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The determination of the Uranium and Thorium content of certain Saudi Arabian ores by Neutron Activation Analysis

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The determination of the Uranium and Thorium content

of certain Saudi Arabian ores by Neutron Activation Analysis

by

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A Thesis Submitted to the

Graduate Faculty in Partial Fulfillment of the

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NOMENCLATURE

AA	Activation Analysis
DGMR	Directorate of General Mineral Resources
ERI	Engineering Research Institute
GS	Gravimeteric Separation
IAEA	International Atomic Energy Agency
ISU	Iowa State University
KAAU	King Abdul-Aziz University
KSA	Kingdom of Saudi Arabia
LDL	Lower Determination Limit
MCA	Multichannel Analyzer
MURR	Missouri University Resesarch Reactor
NA	Neutron Activation
NAA	Neutron Activation Analysis
NBS	National Bureau of Standards
SAEM	Saudi Arabian Educational Mission
SQSA	Semi-Quantitative Spectrographic Analysis
USAEC	United States Atomic Energy Commission
USDOE	United States Department of Energy
USGS	United States Geological Survey
UTR10	University Training Reactor (10 KW)
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence

INTRODUCTION

The purpose of this research was to analyze bore-hole samples from four locations in the KSA,[1] namely Jabal Tawlah, Jabal Hamra, Umm Al-Birak, and Jabal Sayid. The objective was to look for elements of potential interest, specifically, Uranium and Thorium, using NAA. Fig. 1 shows the four sample-source locations in the KSA.

NAA is a very sensitive technique for the identification and precise measurement of elements of interest. The central idea of this technique is the bombarding of the sample with low energy (thermal) neutrons and measurement, by a radiation detector, of the resulting decay photon activity. The selection of AA as the appropriate technique for determining the sample composition was made on the basis of its speed, sensitivity, availability, and economy.

The necessary irradiations can be obtained from nuclear reactors, radioisotopes (Cf), accelerator, and neutron generators. However, selection of an appropriate irradiation facility is based on the choice of the activating particle, the energy required, the intensity and the fluence. Moreover, there are additional factors which effect the final selection. They are as follows:

1. Sensitivity wanted, which requires consideration of the flux intensity,

2. Neutron energy,

3. Physical properties of the sample,

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[1] All abreviations are explained in nomenclature page.
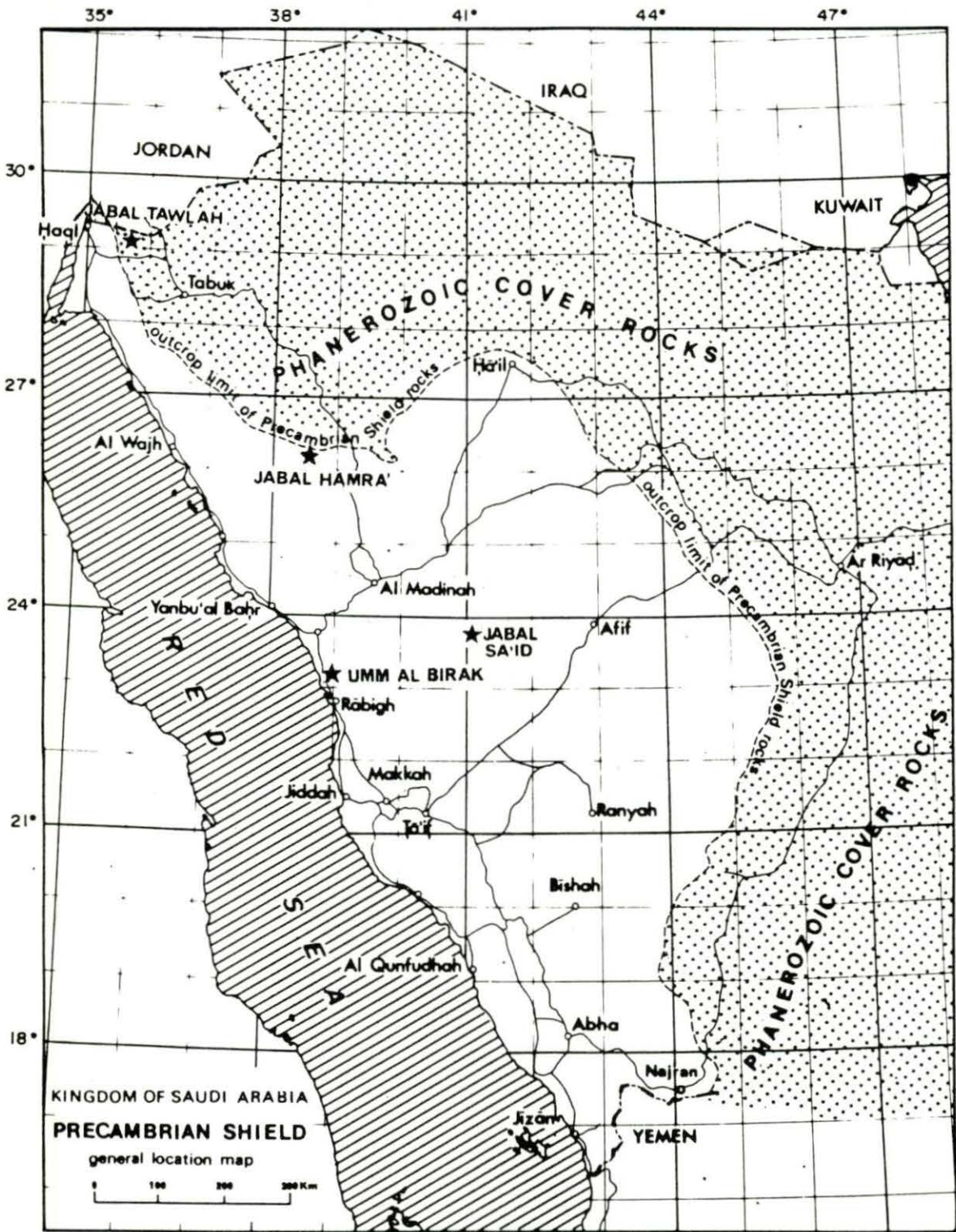
Fig. 1.   Four samples location map

4. Location and availability of the facility,

5. Irradiation cost.

Considering the above factors, MURR was selected for this thesis research. It has the highest neutron flux and steady state power reactor available at a U.S. university. Financial support for this research was provided by KAAU, Jeddah, KSA through the SAEM to the USA (Houston office) and the USDOE University Reactor Sharing Program. In order to be used in NAA the samples, taken from bore holes at various depths, were ground in the DGMR Laboratory, to produce a very finite powder.

The comparative technique for quantitative analysis was employed. This technique required that standard samples of the elements being sought must be provided. The standards and the samples must be simultaneously irradiated and treated (e.g., counted) in a uniform and consistent manner. This technique eliminates the principal sources of errors in activity measurement namely self absorption, scattering, geometric errors, radioactive decay and background radiation. The employed technique also minimizes the effects of neutron flux variation. Minimization of instrumental errors was carried out by frequent energy calibrations of the multichannel analyzer.

The presence of U and Th was determined by measuring photons from the daughter products. Pa-233 and Np-239 with associated half lives and energies of 270 days, 228.2 KeV and 2.35 days, 312 KeV, respectively.

Since U and Th concentration in the samples is generally small; one sample from each area was selected randomly for enhancement by the GS technique. Finally, NAA was used to determine the magnitude of GS enhancement.

REVIEW OF LITERATURE

The value of AA as a research tool was recognized almost from the moment Irene Curie and F. Joliet produced the first recognized activation of an element in 1933 by bombarding light metals with alpha particles from Polonium. This discovery and that of the neutron by J. Chadwick in 1932 led to the application of induced radioactivity to analytical problems. In 1936 the first NAA experiment was carried out by the Nobel Prize winners G. Hevesy and H. Levi [1], when they non-destructively determined dysprosium in rare earth material using a Ra-Be neutron source. The impure yttrium was bombarded by the neutrons in order to activate and measure the contaminant, a small quantity of dysprosium. Since then, interest in AA has grown rather slowly, perhaps awaiting improvements in the two basic experimental components, the irradiation facilities and the gamma detection and signal processing equipment.

In 1938, the determination of 10 mg amounts of europium in gadolium was reported [2]. In the same year, Seaborg and Livingood [3] described a technique in which charged particle activation could be applied to analytical problems. They determined trace quantities of gallium in ultrapure iron by irradiating and activating with cyclotron produced deuterons. These pioneers stimulated substantial interest in this new technique. One year later, Goldschmidt and Djourkovitch [4] showed that irradiation with neutrons and measurements of the intensity of activation was a satisfactory method for determining the dysprosium content in a mixture of yttrium-group oxides. With the advent of the first nuclear reactor on December 2, 1942, and the development of neutron generators,

sources of high neutron fluxes became available. Advances were reported
in proportional, scintillation and gamma ray counting techniques.

By 1952, several review papers on AA had presented details of basic
principles and applications including some experimental data [5-14].
Mahlman and Leddicotte (1955) [15] were the first to use NAA to determine
submicrogram quantities of uranium in silicate material. They separated
the isotope Np^{239} chemically from irradiated samples and used this
isotope to estimate the U^{238} concentration. In the past two decades,
improvements in electronic technology had led to the growth and
refinement of the gamma ray detectors and spectrometers. During the
period of rapid development of nuclear reactors, corresponding rapid
improvements in AA were made when NaI (Tl) scintillation detectors were
introduced. In 1953, Khan and Lyon [16] pointed out the usefulness of
the NaI (Tl) photon spectrometer as an aid to identifying radionuclides.
Other articles rapidly followed pointing out the potential for NAA
employing gamma ray spectrometry [17-21]. However, the full potential of
the scintillation counter was not achieved until multichannel analyzers
appeared in 1955 [18]. Further expansion of AA came with the development
of solid state detectors. In 1965, groups identified with Girardi et al.
[22], Schroeder et al. [23], and Hollander et al. [24] were among the
first to use lithium drifted germanium detectors for activation work.

A number of papers on various aspects of AA were presented at the
two United Nations International Conferences on the Peaceful Uses of
Atomic Energy held in Geneva in 1955 and 1958. By this time, specializa-
tions had appeared in papers devoted to such fields as ion exchange
[25,26], gamma spectroscopy [17,27], nuclear emulsion detectors [28,29],

limitation and sources of errors [30], use of low-level neutron sources
[31], comparison with other analytical methods [32-34], archeology [35],
biological systems [36], and dating of minerals [37]. Advances in AA
have been discussed under the general heading of Nucleonics in the annual
reviews of analytical chemistry published in the journal "Analytical
Chemistry."

Reviews of AA were written by Gordon in 1949, 1951, and 1954
[7,8,38]; by Meinke in 1956, 1958, and 1960 [39-41]; and by Leddicotte in
1962 [42]. These reviews were intended to provide complete coverage of
the available literature. A comprehensive bibliography of radioactiva-
tion analysis was published in 1957 by Gibbons and his co-workers [41].
The bibliographies contain sections devoted to reviews and papers of
general concern, the matrices in which precise elements have been
resolved, and the elements which have been resolved [43]. In 1960, Koch
[44] condensed these reviews and presented them in a handbook of AA.
This useful reference records for each element the relevant nuclear data,
the evaluation of different reactions leading to a particular nuclide,
interfering reactions, competing reactions, and data on, or reference to,
thresholds, cross sections and estimates of sensitivities of the various
activation methods.

A broad bibliography of the AA literature is given by Bock et al.
[45]. Also, Raleigh's [46] book, Literature Search of AA contains 391
selected references through 1963 on the use of radioactivation in a
qualitative and quantitative analysis of materials. Its index classified
entries as 1) reviews and general interest, 2) materials analyzed, and 3)
elements resolved.

A familiarity with activation cross section, nuclear abundances, energy levels, and disintegration methods, is needed to understand and interpret activation measurements. In Koch's [44] handbook, the nuclear data most relevant to AA were tabulated. The works of Salmon [47], and Senftle and Champion [48] provided tabulations of nuclear data for thermal NA, including cross-sections, isotope abundances, half lives, and emission products. In addition, calculated saturation activities and activities produced by one-second exposures at standard fluxes were provided. Detailed nuclear energy levels, decay schemes of the nuclides, and other nuclear data are available in "Nuclear Data Sheets" [49] which is now published as the periodical, "Nuclear Data" [50]. Many nuclear charts have been developed. Probably the best known, the "Chart of the Nuclides" [51], is very useful and is periodically revised. NAA and fission track techniques have been used to determine U abundances in terrestrial ultramafic rocks [52-56].

In the last two decades, six international conferences dealing with AA have been held and their proceedings are valuable concentrated sources of information on AA developments. The first conference, entitled "Modern Trends in AA," was held in 1961 at Texas A&M University, College Station, Texas [57]. It was jointly sponsored by the IAEA, the division of Isotopes Development of the USAEC and the AA Research Laboratory of Agricultural and Mechanical College of Texas (Texas A&M University). Papers presented at the Conference which stimulated the development of such analytical techniques as "AA," were flux monitors, automatic systems, and feedback to improve stability. The papers presented fell into two classifications: 1) Development of techniques: use of

multichannel pulse height analyzer, gamma ray spectroscopy, and explanations of interferences. 2) Applications: determination of tracer elements, metal industry, use of accelerations, (fast neutrons), food industry, aerospace industry, forensic science, and medical use.

A significant change in emphasis occurred at the "1965 International Conference on Modern Trends in AA" held again at Texas A&M University [58]. The subjects presented fell into two different interests areas: a) computer - coupled automatic system, which followed developments of statistical interpretation of decay characteristics, computer programming, optimization of irradiation, decay, and counting times, giving the system the advantage of automation and speed; and b) use of commercially available 14-Mev neutron generators.

The "Third Conference on Modern Trends in AA," was held in 1968 at the National Bureau of Standards (NBS), Gaithersburg, Maryland. This conference featured five plenary lectures, 1) the applications of AA, 2) radiochemical separations, 3) nuclear reactions, 4) radiation detection and data processing and 5) computation methods in AA [59]. The fourth conference was held at Saclay, France, in 1972. The conference proceedings were divided into three sections. The first, included general applications, fast neutron activation and use of isotopic sources and computation techniques; second, thermal neutron activation and radiochemistry, thermal neutron activation and radiochemistry in biological sciences, and photon activation; and third, charged particles activation, charged particle activation and special techniques, analysis by direct observation of nuclear reactions and free communications [60]. The "Fifth International Conference on Modern Trends in AA," was held in

Munich (F.R. Germany) in 1976. The main headings included fundamental contribution, technical development, biological and biomedical applications, and environmental and ecological applications [61]. The last International Conference on Modern Trends in AA was held in Toronto, Canada, 1981. The main paper headings were, analytical studies by NAA and other methods, archeometry and biological applications [62]. Principles of NAA and AA are discussed in many books [63-78].

BASIC PRINCIPLES OF NAA

A number of trace analyses techniques are possible as shown in Table
1 [67]. Based on the consideration summarized in the table, AA is
selected as the best technique. It is an isotopic analysis procedure
achieved by producing artificial radioactivity within a specimen through
particle bombardment. Thus, a stable isotope, when irradiated, for exam-
ple, by neutrons, can undergo a nuclear transformation to produce a
radioactive nuclide. The high sensitivity with which this induced
radioactivity can be detected, the penetrability of many substances for
certain activating and emitted particles, and the individually charac-
teristic modes of decay of each radioisotope lead to the many advantages
of this technique. The definite advantages of AA are as follows [63,64]:

1. The ultimate sensitivity is excellent for nearly every element
    and is for many elements better than can be obtained by any
    other technique. Note, however, those for which it is poor
    (e.g., O, C, and N).

2. The possibility of non destructive analysis.

3. It is generally possible to determine several elements
    simultaneously in a single sample.

4. Provided that no pre-irradiation chemical operation is
    attempted, the technique is free from errors caused by the use
    of contaminated reagents.

5. Before irradiation chemical treatment is greatly facilitated by
    freedom to use carrier techniques, thus eliminating the
    necessity for rigorous microchemical procedures in the

Table 1. Survey of the quality of different trace analysis techniques [67], 1972

Technique	Several elements simultaneously	Sensitivity (ppm)	Specificity	Accuracy	Freedom from contamination and interferences	Possibility of overcoming surface contamination
Activation analysis	in several cases	0.001	good	good	good	good
Atomic absorption and flame spectrophotometry	no	0.01	good	good	bad	reasonable
Emission spectrometry	yes	0.1	good	reasonable	reasonable	reasonable
Gas analysis (reducing fusion)	O_2, N_2, H_2	1	reasonable	reasonable	reasonable	bad
Mass spectrometry spark source	yes	0.01	good	needs stand.	good	good
isotope dilution	no	0.001	good	reasonable	bad	bad
Polarography (cathode ray, pulse)	yes	0.01	reasonable	good	bad	reasonable
Spectrophotometry and fluorimetry	no	0.1-0.01	reasonable	good	bad	bad

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determination of trace constituents.

6. In principle, AA allows the opportunity of distinguishing
    between different isotopes of an element.

7. Determination of the distribution of components can be
    accomplished by autoradiographic or counter scanning
    techniques; and in some instances rapid, nearly automatic
    machine analysis and monitoring, is possible.

8. According to L.A. Haskin [64], NAA is the best technique for
    multielement analysis. A serious problem with early NAA
    measurements was the inability to investigate many elements
    simultaneously, so interelement correlation went unnoticed.
    With more modern techniques, the total work and time required
    for analysis has been reduced because samples need to be
    prepared and handled only once or twice.

