriminator module assembly on the liquid scintillation counter was adjusted to bracket the calcium-45 beta spectrum. The beta spectrum was first determined by counting the vial

containing the calcium-45 reference solution for 0.2 minutes at increasing 20 increment differential window settings. The varible discriminator module assembly was then set at zero and 500 to bracket the spectrum. The counting yield for the liquid scintillation system (sometimes called the counting efficiency) was then determined by dividing the observed net counting rate from the reference solution vial by the disintegration rate corresponding to the calcium-45 activity in the reference vial.

The disintegration rate in the reference vial was corrected to account for small autopipeting errors. The correction factor for a specific autopipet volume setting was determined by first weighing three empty vials on a balance, then pipetting the preset volume of water into each vial, and by reweighing the vial and water. This process was performed for each pipet volume. In every case, the weight of the empty vial was subtracted from the weight of the vial plus the water to determine a net water weight, which was then used to establish the volume correction factor for the autopipet. These correction factors are shown in Table 3.1.

Sample homogeneity was measured on the first batch (batch A) of the test solution by collecting 0.10 ml aliquots at four different vertical locations in the

TABLE 3.1

Autopipet volume correction factors

Autopipet Volume Setting (ml)	Empty Vial Weight (mg)	Vial and Water Weight (mg)	Net Weight of Water (mg)	Volume Correction Factor
0.1	1.2939	1.4208	0.1269	1.223
	1.4208	1.5402	0.1194	
	1.5402	1.6610	0.1206	
0.4	1.3194	1.7346	0.4152	1.041
	1.7346	2.1511	0.4165	
	1.3102	1.7274	0.4172	
0.5	1.3214	1.8409	0.5195	1.040
	1.8409	2.3591	0.5182	
	1.3098	1.8259	0.5161	
1.0	1.3124	2.3279	1.0155	1.017
	1.3215	2.3393	1.0178	
	1.3085	2.3252	1.0167	

solution after the sample was shaken mildly. The 0.10 ml aliquots were added to 10 ml of fluor and counted for 0.2 minutes on the LSC. Batch A of the test solution was then thoroughly mixed by shaking and six duplicate vials were prepared and counted. In addition, to further assess sample homogeneity, two duplicate vials were prepared for each of the subsequent test solution batches (B, C, and D).

The sample to be analyzed by beta spectroscopy was prepared by pipetting a 0.50 ml aliquot of the test solution onto a 2.54 cm diameter aluminum planchet. The liquid was slowly evaporated to dryness under a heat lamp. After the test solution dried, the 2.54 cm planchet was attached to a 5 cm diameter planchet with double sided adhesive cellophane tape and was then covered with a thin (approximately 1 mg/cm^2) plastic wrap to contain any loose material. A duplicate test solution sample and a blank planchet with no calcium-45 were prepared in the same manner.

A beta reference standard containing only calcium-45 was used for comparison when examining the beta spectrum from the test solution. The reference standard was prepared from the same 10 ml apple juice and calcium-45 stock solution previously used to make the liquid scintillation standards. A 0.10 ml aliquot of this stock

