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# A NEUTRON ACTIVATION ANALYSIS PROCEDURE DEVELOPED FOR USE AT THE MICHIGAN STATE UNIVERSITY NUCLEAR REACTOR LABORATORY

presented by

STEVEN A. LELEWER

has been accepted towards fulfillment of the requirements for

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A NEUTRON ACTIVATION ANALYSIS PROCEDURE
DEVELOPED FOR USE AT THE MICHIGAN STATE
UNIVERSITY NUCLEAR REACTOR LABORATORY

Ву

Steven Arthur Lelewer

# A THESIS

Submitted to
Michigan State University
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#### ABSTRACT

1,500

A NEUTRON ACTIVATION ANALYSIS PROCEDURE DEVELOPED FOR USE AT THE MICHIGAN STATE UNIVERSITY NUCLEAR REACTOR LABORATORY

By

#### Steven Arthur Lelewer

The purpose of this thesis is to describe the development of a method of performing neutron activation analysis at the Michigan State University Nuclear Reactor Laboratory. One requirement of the method was that it could be performed in a reasonable amount of time. spectrum analysis is a long and tedious procedure. Philip A. Baedecker developed a spectral analysis computer program entitled SPECTRA. Part of the project described in this thesis entailed modifying SPECTRA to operate on the Michigan State University CDC 6500 computer. SPECTRA requires that the spectrum data be input via punched cards. An interface was designed to allow the interconnection of the Nuclear Data multichannel analyzer and the engineering building IBM 1800 computer. Program ND was created to control the transfer of data from the analyzer to the IBM 1800, and to handle the formatting and punching of the data onto cards for input to program SPECTRA. A 2048

channel spectrum can be transferred from the Nuclear Data analyzer to the IBM 1800 in approximately 15 seconds.

The spectral analysis can be performed by SPECTRA at a cost of \$1 to \$2 on the campus computer.

The final chapter of the thesis provides a detailed procedure for transferring the data from the Nuclear Data analyzer to the IBM 1800, including cable hook-ups and the oppration of the IBM 1800 computer. The procedure also incudes instructions for the usage of program SPECTRA.

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#### INTRODUCTION

Neutron activation analysis (NAA) is a powerful laboratory technique for determining the elemental content of an unknown sample. NAA is a particularly valuable tool in that it is often possible to identify trace amounts of elements in quantities of parts per million or even parts per billion. When the unknown sample is neutron irradiated, radioactive isotopes are formed. Many of the isotopes thus formed decay by gamma emission. Examination of the energy spectrum of the gamma rays emitted will identify the elements contained in the unknown sample. A multichannel analyzer coupled with a gamma ray radiation detector is used to collect the spectral data of the irradiated sample. Elemental identification is performed by analysis of the energies of the photopeaks observed in the collected spectrum. Analysis of the photopeak height, sample irradiation time, counting time, time between irradiation and data collection, sample weights or volumes, and detector efficiency will allow the determination of the quantity of each element present in the unknown sample. Another feature of NAA is its insensitivity to chemical form of the irradiated sample.

In order to more fully understand NAA, a few basic concepts will be discussed. An atomic nucleus is only stable when a specific number of protons and neutrons are present. The number of protons in the nucleus determines the element's identity and the number of neutrons usually determines whether that nucleus is radioactive or non-radioactive (stable). (It should be noted that there are some elements that have no stable nuclei. In some cases, there are other differences that make nuclei radioactive.)

As a sample case, all sodium atoms contain 11 protons, while only those sodium atoms containing 12 neutrons are stable. A sodium atom with fewer or with more neutrons will be radioactive. There are elements that have more than one number of neutrons that result in stability; for instance, tin has ten stable isotopes.

Nuclei can absorb additional neutrons, resulting in the conversion of stable nuclei to radioactive ones. The radioactive nuclei decay in unique ways and emit radiations that are often distinct and can be measured even in very small quantities. The measurement of the radiations can determine the kind and the number of radioactive atoms that are present.

The neutron bombardment of a sample is performed in a nuclear reactor where the neutrons that strike the target atoms have been slowed down so that they have very little energy of motion. These free neutrons react with

the target nuclei resulting in the capture of the neutron and thus creating nuclei with atomic weights one unit greater than the original atomic weight. These newlycreated nuclei are typically radioactive.

Radioactive nuclei almost always decay by emitting negatively charged beta particles, usually accompanied by gamma rays. Each kind of radioactive atom decays with a unique pattern called a decay scheme. By measuring the radiation and identifying the pattern, the source of radiation can be determined. There are some problems in the measurement of gamma rays. These problems are due to the interaction of gamma rays with matter. Due to these interactions, the gamma rays measured by the detector no longer have the energy they started with at the time of emission. The most troublesome of the interactions is Compton scattering.

The Compton effect is a collision between a gamma ray and an electron. Compton scattering will generally involve the outer atomic electrons. When the gamma ray collides with the electron, it only transfers part of its energy. The gamma ray is degraded in energy and deflected from its original path. Compton scattering is the prevailing interference for measurement of gamma rays with energies greater than .5 MeV. Assuming that the radioactive isotope being detected has a 1 MeV gamma ray emission, due to the Compton effect, the detector will see gamma rays at 1 MeV

and a smear of gamma rays from 0 KeV to a value which is approximately 250 KeV less than the original gamma ray energy (for this case, 750 KeV). The end of the smear at 250 KeV less than the original gamma ray energy is called the Compton edge. This smear of gamma rays raises the background counts in the lower-energy gamma ray region. Therefore if low-energy gamma rays are being emitted from another radioactive isotope, the Compton effect from the 1 MeV gamma rays may mask them and make identification of the low-energy emitting isotope difficult. Interaction of gamma rays with matter may effect the sensitivity of the analysis, but the use of good gamma ray detectors can minimize the interference effects.