9. Fifty to 100 samples can now be analyzed accurately,
    efficiently and rapidly in a single experiment [64].

10. After a sufficient cooling time the samples can be reused for
    other purposes [75].

A number of significant limitations of AA are as follows [63]:

(a) Since the technique is based on characteristics of atomic
    nuclei, it does not give any information about the chemical
    form in which a particular element is present.

(b) Near the limit of detection the main source of error in
    activation analysis lies in the fluctuations associated with
    the statistical character of nuclear disintegrations. This
    error, although perhaps negligible, is still present even when

the element sought occurs in amounts well above the limit of detection.

It is to be noted that AA is rarely used for macro-determination except when speed and convenience, rather than sensitivity, are decisive. So far, the main use of the technique has been in microdetermination of elements close to- or beyond the limits of sensitivity offered by other methods.

(c) Though the chemical and instrumental methods used in AA are relatively simple and inexpensive, the irradiation stage of technique requires access to a nuclear reactor or some other source of sub-atomic particles or radiation.

In principal, AA can be based on any artificially induced nuclear reaction. It can be distinguished by three main possibilities:

First: Energy given to a nucleus by bombardment with energetic γ-ray quanta may be used to promote particle emission or to produce excited states from which gamma emission can occur.

Second: Heavier particles, such as protons, deuterons, and other nuclei may be accelerated by electrical means and used to bombard the sample. Such particles may be incorporated in the nucleus, altering its mass, charge and radioactive status, or may bring about an internal rearrangement resulting in the emission of other nuclear particles.

Electrically-charged projectiles may be furnished with any desired energy by electrical means, but they have the disadvantage of penetrating the surface of the sample by only a short distance. The method provides information about the composition of the surface and not about the sample as a whole.

Third: Nuclear reactions of the kind desired for AA are most generally induced by neutron bombardment. Neutrons may be used to produce radioactive nuclides from almost every element, often with very high yield. In general, they have good penetration, allowing nearly uniform activation of the entire sample.

Most AA work is carried out using thermal energy neutrons (2200 m/sec) for the following reasons [65]:

1. Most materials have a large cross-section for thermal neutrons.

2. In many research reactions, high thermal neutron fluxes are available (10^{13} n/cm^2 sec or greater).

3. Radiation capture is the most probable nuclear reaction that takes place at thermal energy enabling the radioisotopes formed to be isotopic with target elements. The radiation capture (n,γ) reaction is one in which the target nucleus captures a neutron and gains one unit in atomic mass. The resulting nuclide, a heavier isotope of the activated element, will usually be radioactive and will decay by emitting beta particles and usually gamma rays.

    (For example: $Fe^{58}(n,\gamma)Fe^{59} \xrightarrow{\quad \beta^- \quad} Co^{59}$)
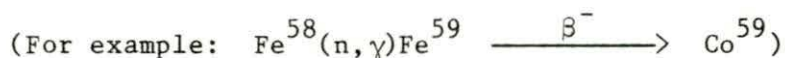
NAA is a method of elemental analysis based upon the properties of the nucleus [66]. It, therefore differs from chemical methods of analysis which are based on the behavior of outer electrons. During NAA, the exposure of nuclei of stable isotopes to neutrons results in the production of radioactive isotopes or radionuclides which emit radiations as they decay to stable nuclei. An understanding of NAA involves nuclear structure, radioactivity, nuclear transformation and the inter-

action of radiation with matter. The theory of these processes is thoroughly discussed in many standard references [67-78]. A summary of those processes which are of direct relevance to NAA and its application to geological material is provided (up to the end of interferences section).

In NAA employing thermal neutrons (n) to activate the sample a typical reaction would be:

$$^{23}_{11}Na + \, ^{1}_{0}n \longrightarrow \, ^{24}_{11}Na + \gamma$$

| target nucleus | neutron | new radioactive isotope | prompt radiation |
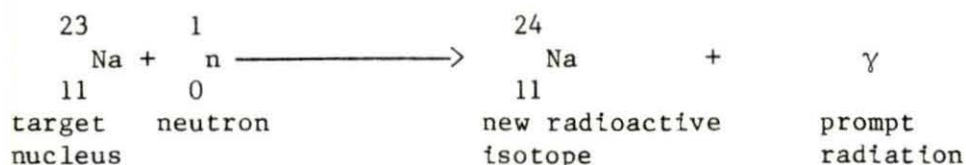
The energy, intensity and kind of radiations, together with decay half life, are used to characterize and quantify the elements present in the sample.

$$e.g., \quad ^{24}_{11}Na_{T_{1/2}} \xrightarrow[15.02 \text{ hrs}]{\beta^-(1.39 \text{ MeV})} \, ^{24}_{12}Mg^* \xrightarrow[\gamma(1.37 \text{ Mev})]{\gamma \, (2.75 \text{ MeV})} \, ^{24}_{12}Mg$$
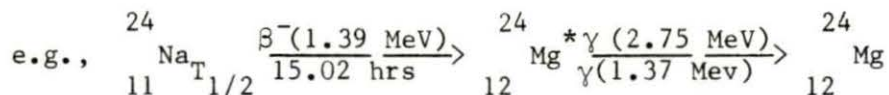
The measurement of the decay radiations is usually made at some convenient time and place after the end of activation. NAA typically uses the comparison method of analysis, which involves activating a standard material of known content, along with a sample containing the same material, but with an unknown concentration, and then comparing the two sets of induced activities to establish the unknown quantity.

Stable and radioactive nuclides are shown as a function of neutron and proton numbers in Fig. 2 [82]. Neutron irradiation usually produces β^- active nuclei, i.e., above the stability level.

ARTIFICIAL RADIOACTIVITY
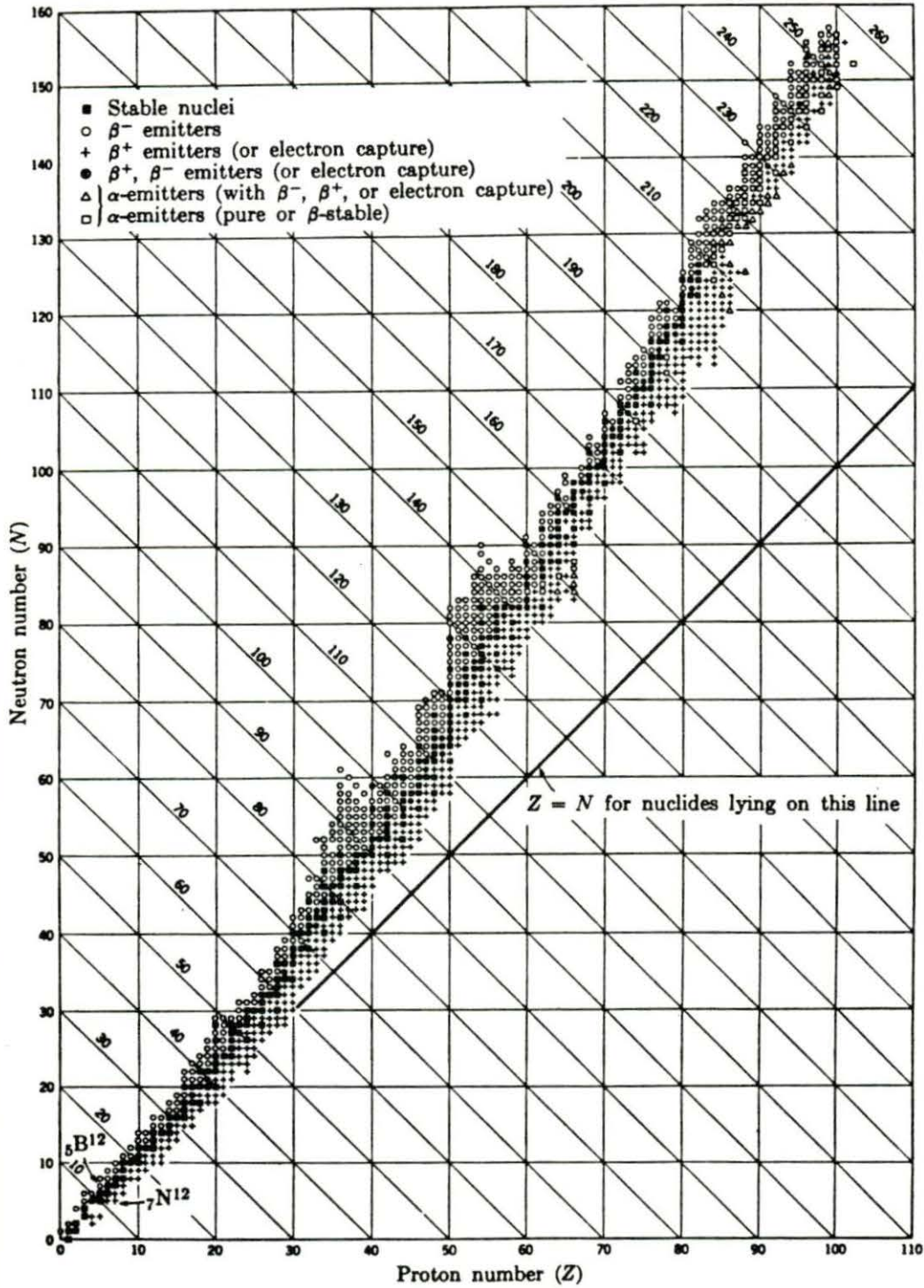
Fig. 2. Stable and radioactive nuclides as a function of neutron
and proton numbers

The energies are well defined, consequently radiations with energies appropriate for the various possible transitions accompanies nuclear reactions which populate the excited nuclear levels.

The various ways in which radioactive nuclei can decay is shown in Fig. 3 [66,79,80]. Since in NAA elements are usually identified by means of the gamma radiation emitted from the radionuclides produced by activating the elements, inspection of the relevant decay schemes is essential for planning and interpretation of the results. For example, in 92% of the disintegrations of Cs-137 by β^- decay, the Ba-137 nucleus is left in the .662 MeV excited level (Fig. 3). The isotope is de-excited mainly by the emission of a .662 MeV gamma ray and by internal conversion of K and L electrons. Two other examples of decay schemes are also given in Fig. 3. Branching ratios of such schemes are independent of temperature, pressure and chemical state.

Although most excited nuclei are de-excited rapidly by gamma emission, there are some nuclei that remain in excited state for considerable period (i.e., greater than a few milliseconds). Thus, the same nuclide may have different nuclear metastable isomer, designated by "m" after the mass number, e.g., $^{60m}_{30}Zn$.

Activation by Neutrons

The probability of a neutron induced reaction depends on the neutron energy. Neutrons can be classified according to their kinetic energy, (Fig. 4) [66], as follows:

1. Slow neutrons have energies up to about 1 KeV and include

(a) Thermal neutrons, which are the most abundant particles in a
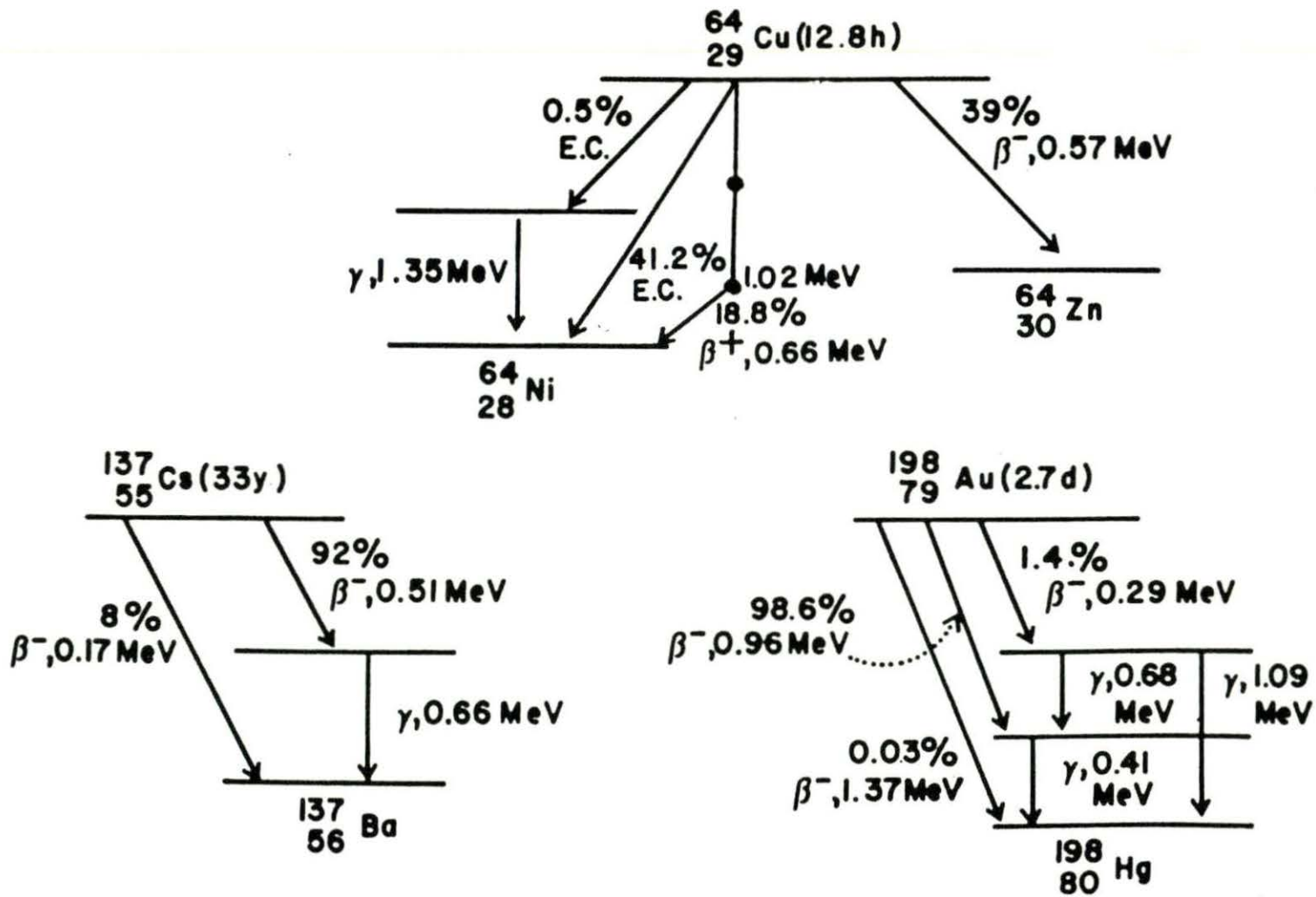
Fig. 3.   Examples of radioisotope decay schemes
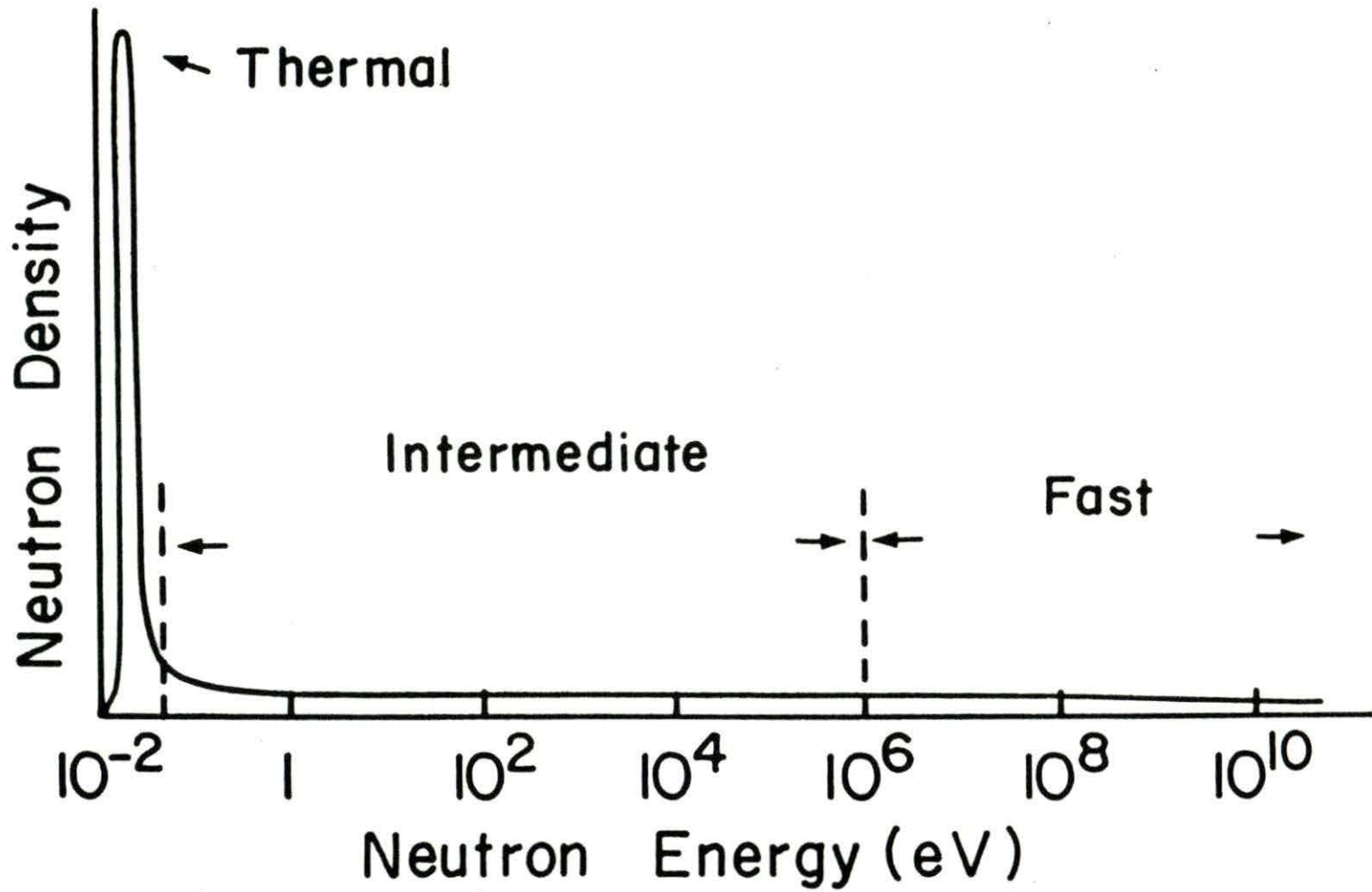
Fig. 4. Generalized distribution of neutron energies in a reactor

reactor, and are in thermal equilibrium with surrounding (moderating) nuclei at the ambient temperature having energies of the order of .025 eV.