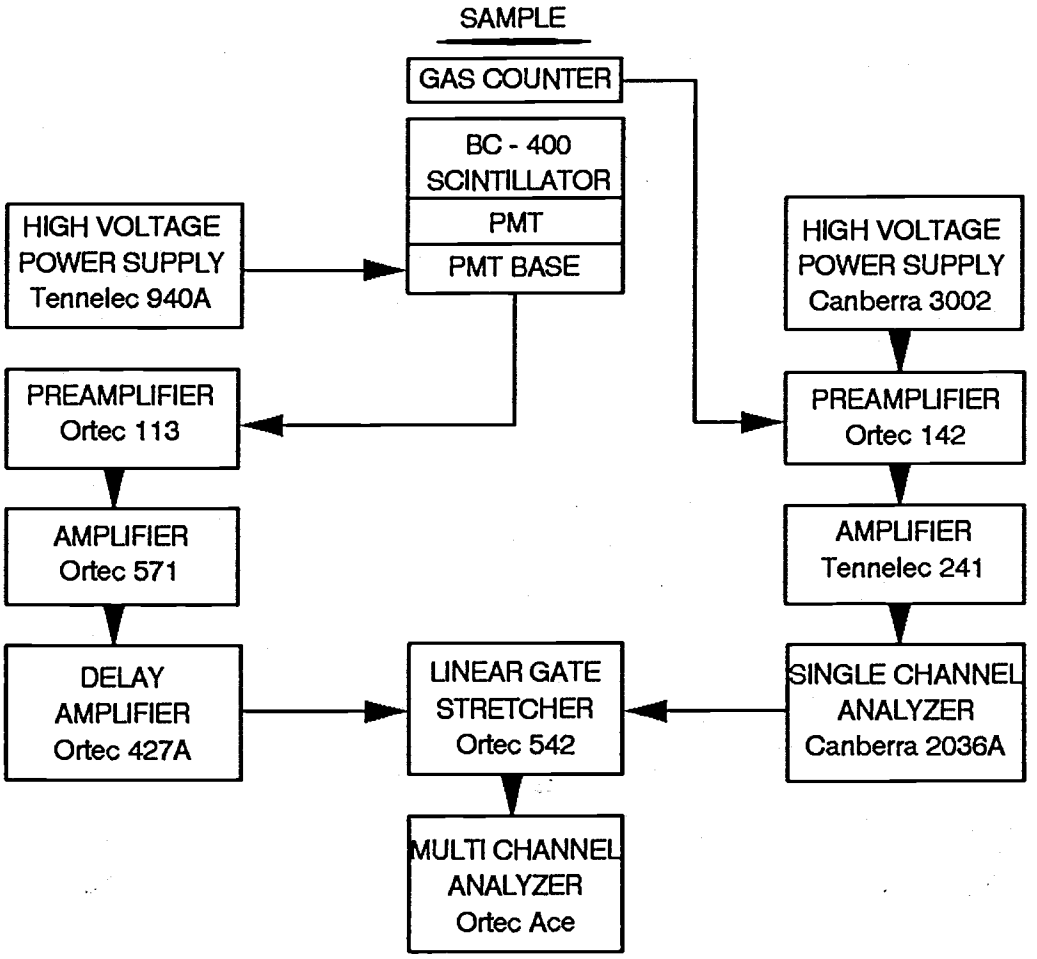
solution was combined with 0.40 ml of pure apple juice and evaporated on a 2.54 cm diameter planchet to achieve a residue deposit and radioactivity concentration approximately equal to that expected on the planchet prepared using the test solution.

The samples were analyzed for 1000 seconds with a source to detector distance of 1 cm using a beta spectrometer developed by J. F. Higginbotham and G. G. Simons at Kansas State University.[3] The spectrometer consisted of a gas flow proportional detector, a BC-400 plastic scintillator, and Nuclear Instrument Modules (NIMs) as shown in Figure 3.1. The gas flow proportional detector, which is insensitive to gamma-ray interactions, functions as a gating detector to control the signal from the scintillator.

The spectrometer has three modes of operation. In the COINCIDENCE (beta) operational mode, the output of the scintillation detector is routed to a multichannel analyzer (MCA) only when the gas flow detector senses the passage of a beta particle. In the ANTICOINCIDENCE (gamma) operational mode, scintillation detector output is channeled to the MCA only when there is no signal from the gas flow proportional detector. This occurs when a gamma-ray passes through the chamber without interacting with the gas. The third operational mode, NORMAL

Figure 3.1

Block diagram of the Oregon State University beta particle spectrometer with active gamma-ray discrimination.



(combined), bypasses the gating function of the gas flow detector so that pulses from both beta and gamma interactions in the scintillator are sent to the MCA. The coincidence mode was used to measure the calcium-45 spectra.

Coincidence timing of the spectrometer gating function was performed using a technique described by W. R. Leo.[6] Additional delay amplifiers were installed for the coincidence timing measurements immediately upstream of the linear gate stretcher for both the scintillation and gas flow detector signals. The coincidence plateau ranged between -4.5 to +0.5 μs. For these measurements, the scintillator signal was delayed to -1.75 μs which is a point near the center of the plateau.