The applications for activation analysis seem unlimited. The only requirement for its use is that the element of interest must yield a gamma ray emitting isotope after irradiation. NAA has been used in the electronics industry to determine trace levels of impurities in semiconductors and the dopant levels in finished semiconductors. It has been useful in detecting trace amounts of oxygen in steel. NAA can be used to detect traces of catalyst residues in plastics. Determination of residual amounts of bromide in foodstuffs and on crops has assisted the agricultural industry.

NAA has provided a new tool for the forensic chemist. The results of activation analysis have been

admitted as evidence in a court of law. NAA can be used to trace a sample of paint, grease, tire rubber, etc. to its manufacturer by the quantities of trace elements present. It can provide a reliable method of identifying traces of gunpowder on a suspect's skin.

Another attractive use of NAA is the introduction of minor quantities of several easily activated elements as a secret coding to guard against counterfeiting of a product. This method of product identification is being considered for use in drugs, foodstuffs, cement and legal tender.

The topic of this paper will be the development and usage of a computerized method of neutron activation analysis utilizing the Michigan State University Triga Mark I nuclear reactor, a Nuclear Data multichannel analyzer, an IBM 1800 computer and the Michigan State University CDC 6500 computer. Prior to the development of this procedure, NAA was performed by manual techniques. A complex spectrum could take many hours to complete. Utilizing the methods described in this paper, spectrum analysis can be completed in a few minutes with a computer usage cost of a few dollars. Included in this paper will be a discussion of the electronic interface developed to allow data transfer from the Nuclear Data multichannel analyzer to the IBM 1800 computer, a discussion of the computer program developed to control the data transfer

and to present the data on computer punch cards, and a discussion of the spectral analysis computer program developed for the CDC 6500 computer. The final topic of this paper will be a user's manual giving instructions on performing NAA utilizing the facilities available at Michigan State University.

#### THE INTERFACE AND THE ND PROGRAM

Prior to the design of the interface and the development of the ND program, there were only two forms of data output for the Nuclear Data analyzer. The first, a pen plotter, provided rapid data output. Its disadvantage was that when peak height analysis was performed by calculating the area beneath the peak, the result was quite The second form of output available was through inaccurate. a Teletype with a paper tape punch. The Teletype output allowed peak height analysis with a high degree of accuracy, but to output a 2048 channel spectrum required approximately 30 minutes. Therefore, the first step necessary for a project that involved experiments using the Nuclear Data multichannel analyzer to count many samples was to find a method of data output that was both rapid and accurate. A bid request for an interface to connect the Nuclear Data analyzer to the IBM 1800 computer was sent to IBM and to Nuclear Data. The cost reported was in excess of the budget for the project. It was decided at this point that the interface design was to be performed in-house.

To develop the interface, the following information had to be ascertained:

- (a) location of an output port on the Nuclear Data analyzer where memory bank data was available in some binary format;
- (b) the binary format of the data at the output port (i.e.: is the data in pure binary (base 2) or in 1-2-4-8 binary coded decimal, where any four bits (on-off switches) represents one decimal digit. In four bits, "0" through "9" can be represented);
- (c) voltages at the output port which represented 0 and 1:
- (d) location of an input port on the Nuclear Data analyzer where the memory bank data at the output port could be advanced;
- (e) voltage needed at the input port to trigger the data to advance as described in (d):
- (f) location of an input port on the IBM 1800 where the data could be received;
- (g) the voltages required at the input port to
  represent 0 and 1;
- (h) the location of an output port on the IBM 1800 where an advance signal could be generated to trigger the data advance;
- (i) voltages generated at the output port for 0 and 1 representation.

Many hours were spent researching the operation and maintenance manuals for both the Nuclear Data analyzer and the IBM 1800. This research, coupled with extensive experimentation, yielded the necessary information.

It was found that at the connector labeled "Printer A" on the Nuclear Data analyzer, the memory bank data could be read in 1-2-4-8 binary coded decimal form with voltages pulled down to within 0.5 volts of ground for "zero" signals and up to +6 volts or floating for "one" signals. It was also discovered that the memory bank could be advanced by simulating a "complete" signal of +12 volts. This was the voltage that the plotter generated to indicate that data had been received and that the next channel of data should be prepared for transfer. The digital inputs or "DI's" on the IBM 1800 were determined to be the best port for data input and the digital outputs (called "DO's" or "ECO's") were found to be the source for generation of the trigger signal. It was determined that for the IBM 1800, a zero was represented by -12 volts (with a range of -6 to -30 volts), and a one by 0 volts (with a range of -1 to +30 volts).

An electronic interface was required to adjust the voltages to conform with the needs of the two devices being connected. The Michigan State University Department of Engineering Research Computer Laboratory developed an interface box powered by the Nuclear Data analyzer NIM bin that provided the required voltage conversions (see Figure 1). It is important to note here that the voltage

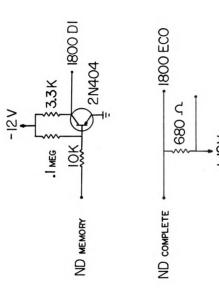


Figure 1.--Wiring Schematic for Interface.

conversions performed by the interface only shift the voltages; they are not reversed. Therefore a "1" generated by the ND analyzer, with voltage shifted by the interface, yields a "0" at the IBM 1800, and a "0" generated by the ND analyzer, with voltage shifted by the interface, yields a "1" at the IBM 1800. The correcting inversion is performed by program ND as the data is transferred.

Once the hardware was developed to enable the IBM 1800 and the Nuclear Data analyzer to communicate with each other, it was necessary to create a program to control the transfer of data from one device to the other. Program ND was written to perform this function. (A listing of ND is provided in Appendix A). Program ND is an interactive program written in Fortran IV. The program elicits information from the user, such as the number of data channels to be transferred and whether or not a line printer listing of the spectrum is desired. The program also instructs the user, with commands to load the card hopper when necessary and to make sure the ND analyzer is properly prepared for data transfer.