(b) Epithermal neutrons are in the process of slowing down and thus are not in complete thermal equilibrium with their environment. They have energies of about .2 eV.

(c) Resonance neutrons with energy in the range of 1 to 1000 eV.

2. Intermediate neutrons (energies between about 1 and 500 KeV.)

3. Fast neutrons with energies above .5 MeV.

Types of Neutron Reactions

There are several different types of reactions induced by neutrons. The most common are:

1. neutron capture,   2. transmutation,

3. fission   and   4. inelastic scattering

1. Neutron Capture:

Neutron capture involves the absorption of a low energy neutron by a nucleus and the prompt emission of γ-ray. e.g., $^{23}Na(n,\gamma)^{24}Na$. As a result, the mass number of the newly formed isotope is increased by 1. The product isotopes are usually unstable and undergo β^{-} decay followed by gamma emission.

2. Transmutation

Transmutation activation reaction takes place when a neutron is absorbed by the nucleus resulting in the release of a charged particle, usually a proton, e.g., $^{27}Al(n,p)^{27}Mg$. The

product nucleus is generally unstable and decays through

β^- emission back to the target nucleus, e.g.,

$^{27}Mg \xrightarrow{\beta^-} {}^{27}Al$. Other transmutation reactions may involve

the release of α, 2n, d particles. Most of these reactions

have a threshold energy, and therefore, occur at relatively

high neutron energies (i.e., fast or intermediate neutrons).

3. Fission

In this process of greatest interest, a thermal neutron is

absorbed by one of several heavy nuclides ($Z > 90$). The

compound nucleus splits into two large fragments and

simultaneously releases 2 to 3 neutrons. Since neutrons are

required for the reaction, and are produced by the reaction,

the process can lead to a chain reaction which produces a

source of neutrons in a nuclear reactor.

4. Inelastic scattering

The neutron is not absorbed by the target nucleus and may

transfer part of its kinetic energy to the target nucleus.

Effective Cross-Sections

The various types of neutron interactions with a nucleus occur with

different probabilities which are measured by the relevant cross-

sections. The neutron flux, ϕ, is the number of neutrons passing through

an area of 1 cm^2 during a time of 1 sec,

$$\phi = n.v \text{ neutrons/cm}^2 \cdot \text{sec}$$

where: n is the number of neutrons (per cm^3) and

v is their velocity in cm sec^{-1}.

The number of neutron interactions, X_i, of a given type occurring per unit time is directly proportional to the number of target nuclei, N, in a sample, and to the neutron flux, and is defined as follows:

$$X_i = \sigma_i \phi N \tag{1}$$

where σ_i is the cross-section of the given interaction. Cross-sections are usually measured in barns (1 barn = 10^{-24} cm^2). If the target nucleus can undergo different nuclear reactions, cross-sections exist for each type of interaction: inelastic scattering, neutron capture, fission, etc. The total cross-section, σ_T, is a measure of the probability that an interaction of any type, will occur. Thus, σ_T is equal to the sum of the cross-sections of all types of interactions. The number of reactions of all types for a given target nucleus is

$$X = \sigma_T \phi N \tag{2}$$

In practice, however, the target nuclei are usually bombarded by neutrons of a limited energy range where a particular type of interaction prevails and other reaction types have very small cross-sections. As a typical example, the dependence of the total cross-section on the energy of the activating neutron is shown in Fig. 5 [66]. For the case of ^{27}Al, at low energies, the total cross section decreases linearly with increasing energy on a log-log plot. The dependence in this energy region follows a "1/v law" which indicates that as the energy, and hence, the velocity of the neutron increases, the time that the neutron spends in the region of the nucleus decreases, and therefore, the probability
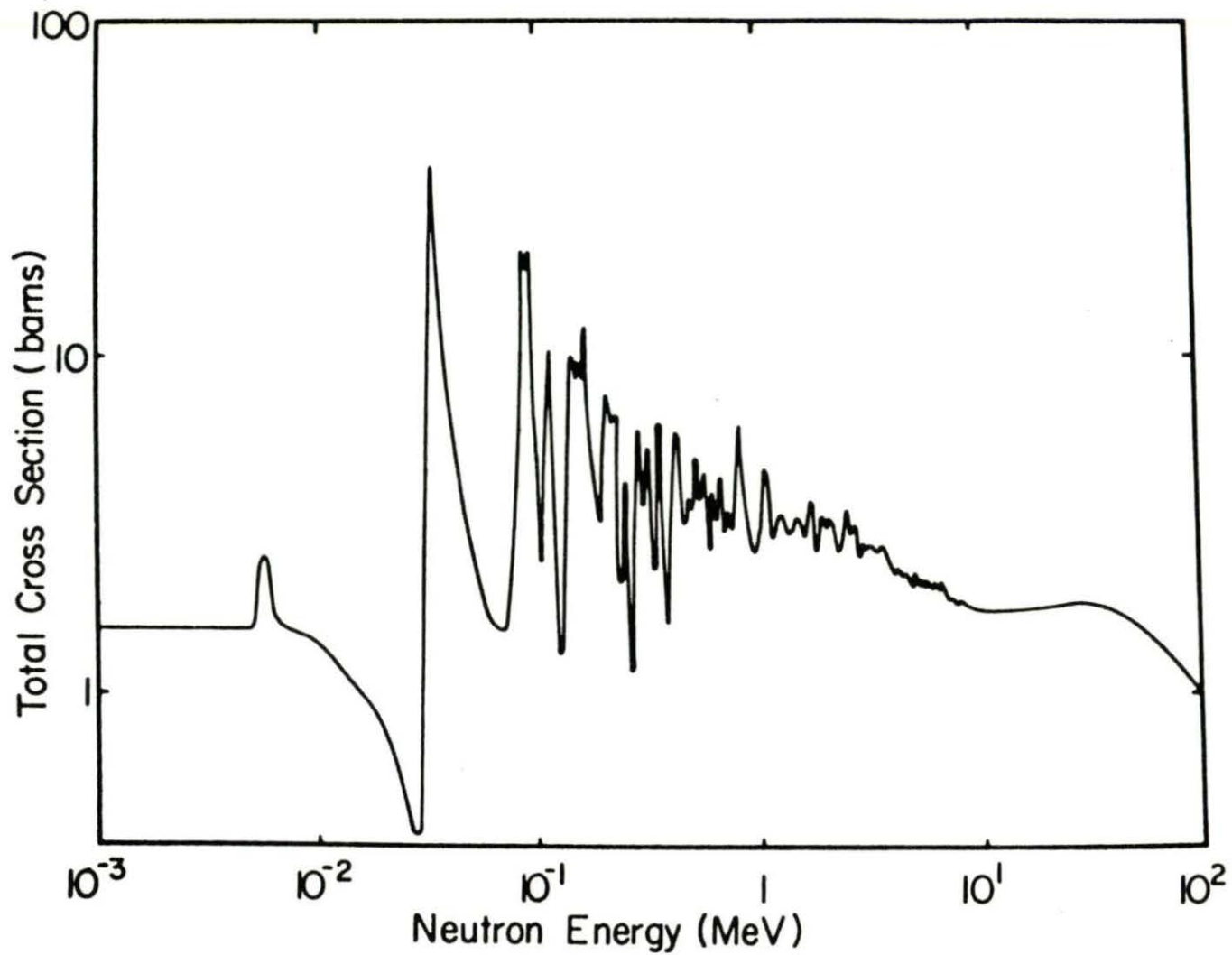
Fig. 5. Total activation cross-section of ^{27}Al

that a reaction will occur also diminishes. At energies in the region of
a 0.1-1 MeV, resonance peaks appear in the plot. These peaks occur when
the sum of the kinetic and binding energies of the neutron in the center
of mass system is equal to the energy of an excited state of the compound
nucleus. The value of σ_T increases dramatically at these energies.
Elastic scattering (n,n) and neutron capture (n,γ) dominate in reactions
with thermal neutrons. Inelastic scattering (n,n') occurs at higher
neutron energies and since transmutation reactions have a threshold
energy they also only occur at higher neutron energies.

Theory of Activation Analysis

During irradiation of a material in a nuclear reactor, some of the
atoms of the sample interact with the bombarding nuclear particles and
are transformed into different isotopes, many of which are radioactive
and decay emitting characteristic radiations. The amount of radiation
emitted from activated samples will be proportional to the amount of the
isotope present in the original sample.

Production of Radioisotopes

The number of neutron interactions, X_i, of a given type occurring in
unit time depends upon the product of the cross-section for the
interaction, σ_i, the neutron flux, ϕ, and the number of target nuclei, N,
as indicated in Equation (1). Assuming that the activating neutrons are
monoenergetic and that a single radionuclide is produced, the increase in
the number of radioactive nuclei, N*, with time from the beginning of
activation is described by:

$$\frac{dN^*}{dt} = \sigma_{act}\phi N - \lambda N^* \tag{3}$$

where σ_{act}: is the activation cross section.

$\sigma_{act}\phi N$: is the production rate of the radioactive nuclei,

and

λN^*: is the rate of decay of those same nuclei.

Thus

$$N^* = \frac{(\sigma_{act}\phi N)}{\lambda} (1-e^{-\lambda t}) \tag{4}$$

The activity, A_t, is given by

$$A_t = \lambda N^* = \sigma_{act}\phi N(1-e^{-\lambda t}) \tag{5}$$

The saturated activity corresponds to $t \longrightarrow \infty$, thus $(1-e^{-\lambda t}) = 1$,

$$A_t = \sigma_{act}\phi N = A_{sat} \tag{6}$$

Thus

$$A_t = A_{sat}(1-e^{-\lambda t}) \tag{7}$$

The relationship between induced radioactivity and time is shown in Fig. 6 [3]. Irradiations times are usually limited for practical reasons and rarely need to be much longer than one half-life, since eight times the exposure only doubles the activity. For irradiation times less than one-half life, the amount of induced activity is nearly proportional to the irradiation time. Fig. 6 shows generalized growth and decay curves of radioactivity.

The number of radioactive nuclei remaining after a cooling or delay
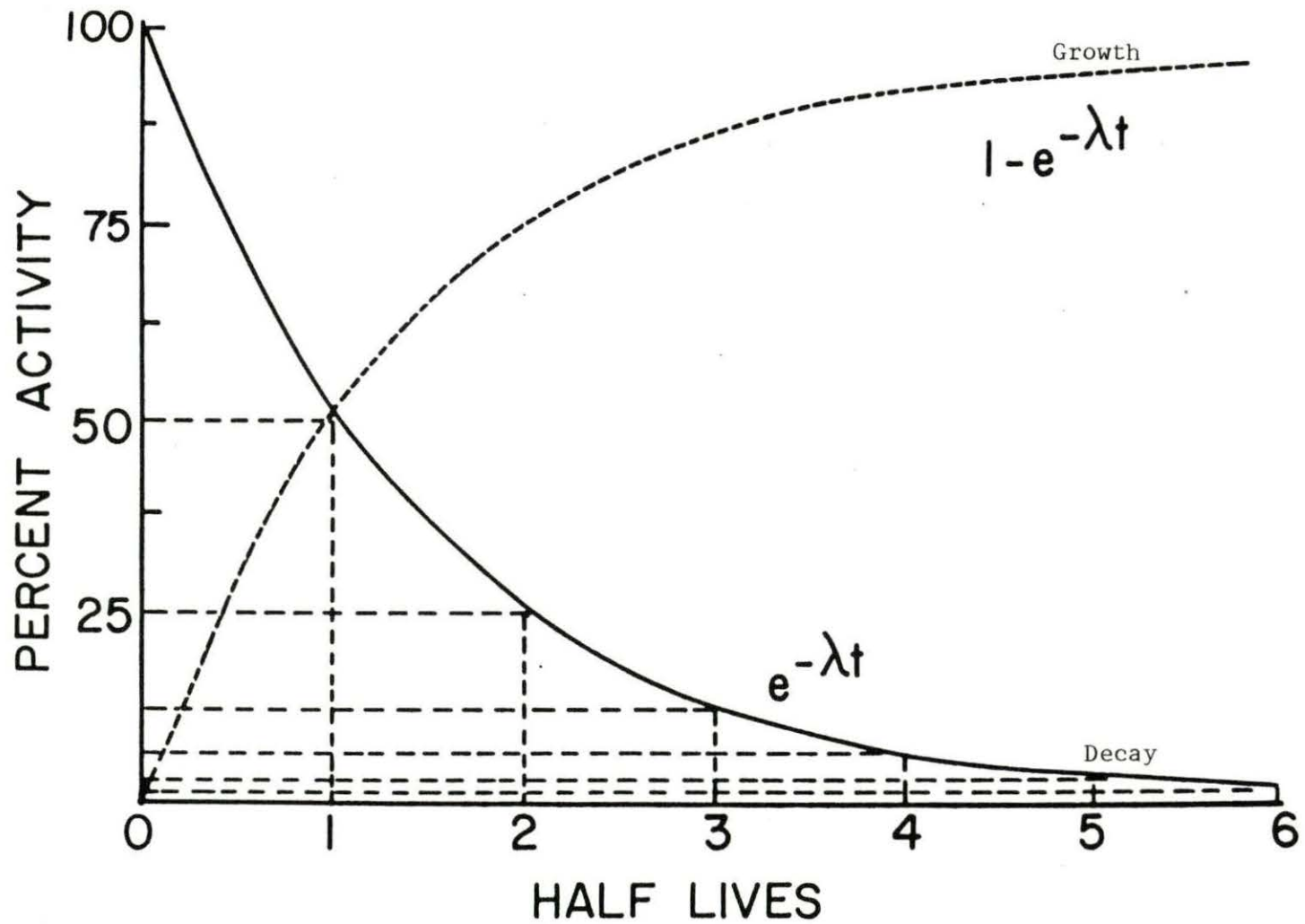
Fig. 6. Growth and decay curves of radioactivity

period, t_d, measured from the end of an irradiation time, which lasted t_i, can be obtained from Equation (4);

$$N^{*\,\prime\prime} = \frac{(\sigma_{act}\phi N)}{\lambda} (1-e^{-\lambda t_i})e^{-\lambda t_d} \tag{8}$$

The number of radioactive nuclei decaying during the counting interval, t_c, is given by:

$$N^{*\,\prime} = \frac{\sigma_{act}\phi N}{\lambda} (1-e^{-\lambda t_i})e^{-\lambda t_d}(1-e^{-\lambda t_c}) \tag{9}$$

If the relative intensity of emitted gamma-rays of a given energy is I , and if the efficiency of the detector including the solid angle correction for this gamma-ray, is E , then the measured count is

$$N^{*} = \sigma_{act}\phi N(1-e^{-\lambda t_i})e^{-\lambda t_d}(1-e^{-\lambda t_c})I_\gamma E_\gamma \tag{10}$$

In comparison type NAA experiments where samples and standards are irradiated together in the same flux, and where the same energy γ-ray is counted for the same period of time, Equation (10) can be simplified to

$$\frac{N^{*}(u)}{N^{*}(k)} = \frac{N(u)}{N(k)} \cdot \frac{e^{-\lambda(t_d)_u}}{e^{-\lambda(t_d)_k}} \tag{11}$$

where u and k refer to unknown samples and standards, respectively.

N(u): proportional to the amount of the element in the sample.

The terms $e^{-\lambda(t_d)}$ with subscripts u and k must be retained in Equation (11) since samples are counted sequentially; i.e., different samples have different t_d and the activity must be normalized.

Equation (11) can be rewritten in terms of the unknown concentration

of the element in the sample, and the known concentration of the same

element in the standard, as:

$$\text{ppm}(u) = \text{ppm}(k) \; \frac{W^*(k)}{W^*(u)} \cdot \frac{N^*(u)}{N^*(k)} \cdot \frac{e^{-\lambda(t_d)_k}}{e^{-\lambda(t_d)_u}}$$

where $W^*(k)$ and $W^*(u)$ are the weights of standards and unknown samples,

respectively, and ppm stands for parts per million.