The energy calibration of the spectrometer was performed by measuring the beta spectra of strontium/yttrium-90, cobalt-60, and cesium-137 sources. This calibration, which was similar to that performed for another study, has been described in detail.[11]

3.4 RESULTS:

Correction was required to account for volume errors using the autopipet. The error at 0.10 ml was over 20%. This error decreased to less than 2% at 1.0 ml.

The liquid scintillation counting yield based on the standard prepared from commercially purchased calcium-45 was 97.7% ±11% at one standard deviation. This value, which is slightly higher than the counting yield for carbon-14, was expected because of the higher energy beta emitted from calcium-45.

As shown in Table 3.2, sample homogeneity was not ensured by mild shaking of the thawed test solution. However, after vigorous shaking, the average count rate of six samples drawn from different locations in the test solution was 38264 cpm ± 494 cpm (error reported at one standard deviation).

Table 3.3 shows the results of the calcium-45 activity analyses in the test solutions compared with the decay corrected radioactivity provided by the supplier. The average ratio of the activity determined by liquid scintillation counting divided by the supplier's stated activity shown in Table 3.3 was 0.78 ± 0.03 (error reported at one standard deviation). Similarly, the ratio of the activity determined by beta spectroscopy

TABLE 3.2

Liquid scintillation counts of samples taken at various vertical positions in the test solution after mild shaking

Sampling Position	Gross Counts (cpm)	Net Counts (cpm)
1/4 Down from Surface	39639	39384
1/2 Down from Surface	38004	37749
3/4 Down from Surface	43059	42804
Near the Container Bottom	47984	47729

TABLE 3.3

Supplier's decay-corrected ^{45}Ca activity, ^{45}Ca activity determined by liquid scintillation counting, and ^{45}Ca activity determined by beta spectroscopy of batches A, B, C, and D of the test solution

Test Solution Batch	Supplier's Decay-Corrected ^{45}Ca Activity (μCi)	^{45}Ca Activity Determined by Liquid Scintillation Counting (μCi $\pm$1 s.d.)	^{45}Ca Activity Determined by Beta Spectroscopy (μCi $\pm$1 s.d.)
A	8.9	6.8 $\pm$ 0.8	7.1 $\pm$ 0.7
B	7.7	5.7 $\pm$ 0.6	6.2 $\pm$ 0.6
C	10.0	8.1 $\pm$ 0.7	8.0 $\pm$ 0.7
D	8.2	6.5 $\pm$ 0.6	6.3 $\pm$ 0.6

divided by the supplier's stated activity was 0.79 ± 0.02. This indicates that the reference standard used by the supplier of the apple juice differs from the reference standard used in this work. The calcium-45 reference material purchased by the Radiation Center was accompanied by a technical data sheet listing the activity, specific activity, concentration, volume, and radionuclidic purity. No traceability to other calcium-45 standards was indicated. However, even in the presence of a 20% difference between the projected and measured values for the test solutions, all of the calcium-45 activities in these solutions were within the range of 5 to 10 μCi per 50 ml volume needed by the midwestern university to conduct the study.

Figure 3.2 shows the beta spectrum of the Radiation Center's calcium-45 apple juice standard measured with the beta spectroscopy system. The endpoint energy was 257 keV which compares favorably with the 258 keV maximum energy of calcium-45. No other beta emitters appeared in the spectrum. Figure 3.3 shows the measured beta spectrum for the radioactive material in the test solution supplied to the health research center. The endpoint energy was 253 keV. This value was also near the expected 258 keV and no other beta emitters appeared in the spectrum. Figure 3.4 shows the beta spectra for

Figure 3.2

Energy distribution of the beta particles from the Radiation Center's calcium-45 apple juice standard measured with the OSU spectrometer for 1000 seconds in the coincidence mode.