Program ND consists of seven parts. The first of these parts is the main program. The main program controls the operation of the rest of the code. The responses of the user to the questions asked by the computer control the title of the experiment, the run number and the form

of output. The main program calls upon the six subprograms listed below:

- (1) Subroutine PRINT is called when the user requests a listing of the data transferred. This listing is outputted to the line printer. Subroutine PRINT converts the binary data to the equivalent character code and fills in leading blanks instead of zeros as needed (when the ND analyzer sends the data in a channel, six digits are transmitted. A count of 900 is sent as 000900). The program also prints the number of the first channel in each row of data listed.
- (2) Subroutine PPUNC is called when the user requests a deck of cards containing the data transferred. The deck is punched in binary coded decimal form. A BCD deck contains ten channels of data on each card. Each column on the card contains either a blank or one numerical character. Subroutine PPUNC performs the same conversion from binary that subroutine PRINT performs, but no "first channel" is punched on the cards.
- (3) Function MINO is called by the main program to calculate which of two numbers is the minimum.

(Note: The following three subroutines are written in IBM 1800 Assembly language. Because of the necessity to do bit manipulation, the use of Assembly language is more practical and efficient than the use of Fortran.)

- (4) Subroutine DI is called by the main program to read the digital inputs from the ND analyzer and to send the "complete" signal to the ND analyzer to advance the memory stack. Subroutine DI also performs the conversion of the input bits from 0 to 1 and 1 to 0. This routine also loads the 1-2-4-8 BCD data into a special portion of the IBM 1800 computer memory called "COMMON," converts the 1-2-4-8 BCD data digits to binary and loads the binary data into "COMMON" also.
- (5) Subroutine ECO is called by the main program to ensure that the digital output port is initialized prior to the ND analyzer being turned to the "Read in/out" mode. This is required so that the first channel of data is not missed.
- when the user requests a "crunch" deck. A crunch deck contains 40 channels per card. The data is in binary form. Each column on the card contains 12 bits of information. Since each word or channel of data requires a maximum of 16 bits when converted to binary, each word requires  $1\frac{1}{3}$  columns. In other words, every four columns contains three data channels. This format is similar to the "crunch" format used in the MSU Cyclotron Laboratory. The CDC 6500 remote input station located in the engineering building is not capable of transmitting binary data to the computer. The spectrum analysis routine, SPECTRA,

available on the CDC 6500 is not provided with the ability to handle the "crunch" decks. The "crunch" feature is provided here in Program ND, but further development of Spectra would be necessary in order to make use of this feature.

#### **SPECTRA**

Program SPECTRA is used to analyze the data decks generated by the IBM 1800 and program ND. Spectra was originally created by Philip A. Baedecker. A description and listing of the program is provided in Appendix C. Spectra was then brought to the Michigan State University Cyclotron Laboratory and modified to operate on the Sigma 7 computer. This conversion was performed by Dr. Charles Spooner of the Geology department along with members of the cyclotron staff. The original Spectra was designed to accept tape input of the spectrum. The cyclotron version of Spectra utilized a compressed binary format ("crunch") deck of data cards for input. To allow use of Spectra with the data generated by the IBM 1800 computer and program ND, the computer code was modified with the assistance of Robert Morris, a computer science student at MSU. As a result of this modification, campus-wide use of Spectra through the CDC 6500 computer became The CDC 6500 version of Spectra is designed to possible. accept BCD decks of input data as provided by the IBM 1800 and program ND. A listing of the CDC 6500 version of Spectra is provided in Appendix B.

Spectra is a computer program that analyzes gamma ray spectrum by searching out photopeaks, calculating the area under the peaks, finding the centroid of each peak and providing the energy corresponding to each peak. In activation analysis runs, when quantitative information is required, the program provides either the concentrations or the quantity of the elements in the unknown samples. Control of the program is accomplished when the user places certain control cards in front of the data decks to be analyzed. A detailed set of instructions for use of Spectra can be found in Chapter 4, the User's Manual.

The following procedure outlines the method by which the program analyzes the data. After reading in the control cards, the program reads in a data deck. The data can be smoothed by a five-point convolution method. Since smoothing can cause undershoot of the baseline on either side of very narrow peaks, smoothing should be avoided for spectrum with 1024 channels or fewer, but for spectrum with 2048 channels or more, smoothing helps to eliminate statistical fluctuations that might be confused with actual photopeaks.

The program then performs a series of operations to locate peaks. The first of these operations is a five-point convolution method of calculating first derivatives for each channel. Then, by looking for sign changes in the first derivatives, the program locates possible peaks.

The program assigns the minimum on each side of the peak as the peak limits or boundaries. The program then checks to verify that the right hand boundary channel for the peak is not too far out. By comparing the left side derivatives to the right side derivatives, the right hand boundary channel is fixed. A baseline calculation is performed next, and then a few statistical tests of peak validity are done. The first test for a valid peak involves consideration of the following criterion:

where peak area is total area minus base area, and SIGMA represents the standard deviation of the peak. The calculation of SIGMA is as follows:

SIGMA = 
$$\sqrt{\frac{R}{L}} D_{i} + (D_{L} + D_{R}) \times \left[ \left( \frac{R-L-1}{2} \right)^{2} - 1 \right]$$

where R represents the right hand boundary channel number, L represents the left hand boundary channel number,  $D_L$  represents the number of counts in the left hand boundary channel,  $D_R$  represents the number of counts in the right hand boundary channel, and  $D_i$  represents the number of counts in the i<sup>th</sup> channel.

The next test is used to eliminate Compton edges.

The slope of the left side of the peak is compared to the slope of the right side of the peak. If the absolute

values of the slopes are within a factor of 1.5, the peak is passed to the baseline calculator. By inspection of Figure 2, one can see that a Compton edge would not pass the aforementioned "1.5" test. Four channels to the outside of the peak boundaries are averaged, with those channels whose count is more than two standard deviations from the peak boundary channel count being disregarded. The right hand and left hand averages are taken as the counts in the boundary channels. The right hand and left hand boundary channels are straight-lined and a baseline is determined. The baseline is defined as the background level. Finally, the centroid of the peak is calculated.