In addition to reactions which lead to the direct formation of

stable nuclei from the product of activation, there are other reactions

during which radioactive intermediares are produced. In a general form,

such reactions can be expressed as:

$$\begin{array}{ccccccc}
& \text{activation} & & \text{decay} & & \text{decay} & \\
A & \xrightarrow{\hspace{2cm}} & B & \xrightarrow{\hspace{2cm}} & C & \xrightarrow{\hspace{2cm}} & D \\
\text{target} & & \text{parent} & & \text{daughter} & & \text{stable}
\end{array}$$
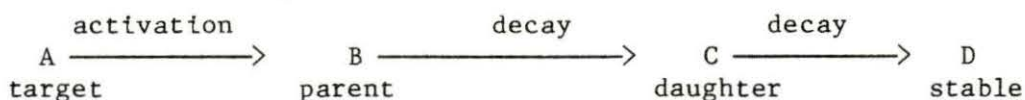
An example of such decay scheme is a radioactive series involving

Th and U in reaction

$$^{232}\text{Th}(n,\gamma)^{233}\text{Th} \xrightarrow[T_{\frac{1}{2}} = 22.2 \text{ min}]{\beta^-} {}^{233}\text{Pa} \xrightarrow[T_{\frac{1}{2}} = 27.0 \text{ days}]{\beta^-} {}^{233}\text{U}$$

$$^{238}\text{U}(n,\gamma)^{239}\text{U} \xrightarrow[T_{\frac{1}{2}} = 23.5 \text{ min}]{\beta^-} {}^{239}\text{Np} \xrightarrow[T_{\frac{1}{2}} = 2.35 \text{ days}]{\beta^-} {}^{239}\text{Pu}$$
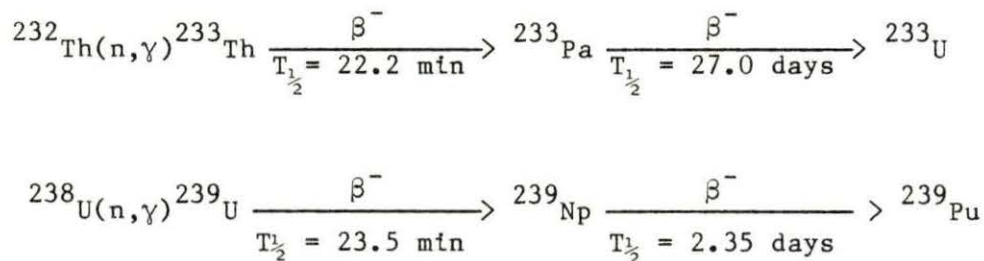
The decay of the daughter radioisotope is used to determine the

amount of parent present in the sample. ^{233}U and ^{239}Pu are considered to be

stable products since their half-lives are 1.592×10^5 and 2.411×10^4 years,

respectively [51]. The associated decay schemes for Th and U are shown

in Figs. 7 and 8, respectively [83].

22.2 m

$_{90}$Th^{233}

β^-

27.0 d   3/2−

$_{91}$Pa^{233}

β^-

26% 6.6
13% 7.0
26% 7.2
27% 7.3

5% 9.2

Q_β− 0.571
Q_α ≈4.4 est
(MTW)

α

3/2+
1/2+
5/2+
3/2+

9/2+
7/2+
5/2+

42 0.4159 E2 + 20% M1
16 0.3754 E2
19 0.10386 M1 + ≈1% E2
23 0.07528 M1
W   0.01726 M1 + ≈8% E2
39 0.3986 E2
61 0.08659 M1 + ≈1% E2
W   0.0579
39 0.3405 M1
63 0.3002 M1
0.6 0.02854 M1 + 2% E2
99.2 0.3119 M1, <2% E2
0.8 0.2716 E2

M1 + 31% E2

0.4158
0.3986
0.3405
0.3119   0.2 ns

0.0920
0.04035
0   1.62 x 10^5 y

$_{92}$U^{233}

Q_α 4.909
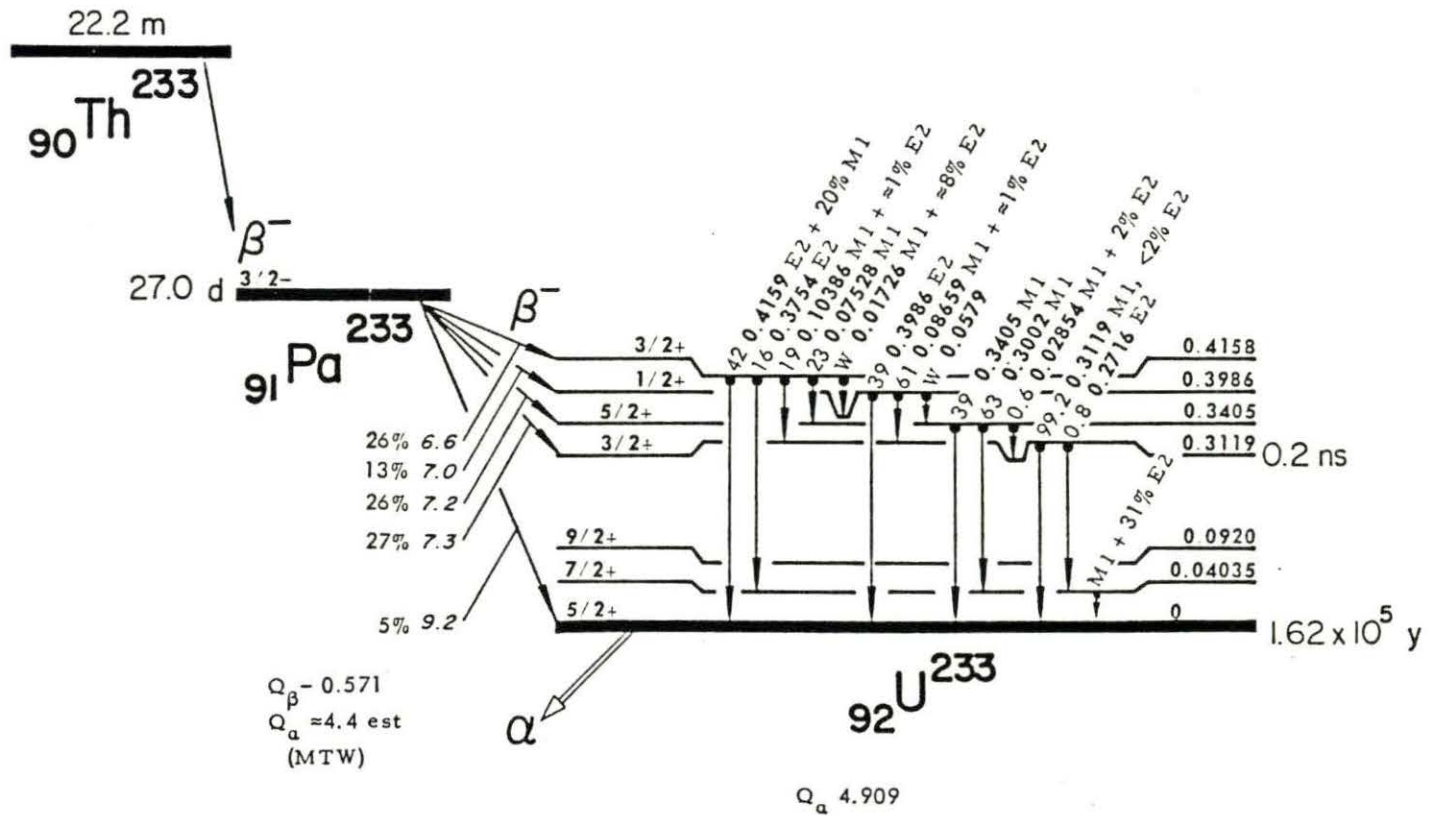
Fig. 7.   Th 233 decay scheme
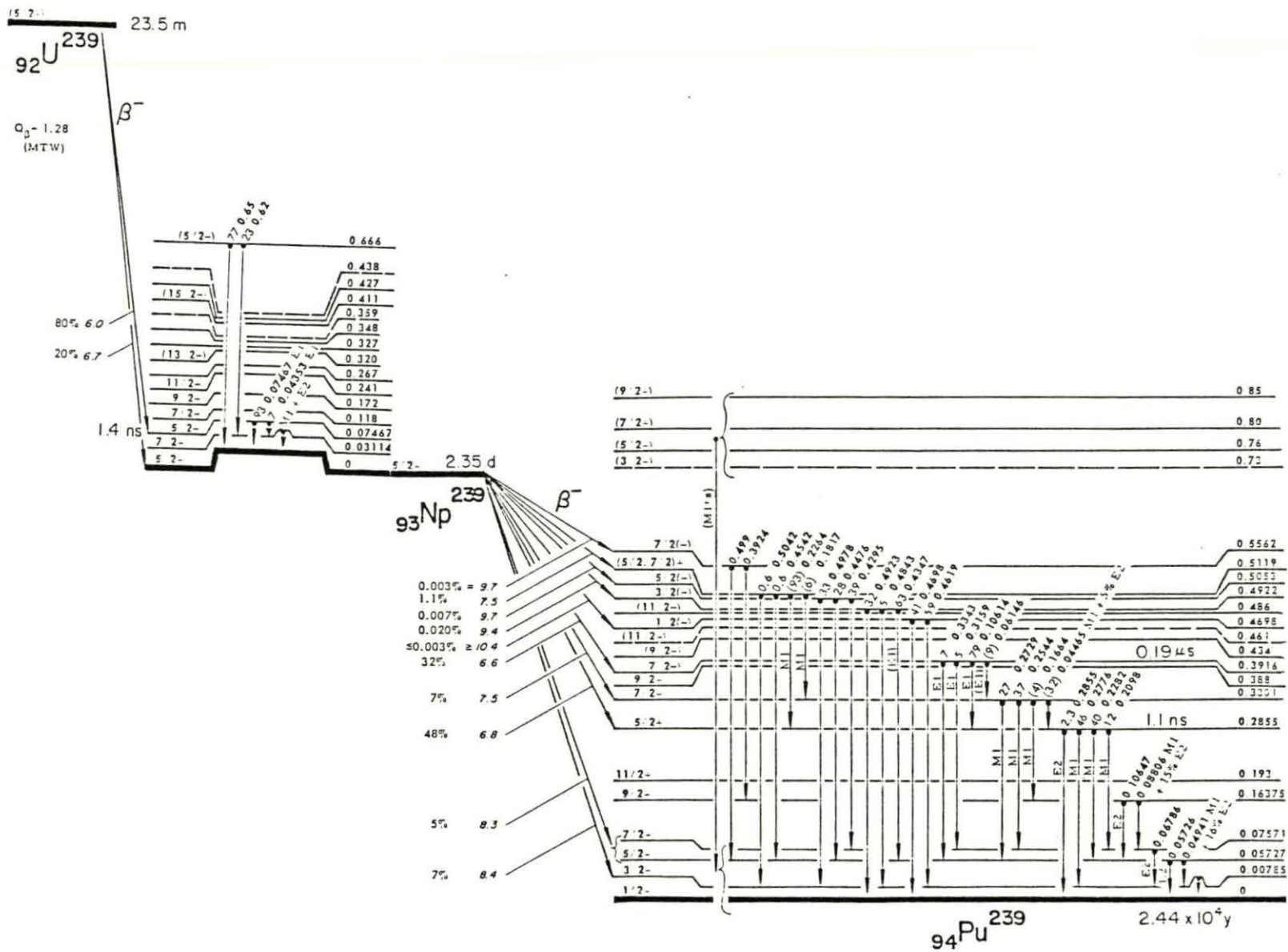
Fig. 8. U239 decay scheme

Decay Times

Many radionuclides are formed during the irradiation of geological samples. The activities of these radioisotopes depend upon the irradiation time (Eq. 5) and cooling time from the end of activation.

In practice, these two times are chosen to maximize the activity of the radioisotopes of interest, and minimize the activity of other radio-isotopes, especially those which have peaks with energies close to that of the peak of interest. Such isotopes may interfere with the activity determination. If the isotope of interest has a half-life shorter than the interfering radioisotopes, a short irradiation time is used. If the desired element has a longer half-life than the interferents, a longer cooling time could be chosen. As an example of minimizing the inter-ference from longer lived isotopes, consider what happens when copper and silicon are neutron irradiated together. The $^{63}Cu(n,\gamma)^{64}Cu$ reactions has a $T_{1/2}$ = 762.6 min [51,81]. ^{31}Si is also formed by the $^{30}Si(n,\gamma)^{31}Si$ reactions and has a $T_{1/2}$ = 157.2 min [51,81]. If the copper activity is measured after 628.8 min cooling, which corresponds to four half-lives of ^{31}Si, the radiosilicon activity would have decayed to one-sixteenth of its original value, while the activity of copper will have decayed to roughly half of its initial value. Thus the copper peak would be easier to find and analyze.

Radioactivity Detection

Gamma-rays detection

Following neutron activation, the radionuclides produced decay by emitting a beta followed usually by one or more gammas. These gamma rays

energies are used as analytical properties to identify the radioisotopes, and the amount of induced radioactivity is used to determine the amount of the desired radioisotopes present. A sample's radioactivity can be measured only when the radiation given off by the decaying nuclei interacts with some material. When gamma rays interact with matter they lose most or all of their energy in one event. Gamma rays interact with matter in three ways: the photoelectric effect, pair production, and the compton effect.

1. Photoelectric effect: This process implies the total absorption of a photon by an atom, through ionization. The photon vanishes during the interaction and transfers all its energy to an electron which is ejected from one of the electronic shells.

The photoelectron energy is close to the gamma-ray energy because the binding energy of an electron to the nucleus is comparatively small. The emitted electron is from the K shell usually, but in a number of cases an L shell electron can be involved. The hole left by the ejected electron is filled by another electron dropping from a higher level, a transition which is accompanied by the emission of characteristic x-rays.

2. Compton effect: If the incident photon collides with an essentially free electron, part of its energy can be transferred to the electron. A new gamma-ray with lower energy emerges while the electron receives the rest of the energy. The scattered photons have a continuous energy spectrum since their energy depends on the scattering angle, θ, which takes all values between 0 and 180° and $E_g' \simeq \frac{m\,c^2}{1-\cos\theta}$ where E_g' is the energy of the scattered ray.

When the scattering angle equals 180°, the term 'backscatter' is applied. The Compton effect is important for gamma-rays with energies between .5-5 MeV.

3. _Pair Production_: High energy gamma rays can be absorbed by interaction with the electrostatic field of a nucleus to produce a negatron (e^{-}) and a positron (e^{+}) pair. Pair production occurs for gamma-rays with energy greater than 1.02 MeV $= 2m_{o}c^{2}$ where m_{o} is electron mass. If gamma-ray energy is more than 1.02 MeV, the extra energy is converted into kinetic energy of the pair. Pair production is the predominant mode of interaction with matter when the gamma-ray energy is above 5 MeV. When the positron loses its kinetic energy, it interacts with a negative electron in the absorber and both particles are annihilated forming two photons each with an energy of .51 MeV.

Typical Gamma Spectrum

The decay of ^{24}Na to the ^{24}Mg yields two gamma-rays as it passes to the ground state:

^{24}Na
11
(parent) β^{-}

^{24}Mg* (daughter)
12

1.39 MeV γ(2.76 MeV)

γ(1.37 MeV)

^{24}Mg (stable isotope)
12
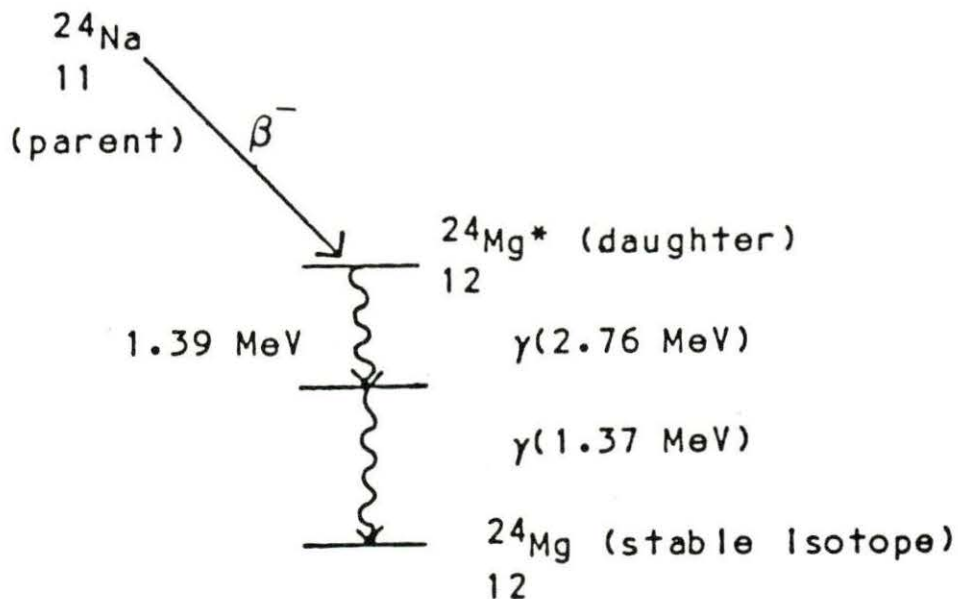
If the resulting gamma-rays enter the detector, a spectrum similar to that shown in Fig. 9 [66] is produced. When gamma-rays emitted by the ^{24}Mg daughter are absorbed in a single event (photoelectric effect), then two photopeaks of energies 1.37 and 2.76 MeV are observed. Gamma rays scattered by the Compton effect produce a continuous Compton spectrum found on the low energy side of each photopeak. At certain energies, where the probability of absorption of the scattered photons is at maximum, a Compton peak shows on the continuous spectrum. The Compton edge is the high energy end of the Compton spectrum immediately below to the photopeak. If the gamma rays energy is more than 1.02 MeV, as in Fig. 9, then pair production can occur. When the positron is subsequently annihilated, two photons of .51 MeV are produced. One or both of these photons may escape from the absorber thus two additional peaks are observed at energies 0.51 and 1.022 MeV below the photopeak energy.

Thus for a 2.76 MeV photon, the pair peaks are formed at E_g-.51 MeV and E_g-2(.51) MeV, i.e., at 2.25 MeV and 1.74 MeV. In some cases, when the gamma ray energy is sufficiently large so that pair production occurs with high probability, it is possible to observe an annihilation radiation peak at .51 MeV due to photons produced by positron annihilation in the medium surrounding the detector.