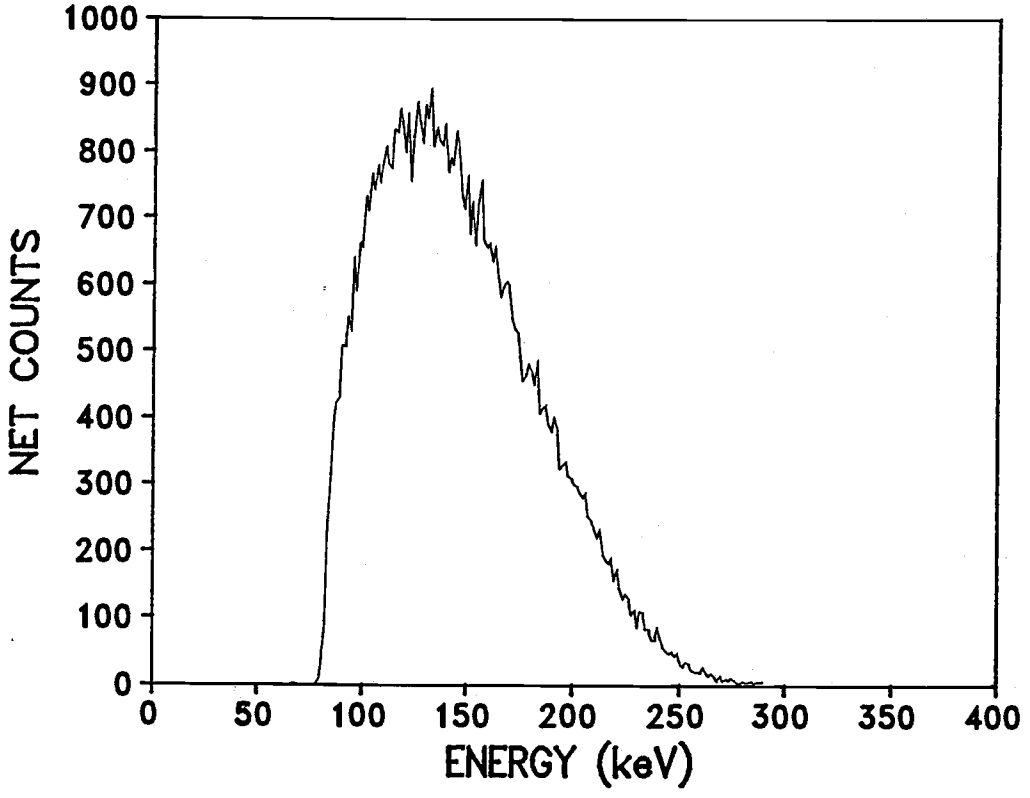


Figure 3.3

Energy distribution of the beta particles from the test solution measured with the OSU spectrometer for 1000 seconds in the coincidence mode.