The user has the option of two different methods of determining peak intensity. The "Total Peak Area" method yields a peak intensity equal to the sum of the counts in the channels bounding the peak as determined by the statistical tests described above. This method uses the redefined counts in the boundary channels for subtracting out background. The second peak intensity method available is the "Wasson" method. In this method, the user specifies the number of channels to either side of the center channel of the peak to be used as the limits of integration. A baseline is determined as described above, but with the boundary channels as defined by the user, and the background is then subtracted out.

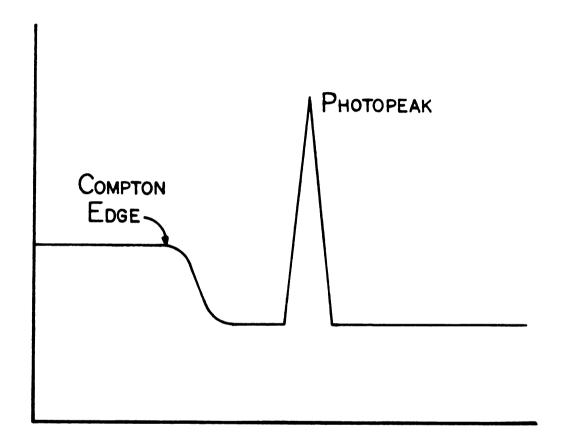


Figure 2.--Typical Photopeak and Compton Edge.

In order to calibrate the spectra (i.e.: assign energy values to each channel), reference and standard spectra must be provided to the program. First, a rough calibration is performed by using a <sup>137</sup>Cs spectrum. has a single peak located at 661.7 KeV. If photopeaks greater than 1836 KeV are to be located, a 88 y spectrum is also necessary, to assist the program in compensating for possible nonlinearity in the spectrometer. The reference spectra addressed above may be deleted if the user supplies the program with the centermost channels for  $^{137}\mathrm{Cs}$  and  $^{88}\mathrm{Y}$ . Standard spectra are used next to give a precise calibration to the spectra. The user supplies the program with the energy levels of the gamma-rays in each standard spectrum. The program assigns the centroid of each peak located in the spectrum with the corresponding energy. Then the program determines energies for the photopeaks of the "unknown" sample spectra by interpolation assuming linearity between standard spectrum peaks. The program will not extrapolate energies above the highest standard peak supplied by the user. This completes the qualitative analysis portion of Spectra.

Spectra is also capable of performing activation analysis calculations (or quantitative analysis). A flux monitor (i.e.: sample containing a known quantity of the element of interest) must be irradiated in the reactor in the same batch as the "unknown" samples. The user must

provide the program with the gamma ray energy of the element of interest, the chemical symbol, the half life of the isotope and the weight or concentration of the flux monitor sample. The user must also supply name of sample, weight of sample, time of day sample was counted and live time duration of count for each spectrum used in the quantitative analysis, both "unknowns" and "flux monitors."

The program then uses the flux monitor spectra to calibrate each peak of interest.

The "unknown" sample spectra are then analyzed for each peak of interest. Each peak is compared to the associated flux monitor peak and from sample weights the concentration of each element of interest is determined. In order to account for decay and live time the following formulae are used:

Formula 1: SCF = 
$$\frac{\text{(peak area)} \times \lambda e^{-\lambda t} 1}{\text{(flux monitor wt.)} \times (1 - e^{-\lambda t} 2)}$$

where SCF is the standard comparator factor or the calibration of the flux monitor peak,

 $\lambda$  is the decay constant for the isotope of interest,

t<sub>1</sub> is the time elapsed between some reference time (e.g.: time of irradiation or time of first count in this analysis) and the start time of the counting of this spectrum, and

t<sub>2</sub> is the duration of the count (live time).

Formula 2: ACF = 
$$\frac{\text{(peak area) } \times \lambda e^{-\lambda t} 1}{\text{(sample wt.) } \times (1 - e^{-\lambda t} 2)}$$

where ACF is the analytical comparator factor, or the weighted intensity of the "unknown" sample peak of interest, and  $\lambda$ ,  $t_1$  and  $t_2$  are as described above, but evaluated for the "unknown."

For a more detailed discussion of Spectra, see Appendix C.

#### USER'S MANUAL

This chapter describes the procedures for transferring data for the ND analyzer to the IBM 1800 and for operating program Spectra. To transfer data to the IBM 1800, the ND analyzer, the "interface" and the IBM 1800 must be connected. The IBM 1800 must be cold started and program ND must be run. Once the data has been collected on data decks, the controls for Spectra must be prepared and finally Spectra is run. The procedure for these operations follows.

### Data Transfer Hook-Up (See Figure 3)

- (1) The following connections must be made:
  - (a) The interface must be in a nimbin:
  - (b) The cable from the back of the interface must be connected to the ND analyzer at "Printer A":
  - (c) The female single pin connection in the back of the interface must be connected to the ND analyzer at the point marked "complete";
  - (d) The cable from the IBM 1800 must be attached to the face of the interface;