Interferences

Two main types of interferences in NAA are 1) interfering nuclear reactions, which occur during activation and 2) spectral interference which appear during the detection of the photons.
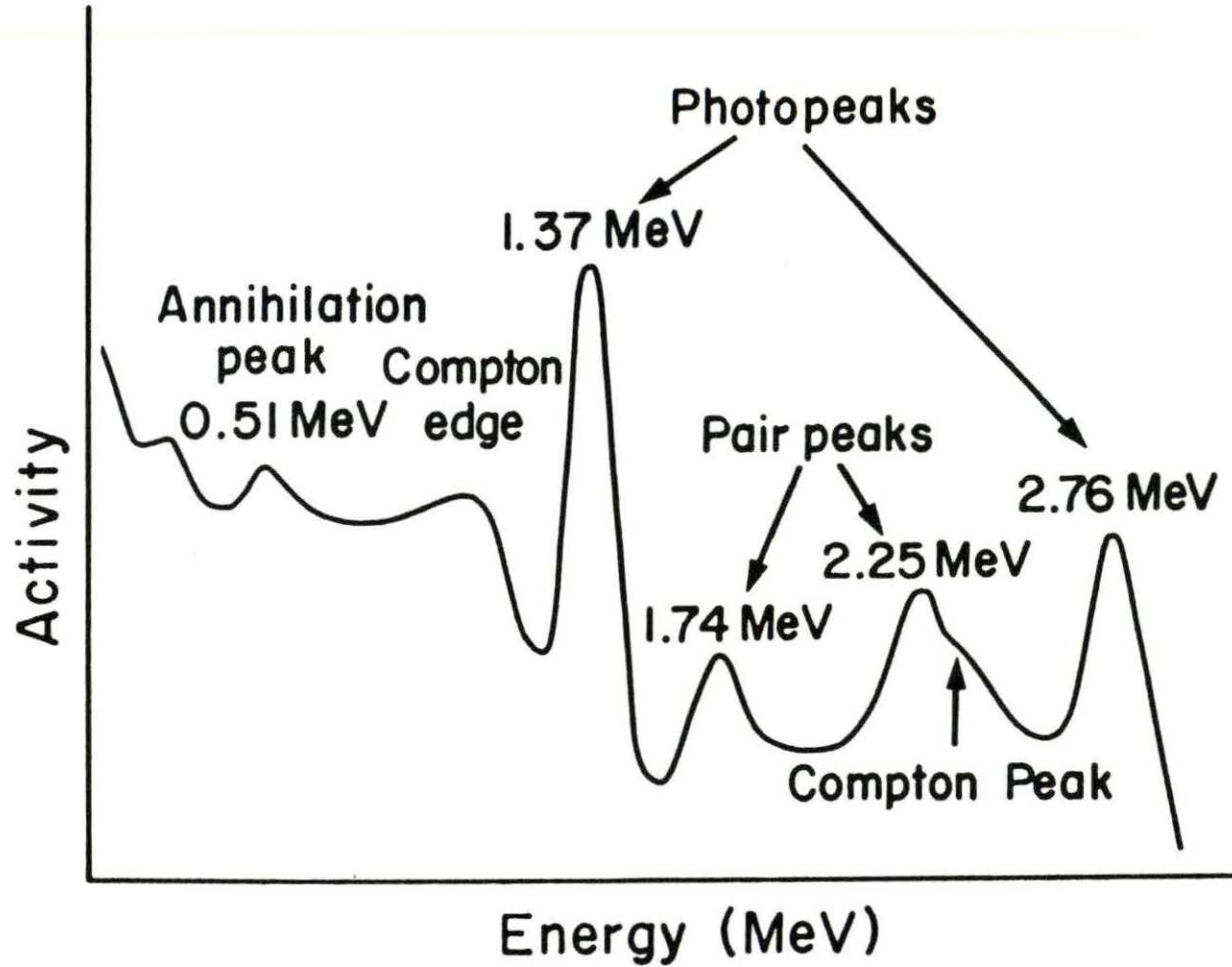
Fig. 9. Gamma-ray spectrum of ^{24}Na obtained with a NaI(Tl) detector

Interfering nuclear reactions

Interfering nuclear reactions are defined as reactions that change
the relationship between the element to be determined and a particular
radionuclide. There are three main types of interfering nuclear
reactions which are listed below and illustrated by examples following
the listing.

1. Primary reactions, the most important of all, involve the
production of the desired radionuclide from elements in the sample other
than the element of interest.

2. Secondary reactions are usually of low probability and involve
secondary particles, released from a nucleus by the action of neutrons,
activating another nucleus to yield the desired radionuclide.

3. Second-order reactions are divided into two subclasses: 1)
reactions that increase the amount of the desired radionuclide by
producing a short-lived product of neighboring element that decays to the
element of interest; and 2) reactions that decrease the amount of the
desired radionuclide as a result of capture by the product nucleus.

Arsenic analysis by NAA displays several types of interference.
Thermal neutron irradiation of arsenic leads to the useful reaction
product: $^{75}As(n,\gamma)^{76}As$. Arsenic 76 is also produced by the primary
interfering reactions: $^{76}Se(n,p)^{76}As$ and $^{79}Br(n,\alpha)^{76}As$. Secondary
interference produces ^{76}As from Selenium: $^{77}Se(\gamma,p)^{76}As$. An example of
second order interference is $^{74}Ge(n,\gamma)^{75}Ge \longrightarrow {}^{75}As (n,\gamma)^{76}As$ which
increases the amount of product. The (n,p), (n,α) and (γ,p) reactions
have small cross-sections so their interference is usually not
significant in comparison with (n,γ) reactions.

Spectral interferences

The area under a photopeak in a gamma spectrum is usually measured
in order to determine the activity of a radionuclide. This can be
accomplished easily if one of the photopeaks associated with a particular
isotope is well defined and free from interference from nearby peaks.
There are several methods which can remove the interference with the
photopeaks of interest and any one could be selected: (1) spectrum
stripping; (2) peak fitting; (3) cooling time selection and (4) analysis
of decay curves.

Most of these methods are facilitated by the use of computer.

1. In spectrum stripping, i.e., subtracting a pure reference
spectrum, multiplied by an appropriate constant from the measured sum of
spectra, thus the interfering radionuclide spectrum is stripped from the
sample spectrum. Another procedure is to calculate an interferent
contribution to the area associated with a certain photopeak of the
sample element, using tabulated values of the relative intensities of two
different photopeaks of the interferent, and the detection efficiency
factors at both gamma-energies. One of the photopeaks will be measured
and the second (which interferes), inferred from the experimental
results.

2. Peak fitting: Many mathematical models can be used to simulate
the shape of a peak without interference, which permits the resolution of
two or more overlapping peaks.

3. Cooling time selection: Cooling time is chosen to allow short
half-life interferents to decay. The total activity, A_t, of both the
element of interest, A_e, and the interferent, A_t, can be measured after a

cooling time, T_1,

Thus, $$A_t(T_1) = A_e(T_1) + A_i(T_1)$$

and again after another long cooling period, T_2, such that the activity of the element has decayed to "zero" and only the interferent remains with activity $A_i(T_2)$. The activity of the standard sample of the interfering radionuclide, $A_{i(s)}$ is also measured after the same two cooling times enabling $A_i(T_1)$ to be calculate from the ratio:

$$\frac{A_i(T_1)}{A_{is}(T_1)} = \frac{A_i(T_2)}{A_{is}(T_2)}$$

The activity $A_i(T_i)$ of the interferent is then subtracted from the total activity to yield the activity of the element of interest.

4. Analysis of decay curves: after irradiation, the total activities of elements of interest and the interfering radionuclides are measured at different times and the logarithm of the total activity is plotted against time. If the half-lives of the radionuclides are sufficiently different, the decay curve, after some time, becomes a straight line on the semi-log plot, and the associated half-life corresponds to the longer half-life. If this line is extrapolated to $t = 0$, the intercept would be this radionuclide's initial activity. If only two radionuclides were present, then subtraction of the extrapolated values of the activity of the longest half-life nuclei from the experimental curve will give another straight line, and its intercept on the $t = 0$ axis would be the initial activity of the shorter half-life isotope.

EXPERIMENTAL DETAILS

Samples Identification

Fifty-one samples were provided by the DGMR at Jeddah in the KSA, which has an exploration program with the USGS. The samples were obtained as a result of an exploration program carried out under contract by the United States Geological Survey, USGS. These samples were collected from boreholes at four different locations, namely Jabal Tawlah, Jabal Hamra, Umm Al-Birak, and Jabal Sayid, in the Kingdom. These samples were later ground to a very fine powder. Sixteen samples were selected randomly, four from each area for the NAA. The samples were originally provided with six digit identification numbers. For convenience, they were renumbered serially. Table 2 depicts the identification scheme and the origin of each sample.

Preliminary Investigation

To select optimum time for neutron irradiation, decay and counting an estimate of the quantitative elemental composition of the samples was required. To carry out an estimate, some of the samples were analyzed by XRF and XRD techniques. The former (XRF) provided information about the major constituents of the samples while the later (XRD) provided clues to the identification of the minerals included in the ores.

For precise detection of U, Th, and what other elements might be encountered, standard GS, XRF, and XRD techniques were employed in the preliminary investigation.

The objective of each technique was as follows:

- GS was carried out to increase the concentration of the heavy

Table 2. Samples identification scheme

Sample I.D. Number		Identification by DGMR/USGS	Location in Saudi Arabia
3.	W-1	311089	Jabal Tawlah
4.	W-3	311094	Jabal Tawlah
5.	W-9	311103	Jabal Tawlah
6.	W-14	311101	Jabal Tawlah
7.	W-16	311692	Jabal Hamra
8.	W-18	311694	Jabal Hamra
11.	W-20	311696	Jabal Hamra
12.	W-23	311700	Jabal Hamra
13.	W-24	348009	Umm Al-Birak
16.	W-26	348222	Umm Al-Birak
17.	W-30	348333	Umm Al-Birak
18.	W-33	348337	Umm Al-Birak
19.	W-39	336680	Jabal Sayid
20.	W-42	336684	Jabal Sayid
21.	W-45	336687	Jabal Sayid
22.	W-48	336690	Jabal Sayid

fraction material, and was intended to increase the U and Th content in the sample. This was done at the Department of Environmental Health and Safety ventilated hoods in the Nuclear Engineering Laboratory at ISU, Ames, Iowa.

The sample was divided into 2 parts, one for the separation and the other for NAA. Bromoform (a liquid with density 2.78) was poured into a separatory funnel and one portion of the sample was mixed with it and left for about 15-20 minutes. The heavy fraction settled to the bottom of the funnel and the light fraction floated on the surface of the Bromoform. Each fraction was collected separately on a filter paper and allowed to dry under a heat lamp. Four samples, one from each location, were treated in this way. The effectiveness of the GS was explored using NAA.

• XRF was done for one of the samples using chromium (Cr), radiation and again with tungsten (W), radiation. This was performed in the ERI Service Group Laboratories in the Town Engineering building at Iowa State University.

• XRD was conducted in the Earth Sciences (Geology) Department laboratory and by the chemistry branch of the Chemistry Department. This analysis was done on the four GS samples.

Sample Preparation Encapsulation and Irradiation for NAA

Samples were weighed and poured into high purity quartz vials type T-21 which were then sealed under vacuum using a torch. The sample weights were between 25-60 mg.

To avoid contamination, gloves were used to handle vials, a scriber

was used to number the vials for identification, and a motor vibrator to concentrate sample material at the bottom. The samples and the standard were wrapped with aluminum foil to form bundles which fit inside the aluminum irradiation cans. Samples were irradiated at the MURR Facility for 24 hours with a flux of 5×10^{13} neutrons $(cm^2 \; sec)^{-1}$.

Standards

The standard reference material used was obtained from the NBS and is identified as SRM-1633a, Fly Ash. Three replicates were used. They were bundled in aluminum foil with the samples so that the same irradiation conditions were experienced by all components, thus minimizing corrections for flux inhomogeneities.

Gamma-ray detection

All instruments and equipment used were provided by the MURR Facility.

Following a decay of 8640 min ($\approx$ six days) after irradiation, the first activity measurements for all samples were taken using 3000 sec counting periods. A second set of activity measurements were taken following a decay 36864 min ($\approx$ 25.6 days) after irradiation. In this case, the samples were counted for 7200 sec per sample.

All samples were counted using an automatic sample changer-detector system available at the MURR Facility.

The equipment used for the NAA experiment included:

1. A Princeton Gamma Tech Ge(Li) detector, serial number 2419, with an efficiency of 17% relative to a "3x3" Na-I detector. The detector resolution is about 1.8 keV for the 1332-keV Co^{60}

ray. The detector operates at 2 kV volts. (Preamplifier was attracted to and was a part of the detector assembly).

2. A Nuclear Chicago sample changer (automatic) modified by MURR for use with Ge detectors. About 8" of lead shielding, prevents photons from samples waiting to be counted from reaching the detector

3. An ORTEC 459 High Voltage Supply.

4. An ORTEC 572 Amplifier.

5. A Nuclear Data ND570 ADC having 8192 channels (calibrated to have a gain of about 0.38 keV per channel).

The output of the ADC went into the memory of a Nuclear Data 6620 analyzer system which controlled all counting, sample changing and storage of data on both a disk, using a Winchester drive, and on a magnetic tape.

The ND6620 analyzer was used to perform the analysis after all standards and samples have been counted. Samples deadtimes were kept below 20% to minimize errors by pulse-pileup. A pulse-pileup correction was made at the time of analysis.