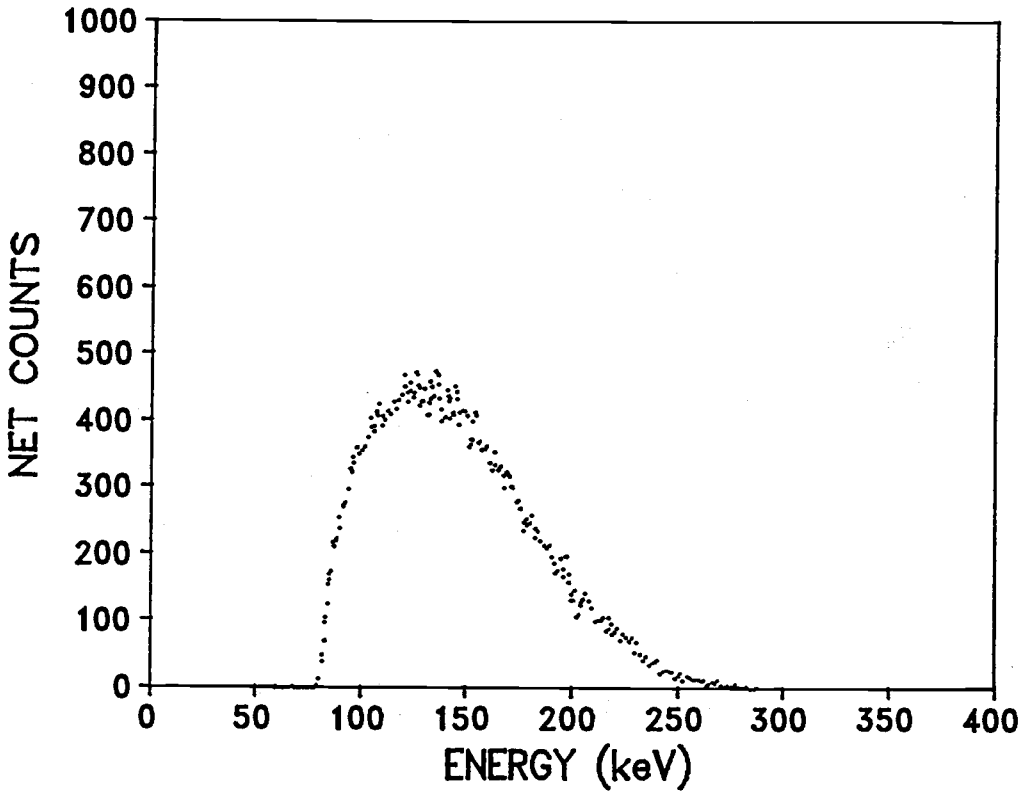
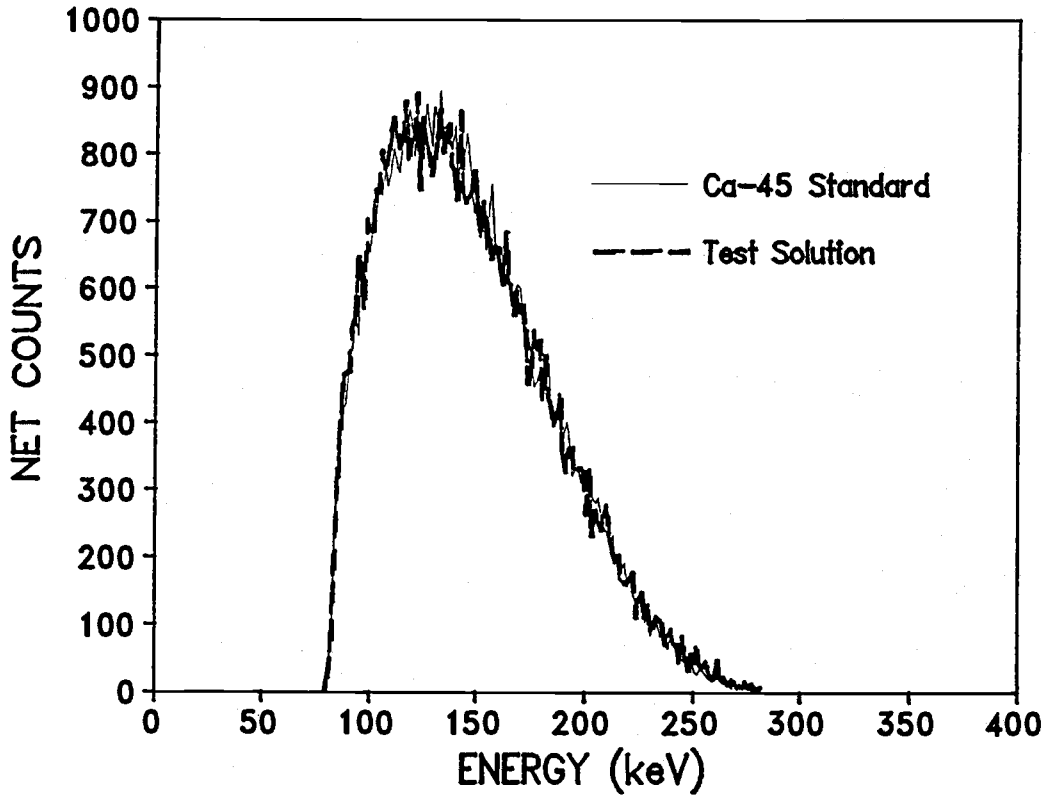


Figure 3.4

Comparison of the beta particle energy distribution of the Radiation Center's calcium-45 apple juice standard and the test solution, normalized to have the standard's activity level.



the Radiation Center's calcium-45 reference standard and the test solution plotted on the same graph. The beta spectrum from the test solution was normalized to the radioactivity level (0.14 μCi) in the Radiation Center's standard. The two spectra in Figure 3.4 appeared as one spectrum when they were normalized, thus confirming that no other beta particle emitters were present.