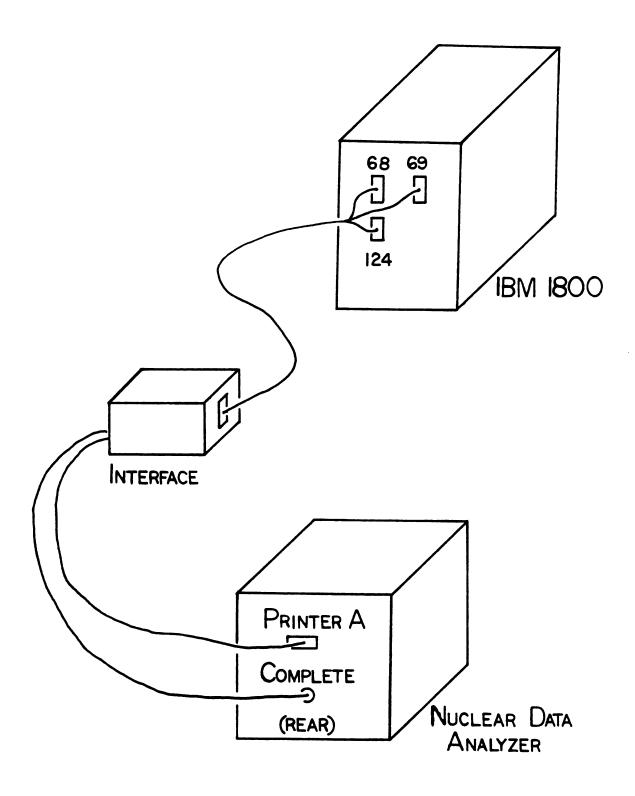


Figure 3.--ND Analyzer--Interface--IBM 1800 Cable Hook-Ups.

(Note: the following connections are in the IBM 1800 room.)

- (a) Connect cable connectors marked "ND-68" and "ND-69" to the IBM 1800 at the digital inputs marked "68" and "69";
- (b) Connect cable connector marked "ND-124" to the IBM 1800 at digital output "124".

#### Cold Start of the IBM 1800

- (1) On console--push POWER ON.
- (2) On printer--push START.
- (3) Place disk marked "Reactor Lab" in disk drive (middle slot).
- (4) On disk drive--push START.
- (5) Wait for READY light on disk drive.
- (6) Place cold start card in hopper.
- (7) On console--push IMMEDIATE STOP.
- (8) On console--push and hold CLEAR STOR.
- (9) On console--push START.
- (10) On console--release START and CLEAR STOR.
- (11) On console--push IMMEDIATE STOP.
- (12) On card reader--push START.
- (13) On console--push PROGRAM LOAD.
- (14) Wait for cold start pattern. The cold start pattern appears on the operating console. Most of the small white lights will be lit except for four lights forming a square near

the center of the light panel. These are the lights corresponding to columns 9 and 10 in the adress register and I register rows.

#### To Run ND

(1) Prepare the following deck: (all cards begin
 in col. 1).

//JOB

/// XEQ ND FX (Note: FX begins in col. 16)
blank card
blank card

- (2) Place deck in hopper.
- (3) On card reader--push START.
- (4) ND is self-explanatory. The program will request information from the user on the IBM selectric terminal in the IBM 1800 room.

## Preparing to Run Spectra

Note: For each "card" a format is prescribed.

There are four types of formats, "A", "I", "F", and "X".

Formats "A" and "I" have the following structure: nAm or nIm. The value of "n" gives the repetition number (e.g.: 3A2 is the same as A2, A2, A2). The value of "m" gives the number of characters allowed in the input field. The "A" format allows the input of alphanumeric characters (e.g.: A4 allows the input of TEST). The "I" format is used to input numeric data in integer form (whole numbers). Note that data in an "I" field must be right justified.

If the data to be inputted in an I6 field is three digits

long, the three digits must be preceded by three blanks (e.g.: 126 in an I6 field must be bbb126, where b represents blank). Format "X" has the following structure: nX. The "X" indicates a blanking format, where "n" is the number of blanks. The "F" format has a structure of: nFm.p where "n" is the repetition number, "m" is the total number of characters allowed in the input field, and "p" is the maximum number of digits to the right of the decimal point. The "F" format is used to input numeric data that is not whole number data. Note that the data need not be right justified in the input field, and that "m" includes the decimal point (e.g.: 125.89 requires a format of F6.2).

In order to run Spectra, the following deck must be prepared:

#### Card 1--Format 416

- columns 1-6 (1) Number of standardizing spectra (including Cs and Y standards, if provided)
- columns 7-12 (2) Maximum energy of standard peaks (in Kev).
- columns 13-18 (3) Cs peak channel number (if spectrum provided, input "0").
- columns 19-24 (4) Y peak channel number (if not used, input "0").

(Note: If max. energy of standards is greater than 1836.08 KeV, a Y spectrum or channel number is required.)

Card 2--Format 12I6 (Multiple cards using the same format may be required, depending on the number of standard spectra used.)

Cards with number of peaks in each standard.

(Sequence must correspond with order in which standard spectrum are run. Not needed for Cs or Y data).

Card 3--Format 8F9.3 (Multiple cards using the same format may be required, depending on the total number of peaks in the standard spectra used.)

Cards with energies of peaks in each standard.

(In the order in which they appear in the decks, i.e.: min. to max. for each spectrum in order.)

Card 4--Format 316 and then 13A4

column 6

- (1) "0"
- columns 7-12
- (2) Number of channels in spectra
- columns 13-18
- (3) Number of spectra to be analyzed (including flux monitors, but not standards)
- columns 19-70
- (4) Labels (run name). A maximum of 52 alphanumeric characters are allowed.

Card 5--Format 4Il and then 1I6

column 1

- (1) 0--raw data printed out
  - 1--raw data not printed
     out

column 2

- (2) 0--smoothed data printed out
  - 1--smoothed data not
     printed out
  - 2--smoothing operation omitted

column 3

- (3) 0--peak area by Wasson's method
  - 1--peak area by total peak area method

column 4

columns 5-10

(5) Integer number of points on each side of the center channel, used for peak area.

(Note: If card 5 col. 4 = 1, omit card 6 and those following.)

#### Card 6--Format 316 and then A4

columns 1-6

- (1) Number of flux monitor spectra. (Note: multiple flux monitors have to have identical concentrations or quantities, only in different locations in reactor core during irradiation. The program does not accommodate different concentration flux monitors. Spectra merely averages the peak areas in multiple flux monitors.)
- columns 7-12
- (2) Number of peaks to be analyzed.
- columns 13-18
- (3) Amount of error in peak find--approximately three KeV (as recommended in the Baedecker write-up, see Appendix C.)

columns 19-22 (4) Four alphanumeric characters that identify the flux monitor spectra.