The experimental setup is shown in Fig. 10. The actual procedure is illustrated in Figs. 11-18.
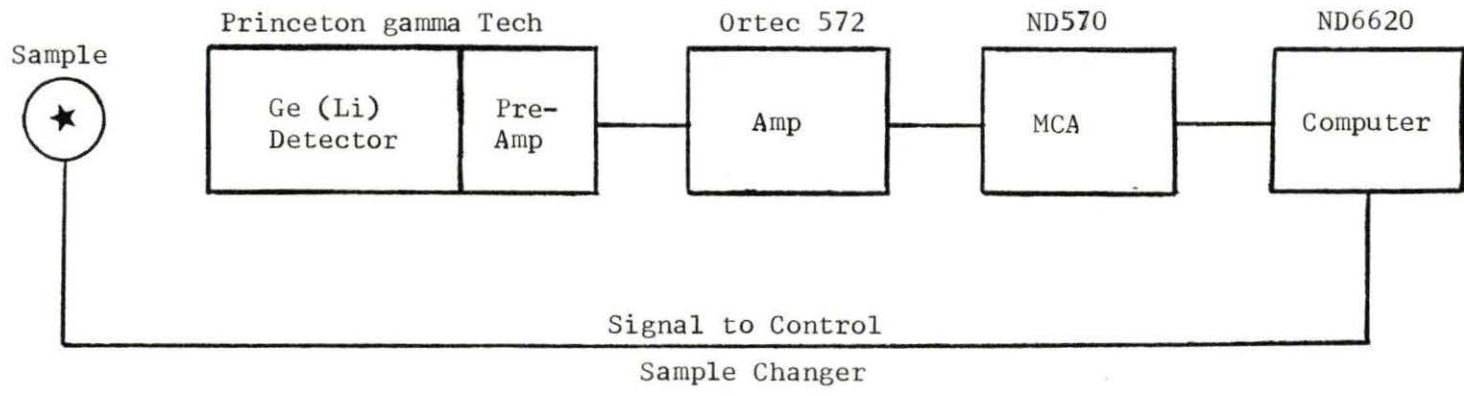
Fig. 10.   Experimental setup
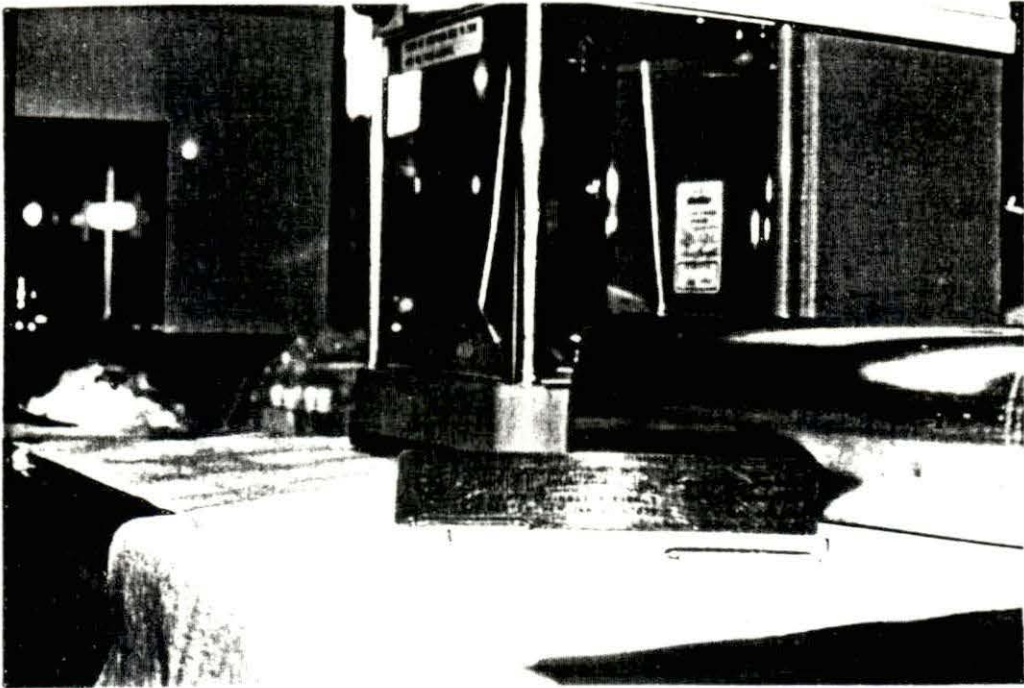
Fig. 11. Sample preparation I: Quartz vial (1), aluminum
can (2), analytical balance (3)
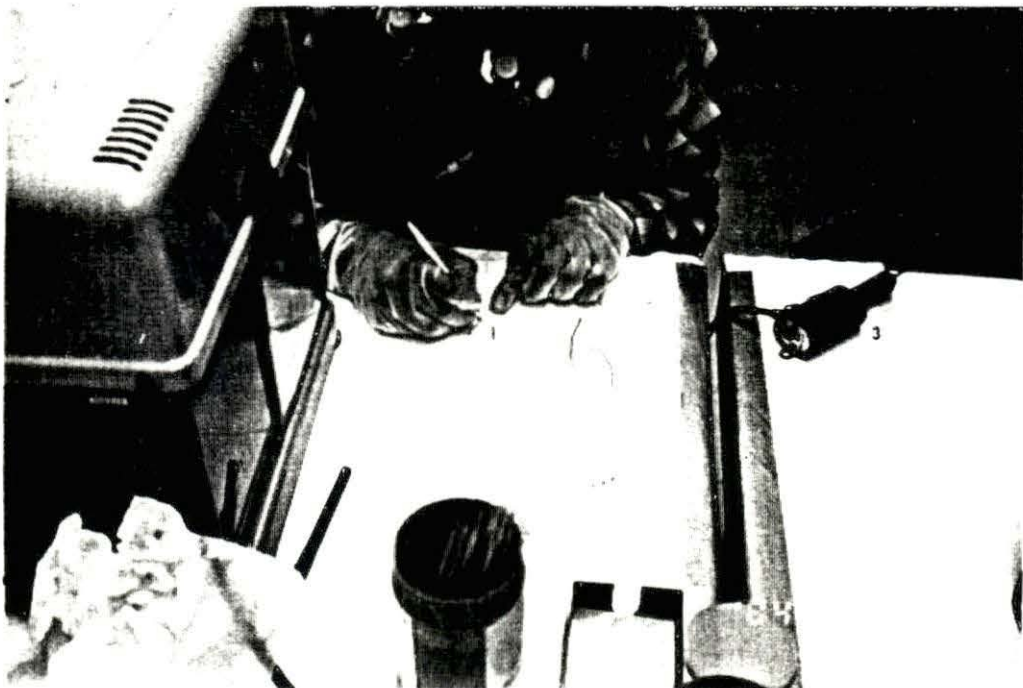
Fig. 12. Sample preparation II: numbering vial (1) for
identification with scribe (2) and vibrator (3)
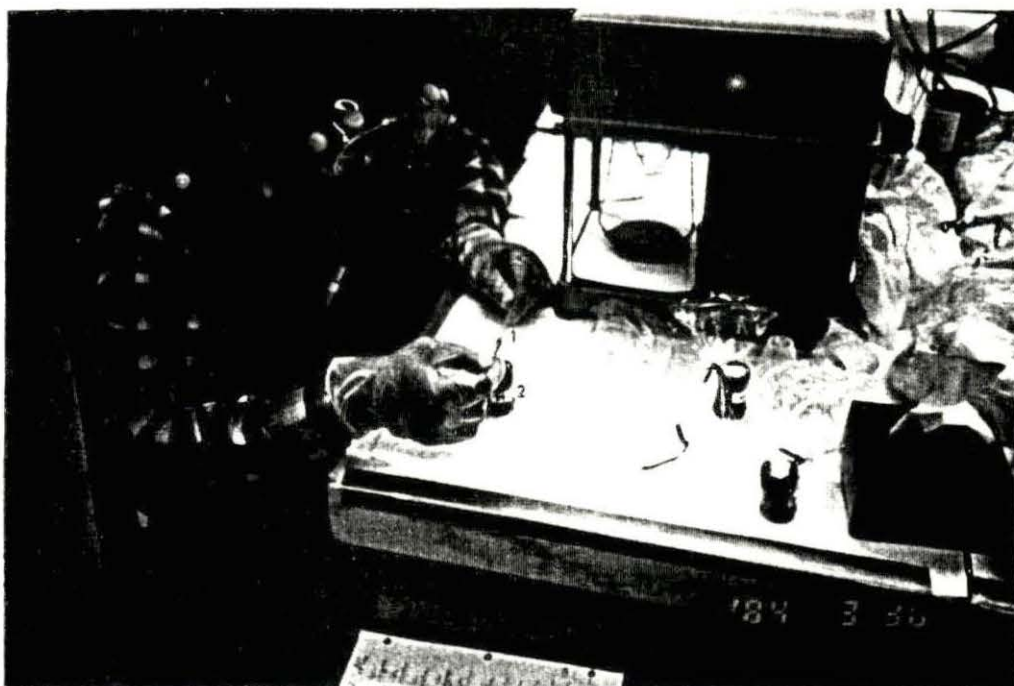
Fig. 13. Sample preparation III: filling vial (1) with
sample material (2)
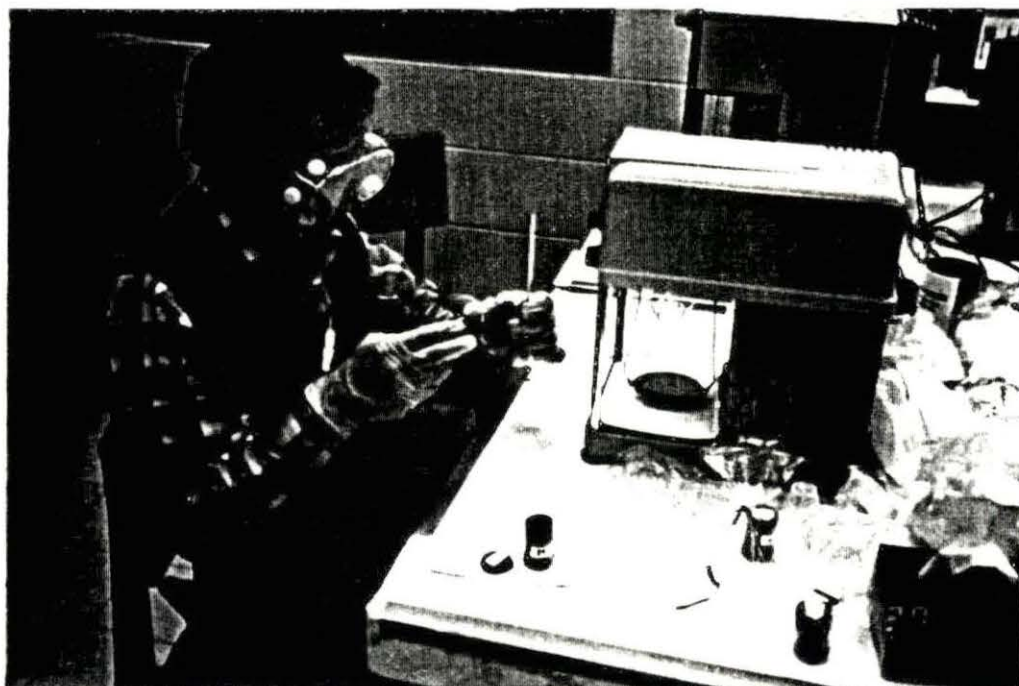
Fig. 14. Sample preparation IV: sample compaction in vial
(2) using motor vibrator (1)

47

Fig. 15. Sample preparation V: sample weighing using
analytical balance (1)
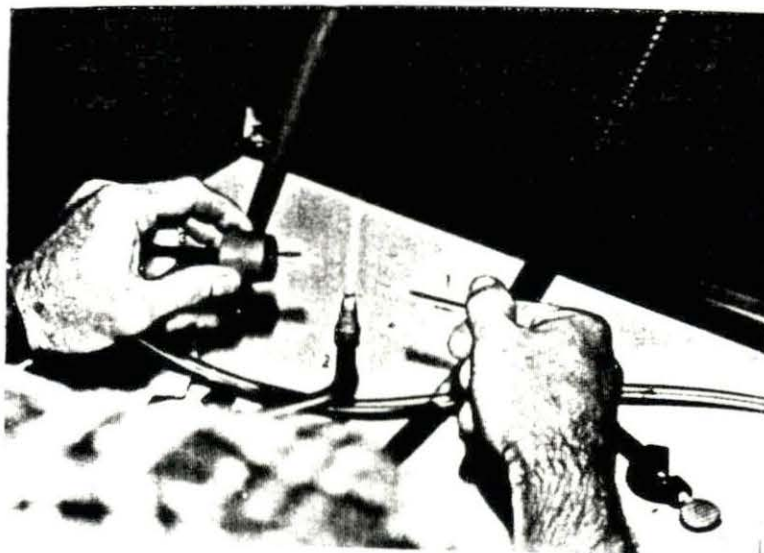
Fig. 16. Sample preparation VI: vial (1) being sealed
using torch (2) and vacuum system (3)
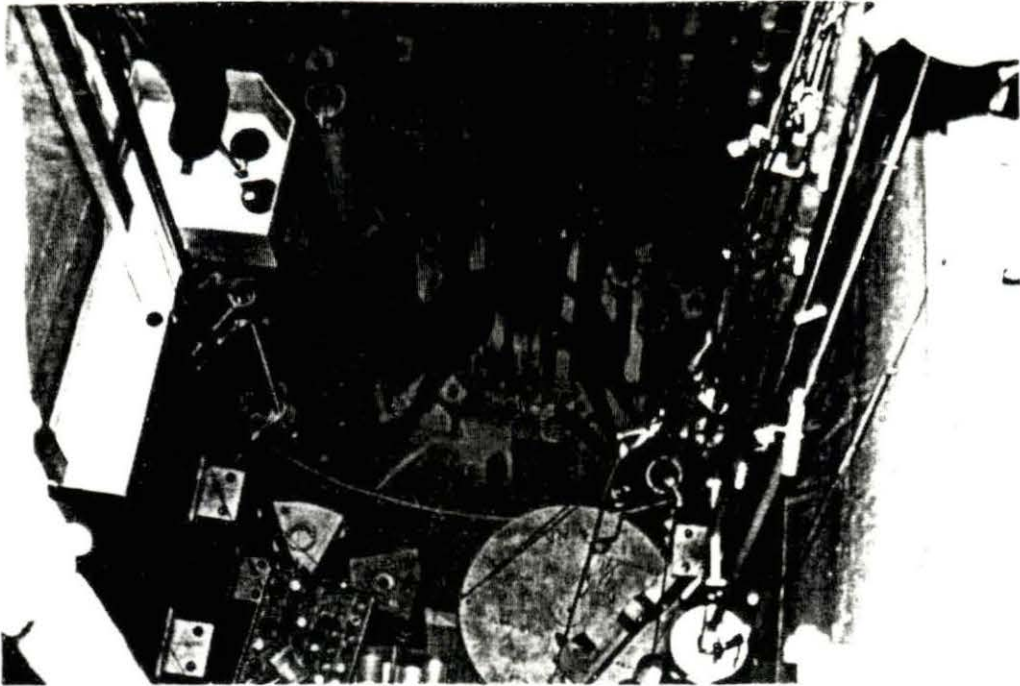
Fig. 17. MURR core: Irradiation locations (1) and (2)
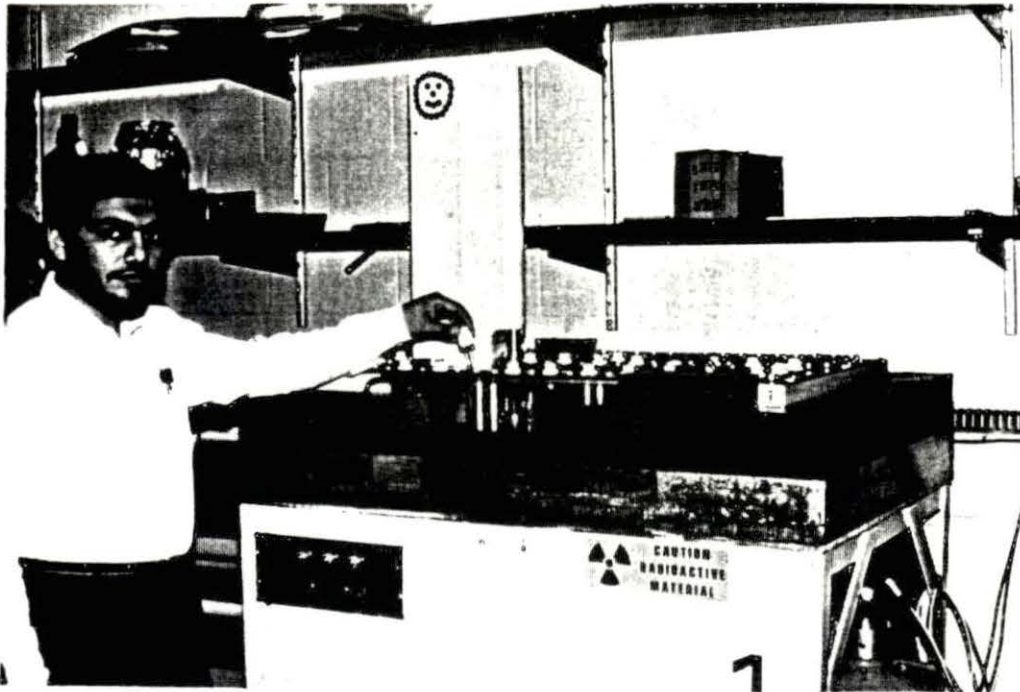
Fig. 18. Automatic counting system: sample changer (1) and Ge(Li) detector (2)

RESULTS

Gravimetric Separation

Four samples were selected for enhancement by gravimetric means. Each sample was divided into two roughly equal portions and one portion from each sample was separated into a heavy and a light fraction. Thus for each sample there was a heavy fraction, a light fraction and an unseparated portion. NAA of the sets of three revealed an enhancement ratio of two or more for heavy elements in the heavy fraction, a similar enhancement ratio for light element in the light fraction and little if any change for intermediate weight cases. Tables 3a and 3b show the gravimetric enhancement for all the elements detected, and for measurements made after both decay periods of about 6 and 25.6 days.

X-Ray Fluorescence

An optimized NAA is possible only if prior investigations have identified the elements present and their approximate concentrations. With this information, suitable irradiation and decay time combinations can be chosen to permit the best possible assay for the elements involved. The initial search for information can be done by repeated NAA or by using other analytical techniques.

Two XRF analyses were conducted using chromium and tungsten radiation. The "chromium" analysis revealed Si, Al, K, Fe, Ca, and Ti to be major constituent elements, Cu, Zn, Ni, Ba, Zr, Sn, Rb, Y, and Nb as minor elements, and Mg and Na as trace elements. The "tungsten" analysis confirmed the elements listed above, and in addition revealed Thorium. Uranium was not found.

Table 3a. Concentrations (ppm) obtained in the gravimetric enhancement study ($t_d \gtrsim 6$ days after irradiation)

Sample I.D. Number	Na-24 1368.6 KeV	As-76 559.10 KeV	Br-82 619.10 KeV	Mo-99 140.4 KeV	Sb-122 564.1 KeV
1. H-1[a]	18582 + 421	16.14 + 1.27	22.99 + 7.36	106.5	4.941 + 0.297
2. L-1[b]	21053 + 479	18.30 + 1.61	6.374+ 2.275	54.33	2.787 + 0.295
3. W-1[c]	21098 + 480	< 3.998[d]	< 3.365[d]	61.24	2.401 + 2.58
9. H-20	1501 + 64	764.9 + 12.7	282.9 + 89.8	60.95	27.37 + 1.07
10. L-20	1567 + 59	64.86 + 3.09	81.25 + 26.8	17.68	8.784 + 0.537
11. W-20	1311 + 51	48.45 + 2.67	< 5.031[d]	22.96	8.024 + 0.451
14. H-26	16113 + 374	11.70 + 2.58	48.99 + 15.66	377.41	< 1.027[d]
15. L-26	14503 + 336	< 5.850[d]	52.32 + 16.66	139.61	< .8146[d]
16. W-26	16229 + 374	< 5.891[d]	< 3.109[d]	152.92	< .7698[d]

[a]H: Heavy fraction.

[b]L: Light fraction.

[c]W: Unseparated fraction.

[d]< Peak not seen, ∴ detection limit was calculated so sample has less than this amount.

Table 3a. (Continued)

Sample I.D. Number	La-140 487 KeV	Sm-153 69.7 KeV	Yb-175 396.3 KeV	W-187 479.6 KeV	U(Np-239) 228.2 KeV
1. H-1	150.3 ± 1.3	120.1 ± 2.7	1067 ± 43	< 4.767	212.7 ± 15.8
2. L-1	60.37 ± 0.85	58.95 ± 1.54	502.3 ± 20.2	< 4.991	95.06 ± 7.24
3. W-1	71.89 ± 0.90	71.01 ± 1.75	553.9 ± 22.3	< 4.832	112.2 ± 8.5
9. H-20	1350 ± 10	315.0 ± 7.0	267.2 ± 10.8	21.41 ± 5.54	109.7 ± 8.6
10. L-20	1079 ± 8	232.6 ± 5.2	115.9 ± 4.8	< 8.072	63.91 ± 5.26
11. W-20	1090 ± 8	226 ± 5	115.1 ± 4.7	< 5.474	59.72 ± 4.84
14. H-26	790.2 ± 5.8	205.7 ± 4.6	438.9 ± 17.7	<12.57	109.7 ± 8.6
15. L-26	263.8 ± 2.1	67.80 ± 1.63	23.46 ± 1.13	5.592 ± 1.925	52.71 ± 4.16
16. W-26	316.1 ± 2.5	81.27 ± 1.94	29.10 ± 1.34	< 5.898	53.75 ± 4.25

Table 3b. Concentrations (ppm) gravimetric enhancement study ($t_d \approx 25.6$ days after irradiation)

Sample I.D. Number	Sc-46 889.3 KeV	Cr-51 320.1 KeV	Fe-59 1099.2 KeV	Co-60 1332.5 KeV	Ni(Co-58) 810.8 KeV
1.H[a]-1	1.559 ± .049	296.2 ± 8.4	26063 ± 434	3.223 ± .254	< 198.5[b]
2.L[c]-1	.8731 ± .037	218.3 ± 6.8	15234 ± 319	2.064 ± .167	< 121.6[b]
3.W[d]-1	1.001 ± .039	218.8 ± 6.8	15905 ± 323	1.857 ± .158	< 166.1[b]
9.H-20	.1209 ± .0359	1054.0 ± 21.0	290732 ± 3431	9.157 ± .319	211.1 ± 97.6
10.L-20	.1021 ± .0257	241.5 ± 6.5	71493 ± 932	1.254 ± .142	127. ± 61.0
11.W-20	.1535 ± .0215	247.3 ± 6.2	71377 ± 901	1.453 ± .133	< 110.1[b]
14.H-26	.9619 ± .043	101.6 ± 8.5	89102 ± 1131	2.844 ± .233	< 201.6[b]
15.L-26	.2052 ± .0196	117.0 ± 3.8	19639 ± 335	1.378 ± .107	< 108.6[b]
16.W-26	.2612 ± .0241	117.4 ± 3.9	22015 ± 361	1.398 ± .109	< 115.7[b]

[a]H: Heavy fraction.