3.5 CONCLUSIONS:

Calcium-45 in apple juice samples can be easily and effectively identified using beta spectroscopy with active gamma-ray discrimination, and the radioactivity level can be easily quantified by liquid scintillation counting. However, due to the lack of readily available calcium-45 standards in the microcurie range which are traceable to the National Institute of Standards and Technology, it was difficult to achieve accuracy better than 10 to 20%. This is esspecially true when comparing sample radioactivity for this radionuclide in the 5 to 10 μCi range when such samples have been prepared by a source external to the user's program. Pipetting errors during preparation of reference sources may also have a significant effect on accuracy.

A quality assurance requirement that calcium-45 activities measured by different laboratories agree to within ±5% is not routinely achievable without access to better calcium-45 standards. Laboratories that use calcium-45 are therefore cautioned not to agree to quality assurance restrictions which cannot be easily met with existing technology.

3.6 NOTES:

* Opti-Fluor, Catalog 6013425.
Packard Instrument Company, Inc.
2200 Warrenville Road
Downers Grove, Il. 60515

† Beckman ß-mate I, Model 1661.
Beckman Instruments, Inc.
Scientific Instruments Division
2500 Harbor Blvd.
Fullerton, Ca. 92634

3.7 ACKNOWLEDGEMENTS:

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Chapter 4
CONCLUSIONS

This study demonstrated two applications of beta spectroscopy. Both applications used the potential of the beta spectrometer to provide useful information about beta energy distribution in practical situations.

In the first application, the beta spectrum from smears taken at various locations in a nuclear power plant were characterized. At locations where the beta spectrum was primarily influenced by activation products, the average beta energy at "contact" (1.5 cm) ranged from 450 to 550 keV. This energy range increased slightly at locations where fission products influence the beta spectrum. The average beta energy hardened about 300 keV at the normal working distance of 30.5 cm. The comparison of a composite beta spectrum from known standards to the spectrum from the fuel transfer canal smear indicated little or no contribution from pure beta emitters.

The average beta energies were higher than those measured in steam generators at the Sequoyah nuclear plant[12]. These differences could be attributed to the isotopic distribution. The Trojan smears were taken near the end of a 300 day outage, while the Sequoyah smears

contained an abundance of cobalt-58 and chromium-51 indicating that they were taken early in the outage. This information characterizing the beta spectra should be useful to health physicists in nuclear power plants who must calculate dose estimates and evaluate the shielding effects of protective clothing.

The second application used the beta spectrometer in a quality assurance program for a health research center. The radionuclide in the apple juice test solution was successfully identified as calcium-45 by comparison to commercially available calcium-45 reference material. It was difficult to achieve activity measurement accuracy better than 10 to 20% when comparing sample radioactivity for this radionuclide in the 5 to 10 μCi range. This was due to the accuracy of the calcium-45 standard. Obtaining $\pm$5% agreement in activity measurements is not routinely achievable without access to better calcium-45 standards. The health research center is administering the calcium-45 in apple juice if all of the activity measurements are within the 5 to 10 μCi range needed to conduct the study.

The National Institute of Standards and Technology (NIST) develops, produces,and distributes Standard Reference Materials (SRM) for science, industry, and government. Requests can be made to develop new standard

reference material. If there was sufficient demand, a calcium-45 standard could be issued. This would permit the radioisotope vendors to distribute calcium-45 reference material that has traceability back to a single standard.

With better reference material, a measurement quality assurance program described by E.H. Eisenhower[13] could be established. A calibrated transfer standard would be used to ensure that the spectrometer was functional before use. Valid comparisons to other activity measurements could be made. The information derived from this application of beta spectroscopy should be useful to researchers in nuclear medicine who are required to establish quality assurance programs to meet regulatory requirements.

These applications have demonstrated two practical uses of beta spectroscopy. Other uses, such as the development of a portable spectrometer to detect "hot particles" at nuclear power plants, need to be investigated further.

Chapter 5
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