Card 7--One for each peak analyzed

- columns 1-2 (1) Format A2--Chemical symbol being determined
- columns 3-10 (2) Format 8X--Space 8
- columns 11-20 (3) Format F10.3--Energy of peak in KeV.
- columns 21-23 (4) Format 3X--Space 3
- columns 24-30 (5) Format F7.3--Half-life
- columns 31-34 (6) Format 4X--Space 4
- column 35 (7) Format Al--Units of halflife (S,M,H,D,Y)
- columns 36-40 (8) Format 5X--Space 5
- columns 41-50 (9) Format F10.5--Flux monitor weight or concentration
- columns 51-56 (10) Format 6X--Space 6
- columns 57-60 (11) Format A4--Units of flux monitor weight or concentration (µg, mg, etc.)

Card 8--One for each spectrum in deck (to be analyzed for activation analysis)

- columns 1-16

  (1) Format 4A4--Sample name
  (The program requires
  that the first four characters must be the same
  as in col. 19-22 if
  spectrum is to be treated
  as a flux monitor.)
- columns 17-20 (2) Format 4X--Space 4
- columns 21-30 (3) Format F10.5--Sample weight

- columns 31-36 (4) Format 6X--Space 6
- columns 37-40 (5) Format A4--Units of weight (µg, mg, etc.)
- columns 41-45 (6) Format 5X--Space 5
- columns 46-48 (7) Format I3--hours, time of count, 2400 hour clock, if next day, add 2400
- columns 49-50 (8) Format I2--Minutes
- columns 51-53 (9) Format 3X--Space 3
- columns 54-59 (10) Format I6--Live time duration of count
- column 60 (11) Format Al--Units of time
- columns 61-64 (12) Format 4X--Space 4
- Column 70 (13) Format I6--"0"

Follow cards 1-8 with the data decks from the IBM 1800 as follows:

- (1) Cs spectrum if used;
- (2) Y spectrum if used;
- (3) Standard spectra;
- (4) Flux monitor spectra if doing activation analysis;
- (5) Unknown spectra.

(Note: Place 789 multipunch card at the end of each spectrum and place 6789 multipunch card at end of deck.)

### Running Spectra

(1) Prepare the following deck (each card begins
 in col. 1)

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JOB CARD (Be sure to specify MTl to mount tape)

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HAL, BANNER, SPECTRA.

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789

(2) Follow this deck with the Spectra cards as described above.

BIBLIOGRAPHY

#### **BIBLIOGRAPHY**

# General References

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- 3. Gardner, R.P. and Ely, R.L., Jr. Radioisotope Measurement Applications in Engineering. Reinhold Publishing Corp. New York, N.Y., 1967.
- 4. Grouthamel, C.E., Adams, F. and Dams, R. Applied
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# APPENDIX A

A LISTING OF PROGRAM ND

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# APPENDIX B

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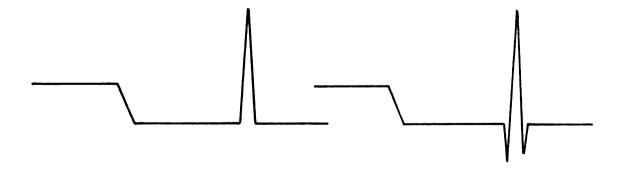
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## APPENDIX C

A DESCRIPTION AND LISTING OF THE PROGRAM SPECTRA by Philip A. Baedecker

The program SPECTRA is a program for the analysis of gamma-ray spectra. The program searches out photopeaks, calculates the area under the photopeak, determines the centroid of the photopeak, and, when spectra of gamma-ray standards are included on the input tape, determines the energies of the photopeaks. In an activation analysis experiment, the program will calculate concentrations of the elements to be determined.

The basic program analyzes a spectrum in the following way. The spectral data are read into the computer from magnetic tape, and the data can then be smoothed by a five point convolution technique, using the method of Savitsky and Golay (Anal. Chem. 36, 1627 (1964)). This smoothing operation may be bypassed however, since, for very narrow peaks, the smoothing operation may cause the spectrum to undershoot the baseline on either side of the peak.



In general, for 1024 channel Ge(Li) spectra, it is best to avoid smoothing; for 4096 spectra smoothing is often desirable to help eliminate fluctuations that might be recognized as peaks by the program.

After smoothing the program uses a five-point convolution technique to determine the first derivative at each channel. The program then locates possible peaks by determining where the first derivative changes sign from positive to negative. The program, having located a possible peak, determines the minimum on each side of the peak as the peak limits. Since there is some danger that the right boundary may be too far out, the routine tests the first derivative of each channel on the right hand side of the peak against the first tangent with a negative slope on the left. If the left hand tangent has a slope X, the program locates the first channel on the right with a tangent which has slope -X. If this channel number is less than the previously fixed right boundary, the boundary is considered to be the newer lower channel number.

The program then draws a linear baseline between the boundary channels and checks the right side of the peak to see if any channel falls below the baseline. If this occurs, the boundary channel is decreased by one and the operation repeated until all channels of the right hand side are above the baseline. A similar process is then carried out on the left hand side of the peak.

The next section of the program tests the peak to decide whether or not it is a valid peak. The first test is a statistical test. The peak area and base area are calculated as follows:

Base Area = 
$$0.5x(D_1 + D_r)x(R - L + 1)$$

where

 $D_1$  = counts in left hand boundary channel (L)

 $D_r = counts$  in right hand boundary channel (R)

Peak Area = 
$$\sum_{i} D_{i}$$
 - base area

A standard deviation of the peak is then calculated.

SIGMA = 
$$\sqrt{\frac{R}{L}} D_{i} + (D_{1} + D_{r})x (\frac{R - L - 1}{2})^{2} - 1$$

A statistical test is then applied to the peak. To be valid a peak must meet the following criterion:

Peak Area 
$$> (5.0 \times SIGMA)$$

The primary purpose of the next test is to eliminate Compton edges, but it is also useful in eliminating spurious peaks which get by the first test. The maximum first derivative is located on the left side of the peak, the minimum derivative on the right. This gives a measure of the slope at the point of inflection on each side of

the peak. The absolute values of the slopes are then compared to see that they are the same within a factor of 1.5.