[b]< Peak not seen, .∴. a detection limit was calculated, so sample has less than this amount.

[c]L: Light fraction.

[d]W: Unseparated fraction.

Table 3b. (Continued)

Sample I.D. Number	Zn-65 1115.5 KeV	Rb-86 1076.6 KeV	Sn-85 514.00 KeV	Sb-124 1691.00 KeV	Cs-134 604.7 KeV
1.H-1	2764 $\pm$ 92	49.94 $\pm$ 13.32	< 646.0	4.429 $\pm$ .361	< 1.235
2.L-1	3282 $\pm$ 109	55.81 $\pm$ 11.77	< 517.6	3.060 $\pm$ .230	< 1.025
3.W-1	3493 $\pm$ 116	83.15 $\pm$ 12.28	< 521.2	3.317 $\pm$ .248	< 1.030
9.H-20	1735 $\pm$ 59	169.5 $\pm$ 22.3	< 734.4	27.44 $\pm$ 1.12	< 1.659
10.L-20	966 $\pm$ 33.6	313.2 $\pm$ 21.0	< 467.7	9.540 $\pm$ .482	1.934 $\pm$ .258
11.W-20	982 $\pm$ 33.7	349.2 $\pm$ 21.6	< 412.8	8.933 $\pm$ .436	1.63 $\pm$ .226
14.H-26	3958 $\pm$ 131	85.93 $\pm$ 14.61	< 887.5	< .7546	< 1.481
15.L-26	377.1 $\pm$ 14.1	250.2 $\pm$ 16.1	< 318.5	1.012 $\pm$ .177	< .6279
16.W-26	451.5 $\pm$ 16.5	250.5 $\pm$ 16.6	< 342.6	< .4371	< .5260

Table 3b. (Continued)

Sample I.D. Number	Ba-131 216 KeV	Ce-141 145.4 KeV	Nd-147 91.1 KeV	Eu-152 1408 KeV	Tb-160 879.4 KeV
1.H-1	12022 + 651	628.9 + 7.5	227.7 + 18.2	4.862 + 0.144	88.95 + 5.18
2.L-1	6268 + 372	263.2 + 4.4	126.1 + 15.2	2.365 + 0.089	45.10 + 2.64
3.W-1	7304 + 419	304.8 + 4.5	175.3 + 15.4	2.649 + 0.098	50.75 + 2.97
9.H-20	11535 + 630	3697 + 34	1471 + 80	40.91 + 0.72	77.82 + 4.55
10.L-20	5504 + 319	2747 + 25	1193 + 65	24.72 + 0.46	38.85 + 2.28
11.W-20	5729 + 320	2766 + 25	1206 + 65	24.93 + 0.45	38.47 + 2.25
14.H-26	10046 + 609	1991 + 19	985.4 + 58.4	19.35 + 0.37	68.63 + 4.01
15.L-26	1761 + 135	699.3 + 7	270.8 + 16.9	4.259 + 0.114	12.48 + 0.76
16.W-26	1875 + 142	811.9 + 7.8	324.5 + 19.8	4.956 + 0.124	13.35 + 0.81

Table 3b. (Continued)

Sample I.D. Number	Yb-169 197.9 KeV	Hf-181 482.2 KeV	Ta-182 1221.4 KeV	Th(Pa-233) 312 KeV	Lu-177 208.4 KeV
1.H-1	999.8 ± 31.1	1508 ± 45	458.2 ± 18.7	1179 ± 13	136.7 ± 8.2
2.L-1	470.3 ± 14.1	887.2 ± 26.3	192.4 ± 7.9	580.6 ± 6.2	65.6 ± 3.95
3.W-1	528.9 ± 16.5	971.3 ± 28.8	221.8 ± 9.1	685.6 ± 7.3	73.09 ± 4.4
9.H-20	293.9 ± 9.2	689.2 ± 20.5	211.9 ± 8.7	248.1 ± 2.7	35.02 ± 2.12
10.L-20	128.8 ± 4.1	329.1 ± 9.8	79.49 ± 3.29	131.7 ± 1.5	15.72 ± 0.96
11.W-20	126.0 ± 4.0	325.7 ± 9.7	78.07 ± 3.22	130.6 ± 1.5	15.38 ± 0.94
14.H-26	451.3 ± 14.1	184 +154	157.6 ± 6.5	89.99 ± 1.29	76.80 ± 4.64
15.L-26	42.06 ± 1.37	211.5 ± 6.3	96.76 ± 3.98	19.72 ± 0.35	3.584 ± 0.240
16.W-26	48.44 ± 1.57	263.0 ± 7.8	108.2 ± 4.5	22.35 ± 0.36	4.648 ± 0.313

Although the preliminary analysis was considered adequate for the intended purpose, more information could have been obtained if a more complete set of XRF standards had been available. The decision to use the MURR (rather than the UTRIO) made this preliminary analysis unnecessary. The results are included here for completeness.

X-Ray Diffraction

An XRD study was carried out on the gravimetrically separated samples in the hope that heavy minerals associated with uranium might be found. The data bank used with the XRD system did not contain all the appropriate and needed standards, so the results were limited. Lines associated with two uranium compounds were found in only one sample (H-26).

NAA

Table 4 shows the experimentally determined Uranium and Thorium concentrations in ppm for all samples investigated. The techniques uncertainty was one standard deviation. High concentrations of Th were found in all samples except samples W-26 at Umm Al-Birak and W-48 at Jabal Sayid where the concentrations were small and sample W-45 at Jabal Sayid where the concentration was even lower. The highest Th concentration was found in samples coming from Jabal Tawlah area, W-3, W-9, W-14, and W-1, and also in two W-39 and W-42 of the four samples from the Jabal Sayid location.

U was present in all four areas. At Jabal Tawlah it was detected in high concentration in one sample, W-9, and was below the LDL for the

Table 4.   Uranium and Thorium concentrations in ppm

Sample I.D. Number		Location in Saudi Arabia	U(Np-239) 228.2 KeV	Th(Pa-233) 312.00 KeV
1	H[a]-1	Jabal Tawlah	212.7 $\pm$ 15.8	1179 $\pm$ 13.
2	L[b]-1	Jabal Tawlah	95.06 $\pm$ 7.24	580.6 $\pm$ 6.2
3	W[c]-1	Jabal Tawlah	112.2 $\pm$ 8.5	685.6 $\pm$ 7.3
4	W-3	Jabal Tawlah	< 5.847[d]	1043 $\pm$ 11.0
5	W-9	Jabal Tawlah	< 5.623[d]	901.7 $\pm$ 9.6
6	W-14	Jabal Tawlah	< 6.527[d]	749.5 $\pm$ 8.0
7	W-16	Jabal Hamra	68.76 $\pm$ 5.57	264.8 $\pm$ 2.9
8	W-18	Jabal Hamra	60.71 $\pm$ 4.94	416.8 $\pm$ 4.5
9	H-20	Jabal Hamra	109.71 $\pm$ 8.6	248.1 $\pm$ 2.7
10	L-20	Jabal Hamra	63.91 $\pm$ 5.26	131.7 $\pm$ 1.5
11	W-20	Jabal Hamra	59.72 $\pm$ 4.84	130.6 $\pm$ 1.5
12	W-23	Jabal Hamra	73.09 $\pm$ 5.80	158.8 $\pm$ 1.7
13	W-24	Umm Al-Birak	104.4 $\pm$ 7.9	152.8 $\pm$ 1.7
14	H-26	Umm Al-Birak	109.7 $\pm$ 8.6	89.99 $\pm$ 1.29
15	L-26	Umm Al-Birak	52.71 $\pm$ 4.16	19.72 $\pm$ 0.35
16	W-26	Umm Al-Birak	53.75 $\pm$ 4.25	22.35 $\pm$ 0.36
17	W-30	Umm Al-Birak	31.51 $\pm$ 2.71	140.2 $\pm$ 1.5
18	W-33	Umm Al-Birak	46.15 $\pm$ 3.81	163.0 $\pm$ 1.8
19	W-39	Jabal Sayid	427.5 $\pm$ 31.8	743.2 $\pm$ 7.9
20	W-42	Jabal Sayid	556.2 $\pm$ 41.2	791.8 $\pm$ 8.5
21	W-45	Jabal Sayid	< 2.761[d]	4.752 $\pm$ 0.19
22	W-48	Jabal Sayid	22.75 $\pm$ 2.21	24.04 $\pm$ 0.33

[a]H: Heavy fraction.

[b]L: Light fraction.

[c]W: Unseparated fraction.

[d]<: Peak not seen, a detection limit was calculated, so sample has less than this amount.

other three samples from this location. The highest U concentration was
found for two of the four Jabal Sayid location samples only, W-42 and
W-39. High concentrations of U were found also in samples W-1 at Jabal
Tawlah and W-24 at Umm Al-Birak.

Tables 5a and 5b show all NAA determined elemental concentrations (in
ppm), for measurements following decay time of about 6 and 25.6 days,
respectively.

Comparison of DGMR Results with the Results Obtained
with the ISU/MURR Results

The DGMR laboratory results (SQSA, 6 Step D-C-Arc) are found in
Tables 6-9, NAA results in Tables 5a and 5b. The available results for U
and Th obtained by different techniques are compared in Table 10. A
comparison of the concentrations all elements determined both SQSA and
NAA techniques is presented in Tables 11 - 14.

DGMR Laboratory carried out two analyses, XRF in search of Th and
Ta, and Fluorometric in search of U, in Jabal Tawlah samples. The
results are shown in Table 6.

Atomic Absorption analysis was carried out by the Bondar-Clegg
Company looking for Th and Ce, in Jabal Sayid samples. The results are
shown in Table 9. They did not search for U in Jabal Sayid samples.

The Th concentrations in samples W-3 and W-9 at Jabal Tawlah,
as determined by NAA, were the highest found. The NAA determinations
were about 200 ppm higher than results obtained by SQSA, as reported by
DGMR. The same result was obtained with sample W-39 at Jabal Sayid. The
results for the other three samples at Jabal Sayid follow. For sample W-

Table 5a.   Concentrations in ppm of elements found by NAA $t_d \approx 6$ days after irradiation

Sample I.D. Number	Na-24 1368.6 KeV	As-76 559.10 KeV	Br-82 619.10 KeV	Mo-99 140.40 KeV	Sb-122 564.1 KeV
3. W-1[a]	21098 + 480	< 3.998[b]	< 3.365[b]	61.24 + 24.9	2.401 + 2.58
4. W-3	13489 + 308	14.52 + 1.25	< 2.808[b]	< 36.909[b]	15.47 + 0.57
5. W-9	13860 + 316	7.058 + 1.076	< 2.105[b]	< 36.296[b]	2.520 + .237
6. W-14	27808 + 631	30.78 + 1.78	< 3.678[b]	< 41.187[b]	1.955 + .284
7. W-15	743.5 + 42.0	103.1 + 3.3	7.671 + 2.859	40.85 + 19.24	8.155 + .507
8. W-18	779.6 + 51.7	66 65 + 2.61	< 4.626[b]	32.39 + 16.99	6.992 + .439
11. W-20	1311 + 51	48.45 + 2.67	< 5.031[b]	22.96 + 15.11	8.024 + .451
12. W-23	1212 + 54	35.71 + 2.62	< 4.908[b]	55.19 + 20.52	9.612 + .533
13 W-24	12234 + 286	< 7.506[b]	< 4.324[b]	51.37 + 23.1	1.027 + .309
16. W-26	16229 + 374	< 5.891[b]	< 3.109[b]	152.92 + 28.4	< 0.7698[b]
17. W-30	17272 + 398	44.01 + 2.11	< 3.563[b]	22.33 + 11.08	< 0.7504[b]
18. W-33	23861 + 546	44.21 + 2.30	4.153 + 1.816	73.37 + 18.20	1.033 + .269
19. W-39	2669 + 108	< 13.26[b]	8.239 + 3.609	156.23 + 78.5	2.164 + .508
20. W-42	7315 + 198	< 12.30[b]	< 7.261[b]	232.47 +104.5	< 1.489[b]
21. W-45	22146 + 509	< 4.545[b]	< 3.120[b]	< 19.55[b]	< 0.6111[b]
22. W-48	15864 + 369	< 5.406[b]	< 3.386[b]	16.68 + 8.66	< 0.6677[b]

[a]W: Unseparated fraction.

[b]< Peak not seen, .˙. a detection limit was calculated, so sample has less than this amount.

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Table 5a. (Continued)

Sample I.D. Number	La-140 487.00 KeV	Sm-153 69.7 KeV	Yb-175 396.30 KeV	W-187 479.6 KeV	U(Np-239) 228.20 KeV
3. W-1	71.89 + 0.90	71.01 + 1.75	553.9 + 22.3	< 4.832	112.2 + 8.5
4. W-3	1.877 + 0.423	< 2.441	980.4 + 39.4	< 5.281	< 5.847
5. W-9	3.214 + 0.409	< 2.432	1289 + 52	< 5.087	< 5.623
6. W-14	10.80 + 0.60	< 2.821	1166 + 47	< 5.839	< 6.527
7. W-15	1643 + 12	276.0 + 6.1	120.2 + 5.0	< 7.289	68.76 + 5.57
8. W-18	1473 + 10	249.6 + 5.5	111.7 + 4.6	< 6.850	60.71 + 4.94
11. W-20	1090 + 8.	227.0 + 5.0	115.1 + 4.7	< 5.474	59.72 + 4.84
12. W-23	1762 + 12	314.2 + 6.9	151.1 + 6.2	< 7.975	73.09 + 5.80
13. W-24	796.0 + 5.8	126.9 + 2.9	76.26 + 3.21	11.95 + 3.49	104.4 + 7.9
16. W-26	316.1 + 2.5	81.27 + 1.94	29.10 + 1.34	< 5.898	53.75 + 4.25
17. W-30	451.9 + 3.4	102.0 + 2.3	68.29 + 2.82	< 5.821	31.51 + 2.71
18. W-33	859.1 + 6.2	180.3 + 4.0	53.63 + 2.29	11.13 + 3.25	46.15 + 3.81
19. W-39	2357 + 17	1605 + 35	377.2 + 15.2	< 12.64	427.5 + 31.8
20. W-42	1518 + 11	936.0 + 20.3	593.3 + 23.9	< 11.94	556.2 + 41.2
21. W-45	5.988 + .439	3.812 + 0.488	4.860 + .441	< 4.555	< 2.761
22. W-48	143.0 + 1.4	66.86 + 1.62	31.30 + 1.38	< 5.153	22.75 +2.21

Table 5b. Concentrations in ppm of elements by NAA $t_d \approx 25.6$ days after irradiation

Sample I.D. Number	Sc-46 889.30 KeV	Cr-51 320.10 KeV	Fe-59 1099.2 KeV	Co-60 1332.5 KeV	Ni(Co-58) 810.8 KeV
3. W-1[a]	1.001 + 0.039	218.8 + 6.8	15905 + 323	1.857 + 0.158	< 166.1[b]
4. W-3	0.5845 + 0.0362	111.7 + 6.8	14659 + 325	1.159 + 0.179	< 149.8[b]
5. W-9	1.238 + 0.038	163.9 + 7.1	16506 + 312	1.901 + 0.179	< 114.1[b]
6. W-14	7.472 + 0.135	271.8 + 8.3	21955 + 394	10.06 + 0.29	< 184.6[b]
7. W-16	0.1601 + 0.0246	203.8 + 5.5	67096 + 855	1.548 + 0.141	< 107.7[b]
8. W-18	0.1665 + 0.0215	242.7 + 6.0	64747 + 819	1.334 + 0.149	138.4 + 59.8
11. W-20	0.1535 + 0.0215	247.3 + 6.2	71377 + 901	1.453 + 0.133	< 110.1[b]
12. W-23	0.2050 + 0.0232	269.9 + 6.5	58417 + 746	1.814 + 0.142	125.8 + 57.9
13. W-24	0.5314 + 0.0295	265.1 + 6.2	22300 + 382	1.284 + 0.137	< 112.6[b]
16. W-26	0.2612 + 0.0241	117.4 + 3.9	22015 + 361	1.398 + 0.109	< 115.7[b]
17. W-30	0.3521 + 0.0192	146.3 + 3.9	16753 + 281	1.371 + 0.096	< 79.29[b]
18. W-33	0.6396 + 0.0228	105.5 + 3.1	23231 + 343	4.261 + 0.146	89.68 + 40.34
19. W-39	2.120 + 0.061	283.7 + 8.5	55772 + 773	1.854 + 0.287	466.2 + 182.1
20. W-42	0.4794 + 0.0470	249.2 + 8.4	42700 + 669	1.453 + 0.262	307.7 + 132.4
21. W-45	17.36 + 0.29	110.7 + 3.3	30283 + 438	8.226 + 0.214	< 93.86[b]
22. W-48	0.1541 + 0.0143	214.9 + 4.7	24836 + 377	1.562 + 0.093	< 60.33[b]

[a]W: Unseparated fraction.