After a peak has passed the previous two tests, a linear baseline is defined for the photopeak. Several channels on each side of the peak are used to define the background. Four channels to the left of the left peak limit are examined. Those channels which have the same total accumulated counts as the left hand boundary channel (within two standard deviations) are averaged and this average is taken as a new value for the counts accumulated at the left hand peak limit and used to define the background. The process is repeated on the right hand side of the photopeak. The baseline is then a straight line drawn between the two boundary channels.

The centroid of the peak is determined next using seven channels (three on each side of the channel with the greatest number of counts) using the method of Savitzky and Golay.

Two methods for measuring the intensity of the peak are built into the program, to be selected by the user. These methods are discussed in Appendix A, where they are referred to as the "Total Peak Area Method" and the "Wasson Method." In the total peak area method, the area is calculated as for the statistical test above, but using the redefined values for the boundary channels. In

the Wasson method a number of channels, specified by the user, are taken as the limits of integration. The baseline is determined as above, and using that baseline, background is subtracted from the channels within the selected limits for integration. The peak area is calculated as:

Base Area = 
$$(B_1 + B_r) \times (X + 0.5)$$

Peak Area = 
$$\begin{bmatrix} 1+X \\ \Sigma \end{bmatrix}$$
 D<sub>i</sub> - base area 1-X

where

1 = centermost channel

X = number of channels on each side of centermost channel to be included in peak area determination

D; = number of counts in ith channel

B<sub>1</sub> and B<sub>r</sub> = calculated values for the background
 in channels 1-X and 1+X respectively, computed
 from a straight line drawn between the peak
 limits

SIGMA = 
$$\sqrt{\frac{1+x}{1-x}}$$
 D<sub>i</sub> + (B<sub>1</sub> + B<sub>r</sub>) x (x + 0.5)<sup>2</sup>

The program will determine the energies of the photopeaks from their centroids, provided spectra of gamma-ray standards are included on the input tape. If the energies are to be determined, the first spectrum on the tape should be a Cs spectrum. This spectrum serves as

a reference spectrum to provide an approximate energy calibration for locating peaks of known energy in the standard spectra. Due to possible nonlinearity in the spectrometer, Y must be provided as a second reference spectrum if any of the photopeaks used as standards have energies greater than 1836 Kev (the Cs and Y reference spectra may be deleted providing the centermost channels for the Cs (and Y) peaks are included as input data on the first data card).

Following the reference spectra on the tape, up to 20 spectra of standards may be used to obtain the energy calibration of spectrometer. The energies of the gamma-rays in each standards spectrum are read in on data cards and the corresponding centroids are determined. Up to 50 lines may be used in the standards spectra.

The program determines the gamma-ray energies in all subsequent spectra by interpolation, assuming that the energy calibration is linear between standard lines. The program does not extrapolate beyond the highest energy of the gamma-ray standards.

When the program is used to process spectral data from an activation analysis experiment, the energies of the gamma-ray lines to be used in the analyses are read in on one set of data cards. Along with the gamma-ray energy, the chemical symbol of the element to be determined, the half-life of the radioisotope used and the weight (or

concentration) of the element in the flux monitor are included on the data card. A second set of data cards (one card for each spectrum on the tape) provides the following information: (1) the name of the sample; (2) the weight of the sample; (3) the units in which the sample weight is expressed; (4) the time of day the sample was counted; (5) the live-time duration of the count. If the weights of the elements in the flux monitors are constant from flux monitor to flux monitor, they may be entered in the appropriate field on the first set of data cards, and no sample weights entered for the flux monitors in the second set of data cards. If the flux monitors used are standard powders or solutions, the concentrations of the elements in the standard powder or solution may be placed on the first set of data cards, and the weights of the standard powders or solutions entered in the sample weight field on the second set of data cards.

The program first makes a pass through the spectra on the tape and analyzes the flux monitor spectra. It picks out the peaks of interest in each flux monitor spectrum and calculates the "standard comparator factor" for each peak. After all flux monitor spectra have been analyzed (the program can process up to ten), the standard comparator factors for each peak are averaged to yield an "average standard comparator factor" for each peak, which

is used to calculate concentrations when the sample spectra are analyzed.

The tape is now backspaced to the first spectrum for the activation analysis run under consideration (there may be more than one run on a given reel of tape). Each sample spectrum is then analyzed in turn, an "analytical comparator factor" calculated for each peak of interest, and, from the average standard comparator factors previously calculated, concentrations are calculated for the elements of interest in the sample. The standard deviation for the concentration is calculated based on counting statistics alone.

In an activation analysis experiment involving the counting of short-lived nuclides, one flux monitor may be irradiated and counted for each sample analyzed. The program will handle this as a special case, as follows: in an activation analysis run a card is provided which tells the computer how many flux monitor spectra are in a given run. If this number is put to less than zero, the program expects that every other spectrum will be a flux monitor spectrum, and that each sample spectrum on the tape.

If, for some reason, no flux monitors were counting during an activation analysis run, the number of flux monitor spectra can be put equal to zero on the data card

and the program will process the spectra and calculate analytical comparator factors for each peak in each sample spectrum.

In calculating standard comparator factors and analytical comparator factors, the program corrects the counting data for decay during the count and corrects each count for decay back to the time of the start of the first count in the activation analysis run. That is:

SCF = 
$$\frac{\text{(peak area)} \times \lambda e^{\lambda t_1}}{\text{(flux monitor weight)} \times (1 - e^{-\lambda t_a})}$$

ACF = 
$$\frac{\text{(peak area)} \times \lambda e^{\lambda t_1}}{\text{(sample weight)} \times (1 - e^{-\lambda t_a})}$$

where

t<sub>1</sub> = the elapsed time between the start of the first count in the experiment and the start of the count being processed

t<sub>a</sub> = duration of the count (live time)

The program operates on various different levels of complexity, determined by the input data cards. It can simply read spectra and analyze the spectral data. On another level the program will determine the energies of the photopeaks in the spectra, provided appropriate standard spectra are provided. Thirdly, the program can process spectral data from an activation analysis experiment and calculate element concentrations. Some output

Table 1.--Gamma Rays Used as Primary Energy Standards.

Source	Energy	(keV)	Source	Energy (keV)
241 <sub>AM</sub>	59.536	± 0.001	192 <sub>Ir</sub>	468.060 ± 0.010
109 <sub>Cd</sub>	88.034	± 0.010	Annihilation	511.003 ± 0.002
182 <sub>Ta</sub>	100.106	± 0.001	207 <sub>Bi</sub>	569.690 ± 0.030
<sup>57</sup> Co	122.046	± 0.020	208 <sub>T1</sub>	583.139 ± 0.023
144 <sub>Ce</sub>	133.503	± 0.020	192 <sub>Ir</sub>	604.378 ± 0.020
57 <sub>Co</sub>	136.465	± 0.020	192 <sub>Ir</sub>	612.430 ± 0.020
<sup>141</sup> Ce	145.442	± 0.010	137 <sub>Cs</sub>	661.615 ± 0.030
182 <sub>Ta</sub>	152.435	± 0.004	54 <sub>Mn</sub>	834.840 ± 0.050
<sup>139</sup> Ce	165.852	± 0.010	88 <sub>Y</sub>	898.023 ± 0.065
182 <sub>Ta</sub>	179.393	± 0.003	207 <sub>Bi</sub>	1063.635 ± 0.040
182 <sub>Ta</sub>	222.110	± 0.003	60 <sub>Co</sub>	1173.231 ± 0.030
212 <sub>Pb</sub>	238.624	± 0.008	22 <sub>Na</sub>	1274.550 ± 0.040
203 <sub>Hg</sub>	279.179	± 0.010	60 <sub>Co</sub>	1332.508 ± 0.015
192 <sub>Ir</sub>	295.938	± 0.010	140 <sub>La</sub>	1596.200 ± 0.040
192 <sub>Ir</sub>	308.440	± 0.010	124 <sub>Sb</sub>	1691.022 ± 0.040
192 <sub>Ir</sub>	316.490	± 0.010	88 <sub>Y</sub>	1836.127 ± 0.050
131 <sub>I</sub>	364.491	± 0.015	208 <sub>T1</sub>	2614.708 ± 0.050
198 <sub>Au</sub>	411.792	± 0.008	24 <sub>Na</sub>	2754.142 ± 0.060

options are included in the program in that the user can tell the program whether or not to print out the raw spectral data and whether or not to print out the smoothed data.

The following data cards must be provided by the user.

## Format Card Card with number of reference and stand-1 416 ards spectra on tape, and the maximum gammaray energy of standard lines. The centermost channels of the Cs (and Y) peaks may be included on this card if the gamma-ray energies are to be determined and the Cs (and Y) reference spectra are left off the input tape. IF THE NUMBER OF STANDARDS SPECTRA IS ZERO, OMIT CARDS 2 AND 3. Cards with the number of photopeaks in 1216 2 each standards spectrum. 3 Cards with the energies of the photo-8Fq.3 peaks in each standards spectrum in the order that the spectra appear on the tape. Card with number of spectra to be 316, 4 skipped, size of spectra, number of 13A4

spectra (not including reference and

Card

standard spectra or any spectra to be skipped following the first spectrum in an activation analysis run), and labels.

5 Card which controls various options built 4I1,I6 into program. The first four columns control the operation of the program.

Column 1--zero-raw data printed out

one-raw data not printed out

Column 2--zero-smoothed data printed out

one-smoothed data not printed out

two-smoothing operation deleted

Column 3--zero-peak area integrated by Wasson's method

one-peak integrated to give total peak area

Column 4--zero-program will calculate
elemental concentrations
from activation analysis
data

one-activation analysis program bypassed

Column 5--10--integer number of points on
each side of center channel
to be included in peak area
determined by Wasson's method

<u>Card</u> Format

Cards 4 and 5 may be repeated together any number of times. If there is more than one run per tape, place a card with a negative number (16 format) between each set of data cards.

6 If column 4 of card 5 is 1, omit this and the following data cards:

Card with number of flux monitor spectra 216,5Al, on tape and number of peaks in spectra A4 which are to be analyzed. Columns 13-17 must have the letters SMHDY in that order. Columns 18-21 must have the first four characters to be used in the sample name field on cards described under 8 below, which identify the flux monitor spectra. The card for each flux monitor spectrum must have the first four characters in the sample name field, identical to the listed characters.

- 7 Set of cards, one for each peak to be analyzed. Each card must have:
  - a. chemical symbol for element being determined using given photopeak

A2,BX, F10, 3,

3X, F7, 3, 4X,

Al, 5X

F10, 5, 6x, A4

- b. energy of photopeak in KeV
- c. radioisotope half-life

Card			Format
	e.	flux monitor weight or concentration	
	f.	units in which flux monitor weight is expressed (µg, mg, etc.)	μg, mg
8	Set	of cards, one for each spectrum on	4A4, 4X
	tape <u>ir</u>	order of spectra appearance on	F10.5, 6X, A4,
	tape.	Each card must have:	5X, I3, I2, 3X, I6, A1,
	a.	sample name	4X, I6
	b.	sample weight	
	c.	sample weight units (GRAM, Mg, etc.)	
	d.	time of day count started8:30 PM would be represented as 2030. If an activation analysis experiment runs into another day, time of second day starts at 2400 (e.g., 1:00 AM would be 2500)	
	e.	live time duration of count	
	f.	live time units	
	g.	spectra to be skipped after processing spectrum described by card.	

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