[b]< Peak not seen, .˙. a detection limit was calculated, so sample has less than this amount.

Table 5b. (Continued)

Sample I.D. Number	Zn-65 1115.5 KeV	Rb-86 1076.6 KeV	Sn-85 514.00 KeV	Sb-124 1691.00 KeV	Cs-134 604.7 KeV
3. W-1	3493 + 116	83.15 + 12.28	< 521.2	3.317 + 0.248	< 1.030
4. W-3	5936 ∓ 196	484.2 + 28.6	< 641.9	17.79 ∓ 0.73	36.98 + 0.88
5. W-9	5539 ∓ 183	288.8 + 19.3	< 593.4	2.403 + 0.218	1.820 ∓ 0.286
6. W-14	4370 ∓ 145	519.3 + 30.6	< 621.4	2.238 + 0.237	9.245 ∓ 0.436
7. W-16	651.6 ∓23.2	345.8 + 21.1	< 426.1	8.491 ∓ 0.414	2.157 ∓ 0.224
8. W-18	515.3 ∓18.6	378.5 + 23.0	< 393.8	7.093 ∓ 0.351	1.494 ∓ 0.214
11. W-20	982.0 ∓33.7	349.2 + 21.6	< 412.8	8.933 ∓ 0.436	1.630 ∓ 0.226
12. W-23	679.8 ∓23.9	213.2 + 15.4	< 431.0	10.09 ∓ 0.46	28.57 ∓ 0.70
13. W-24	312.1 ∓12.7	213.8 + 15.3	< 396.9	1.156 ∓ 0.148	< 0.7818
16. W-26	451.5 ∓16.5	250.5 + 16.6	< 342.6	< 0.4371	< 0.5260
17. W-30	148.1 ∓ 7.0	236.5 + 14.7	< 269.3	0.4941 + 0.1023	< 0.5418
18. W-33	244.1 ∓ 9.7	144.7 + 10.6	< 282.0	1.098 ∓ 0.122	0.6588 + 0.1474
19. W-39	2093 ∓ 71	414.7 + 29.8	< 819.5	1.103 ∓ 0.328	5.525 ∓ 0.449
20. W-42	908.4 ∓33.4	412.1 + 29.6	< 685.9	1.081 ∓ 0.261	3.315 ∓ 0.480
21. W-45	114.5 ∓ 5.7	75.77 + 7.62	< 291.7	0.6549 ∓ 0.0906	13.20 ∓ 0.38
22. W-48	637.1 ∓22.2	319.2 + 18.5	< 222.5	0.3804 ∓ 0.0812	1.705 ∓ 0.144

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Table 5b. (Continued)

Sample I.D. Number	Ba-131 216.00 KeV			Ce-141 145.40 KeV			Nd-147 91.90 KeV			Eu-152 1408.00 KeV			Tb-160 879.40 KeV		
3. W-1	7304	+	419	304.81	+	4.5	175.3	+	15.4	2.649	+	0.098	50.75	+	2.97
4. W-3	7172	+	441	98.91	+	4.91	< 58.42			0.7919	+	0.064	50.87	+	2.98
5. W-9	11131	+	614	103.1	+	4.2	< 54.20			1.015	+	0.059	78.14	+	4.55
6. W-14	12202	+	665	139.1	+	4.9	< 54.85			1.693	+	0.077	85.35	+	4.97
7. W-16	6329	+	351	3443	+	31	1520	+	82	28.77	+	0.51	41.95	+	2.45
8. W-18	5337	+	300	3236	+	30	1424	+	77	26.06	+	0.46	38.12	+	2.23
11. W-20	5729	+	320	2766	+	25	1206	+	65	24.93	+	0.45	38.47	+	2.25
12. W-23	7824	+	420	3998	+	36	1718	+	92	34.45	+	0.59	53.48	+	3.12
13. W-24	3399	+	219	1778	+	16	704.4	+	39.4	7.636	+	0.169	22.48	+	1.34
16. W-26	1875	+	142	811.9	+	78	324.5	+	19.8	4.956	+	0.124	13.35	+	0.81
17. W-30	3398	+	196	850.8	+	8.2	428.3	+	24.1	6.647	+	0.147	23.64	+	1.39
18. W-33	3776	+	214	1824	+	17	820.9	+	44.6	10.08	+	0.20	25.35	+	1.49
19. W-39	59112	+	3024	7861	+	71	5479	+	292	86.21	+	1.43	389.3	+	22.6
20. W-42	43592	+	2243	4137	+	38	2533	+	136	53.02	+	0.91	282.4	+	16.4
21. W-45	228.1	+	61.1	18.75	+	1.04	< 11.86			0.7511	+	0.0398	1.088	+	0.168
22. W-48	1857	+	123	405.5	+	4.2	251.9	+	15.0	3.082	+	0.094	11.55	+	0.69

Table 5b.   (Continued)

Sample I.D. Number	Yb-169 197.9 KeV		Hf-181 482.20 KeV		Ta-182 1221.40 KeV		Th(Pa-233) 312.00 KeV		Lu-177 208.40 KeV	
3. W-1	528.9	+ 16.5	971.3	+ 28.8	221.8	+ 9.1	685.6	+ 7.3	73.09	+ 4.4
4. W-3	912.8	+ 28.4	2298	+ 68	234.1	+ 9.6	1043	+ 11	133.2	+ 8.0
5. W-9	1168	+ 36	2282	+ 68	167.4	+ 6.9	901.7	+ 9.6	167.2	+ 10.1
6. W-14	1065	+ 33	1434	+ 43	193.0	+ 7.9	749.5	+ 8.0	142.9	+ 8.6
7. W-16	135.7	+ 4.3	332.3	+ 9.9	90.21	+ 3.72	264.8	+ 2.9	16.18	+ 0.98
8. W-18	125.2	+ 3.9	321.4	+ 9.5	85.04	+ 3.50	416.8	+ 4.5	14.99	+ 0.91
11. W-20	126.0	+ 4.0	325.7	+ 9.7	78.07	+ 3.22	130.6	+ 1.5	15.38	+ 0.94
12. W-23	165.3	+ 5.2	336.1	+ 10.0	112.9	+ 4.6	158.8	+ 1.7	18.48	+ 1.12
13. W-24	99.49	+ 3.15	398.0	+ 11.8	149.3	+ 6.1	152.8	+ 1.7	10.80	+ 0.62
16. W-26	48.44	+ 1.57	263.0	+ 7.8	108.2	+ 4.5	22.35	+ 0.36	4.684	+ 0.313
17. W-30	76.00	+ 2.40	123.9	+ 3.7	50.56	+ 2.0	140.2	+ 1.5	8.864	+ 0.544
18. W-33	76.77	+ 2.42	87.24	+ 2.61	67.18	+ 2.77	163.0	+ 1.8	6.900	+ 0.427
19. W-39	952.2	+ 29.7	124.8	+ 3.8	309.3	+ 12.7	743.2	+ 7.9	39.90	+ 2.42
20. W-42	786.3	+ 24.5	191.9	+ 5.7	319.9	+ 13.1	791.8	+ 8.5	65.38	+ 3.95
21. W-45	4.151	+ 0.260	2.880	+ 0.214	0.7930	+ 0.0722	4.752	+ 0.190	0.7404	+ 0.0824
22. W-48	37.12	+ 1.20	61.48	+ 1.86	13.11	+ 0.57	24.04	+ 0.33	3.892	+ 0.248

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Table 6. SQSA for Jabal Tawlah. 6-Step D.C. Arc (requested by C. T. Douch and done, at DGMR Lab., by K. J. Curry in Oct. 1979. These sample depths range from 25.30-71.40 m)

Element	LDL[a]	W-1	W-3	W-9	W-14
Fe[b]	500	10000	10000	10000	10000
Mg	200	300	500	700	7000
Ca	500	700	700	700	7000
Ti	20	1000	2000	1500	1500
Mn	10	1000	1500	1000	700
Ag	.5	N	N	N	N
As	200	N[c]	N	N	N
Au	10	N	N	N	N
B	10	N	10	15	L[d]
Ba	20	L	L	70	50
Be	1	7	10	10	15
Bi	10	N	N	N	N
Cd	20	N	N	N	N
Co	5	N	N	N	15
Cr	10	150	100	70	150
Cu	5	70	70	30	15
La	20	50	N	N	N
Mo	5	N	N	N	15
Nb	20	1500	1500	1000	700
Ni	5	7	N	L	50
Pb	10	300	700	300	150
Sb	100	N	N	N	N
Sc	5	20	30	50	30
Sn	10	150	300	150	150
Sr	100	L	100	L	L
V	10	L	L	L	20

[a]LDL: lower determination level.

[b]Fe, Mg, Ca, and Ti were reported in % but were converted to ppm.

[c]N: Not detected at limit of detection, or at value shown.

[d]L: Detected, but below limit of determination.

Table 6. continued

Element	LDL	W-1	W-3	W-9	W-14
W	50	N	N	N	N
Y	10	1500	G(2000)[e]	2000	2000
Zn	100	3000	5000	3000	2000
Zr	10	G1000	G1000	G1000	G1000
Th[f]	1	572	823	711	559
U[g]	–	55	.3		1.0 .8
Ta	3	328	312	204	148

[e]G: Greater than 10%, or greater than value shown.

[f]XRF experiment for Th and Ta.

[g]Fluorometric experiment for U.

Table 7. SQSA for Jabal Hamra. 6-Step D.C. Arc (requested by C. T.
Douch and done, at DGMR Lab., by Analyst Jambi, in Dec. 1980.
These samples were obtained at ground level)

Element	LDL[a]	W-16	W-18	W-20	W-23
Fe[b]	500	70000	70000	70000	70000
Mg	200	1000	300	300	500
Ca	500	700	700	700	5000
Ti	20	5000	5000	5000	5000
Mn	10	2000	1500	2000	3000
Ag	.5	N[c]	N	N	N
As	200	N	N	N	N
Au	10	N	N	N	N
B	10	L[d]	L	L	L
Ba	20	300	150	150	300
Be	1	70	30	70	70
Bi	10	N	N	N	N
Cd	20	N	N	N	N
Co	5	N	N	N	N
Cr	10	200	300	200	300
Cu	5	5	5	10	15
La	20	G(1000)[e]	G(1000)	G(1000)	G(1000)
Mo	5	N	N	N	N
Nb	20	G(2000)	G(2000)	G(2000)	G(2000)
Ni	5	N	N	N	N
Pb	10	300	100	100	70
Sb	100	N	N	N	N
Sc	5	20	15	15	20
Sn	10	150	100	100	150
Sr	100	N	N	N	N
V	10	N	N	N	10
W	50	70	100	70	70
Y	10	G(2000)	G(2000)	G(2000)	G(2000)
Zn	200	N	N	N	N
Zr	10	G(1000)	G(1000)	G(1000)	G(1000)

[a]LDL: lower determination level.

[b]Fe, Mg, Ca, and Ti were reported in % but were converted to ppm.

[c]N: Not detected at limit of determination, or at value shown.

[d]L: Detected, but below limit of determination, or below value shown.

[e]G: Greater than 10%, or greater than value shown.

Table 8. SQSA for Umm Al-Birak. 6-Step D.C. Arc (requested by N.
Jackson, done at DGMR Lab., by Analyst Ali Bone, on Jul. 1982.
These samples were obtained at ground level)

Element	LDL[a]	W-24	W-26	W-30	W-33
Fe[b]	500	20000	30000	20000	20000
Mg	200	700	300	200	1000
Ca	500	3000	7000	2000	3000
Ti	20	500	1000	500	1500
Mn	10	300	500	200	3000
Ag	.5	1.5	N[c]	3	1
As	200	N	N	N	N
Au	10	N	N	N	N
B	10	L[d]	10	L	L
Ba	20	N	30	N	N
Be	1	3	3	5	10
Bi	10	N	N	N	N
Cd	20	N	N	N	N
Co	5	N	N	N	L
Cr	10	200	100	100	100
Cu	5	15	20	15	15
La	20	1000	500	500	G(1000)[e]
Mo	5	5	100	L	50
Nb	20	1000	500	300	500
Ni	5	7	L	N	L
Pb	10	200	150	70	20
Sb	100	N	N	N	N
Sc	5	N	N	N	N
Sn	10	50	150	50	70
Sr	100	N	N	N	N
V	10	L	10	L	15
W	50	N	N	N	N
Y	10	1500	200	500	1500
Zn	100	200	500	L	L
Zr	10	G(1000)	G(1000)	G(1000)	G(1000)

[a]LDL: lower determination limits.

[b]Fe, Mg, Ca, and Ti were reported in % but were converted to ppm.

[c]N: Not detected at limit of detection, or at value shown.

[d]L: Detected, but below limit of determination, or below value.

[e]G: Greater than 10%, or greater than value shown.

Table 9. SQSA for Jabal Sayid. 6-Step D.C. Arc (Requested by D. Hackett, done at DGMR Lab., by Ali Bone, in July 1982. These sample depths range from 18.0-27.0 m)

Element	LDL[a]	W-39	W-42	W-45	W-48
Fe[b]	500	50000	30000	30000	30000
Mg	200	1000	700	15000	700
Ca	500	20000	50000	2000	1500
Ti	20	3000	300	1500	700
Mn	10	1000	500	700	300
Ag	.5	N[c]	N	N	N
As	200	N	N	N	N
Au	10	N	N	N	N
B	10	20	L[d]	15	10
Ba	20	70	200	300	20
Be	1	20	10	7	7
Bi	10	N	N	N	N
Cd	20	N	N	N	N
Co	5	N	N	7	N
Cr	10	200	300	150	300
Cu	5	100	100	15	7
La	20	G(1000)[e]	G(1000)	L	500
Mo	5	N	N	N	L
Nb	20	G(2000)	2000	L	200
Ni	5	N	N	7	7
Pb	10	3000	1500	70	150
Sb	100	N	N	N	N
Sc	5	N	N	20	N
Sn	10	150	200	L	30
Sr	100	N	N	200	N
V	10	L	L	100	10

[a]LDL: lower determination limits.

[b]Fe, Mg, Ca, and Ti were reported in % but were converted to ppm.

[c]N: Not detected at limit of detection, or at value shown.

[d]L: Detected, but below limit of determination, or below value.

[e]G: Greater than 10%, or greater than value shown.

Table 9. continued

Element	LDL	W-39	W-42	W-45	W-48
W	50	N	N	N	N
Y	10	G(2000)	G(2000)	50	700
Zn	200	1500	L	N	1000
Zr	10	G(1000)	G(1000)	70	G(1000)
Ce[f]	–	2891	2420	< 10	518
Th	–	533	842	< 1	22

[f] By atomic absorption experiment for Ce and Th.

Table 10. Concentrations (ppm) comparison of DGMR results with NAA
results

Jabal Tawlah

Elements	Samples Techniques	W-1	W-3	W-9	W-14
Th	XRF	572	823	711	559
	NAA	685± 7.3	1043±11.0	901±9.6	749±8.0
U	XRF	55	.3	.8	1.0
	NAA	112±8.0	< 5.8[a]	< 5.6	< 6.5

Jabal Sayid

Elements	Samples Techniques	W-39	W-42	W-45	W-48
Th	AA Spectrography	533	842	< 1.0	22
	NAA	743±8.0	791±8.5	4.7±2	24±0.3

[a] < Peak not seen, .˙. a detection limit was calculated, so samples
has less than this amount.

Table 11. Concentrations (ppm) comparison of NAA results with SQSA results for Jabal Tawlah (samples depth ranges from 25.3 - 71.4 m)

Samples Elements	W-1	W-3	W-9	W-14
As	< 3.998[a] N[b]	14.52 ± 1.25 N	7.058 ± 1.076 N	30.78 ± 1.78 N
Mo	61.24 ± 24.9 N	< 36.909 N	< 36.296 N	< 41.187 15
Sb	2.401 ± 2.58 N	15.47 ± .57 N	2.520 ± .237 N	1.955 ± .284 N
La	71.89 ± .90 50	1.877 ± .423 N	3.214 ± .409 N	10.80 ± .60 N
W	< 4.832 N	< 5.281 N	< 5.087 N	< 5.839 N
Sc	1.001 ± .039 20	.5845 ± .0362 30	1.238 ± .038 50	7.472 ± .135 30
Cr	218.8 ± 6.8 150	111.7 ± 6.8 100	163.9 ± 7.1 70	271.8 ± 8.3 150
Fe	15905 ± 323 10000	14659 ± 325 10000	16506 ± 312 10000	21955 ± 395 10000
Co	1.857 ± .158 N	1.159 ± .179 N	1.901 ± .179 N	10.06 ± .29 15
Ni	< 166.1 7	< 149.8 N	< 114.1 L[c]	< 184.6 50

[a] NAA results. <: peak not seen ∴. a dection limit was calculated, so sample has less than this amount.

[b] SQSA results. N: not detected at limit of detection, or at value shown.

[c] L: Detected but below limit of determination.

Table 11. (Continued)

Samples Elements	W-1	W-3	W-9	W-14
Zn	3493 $\pm$ 116 3000	5936 $\pm$ 196 5000	5539 $\pm$ 183 3000	4370. $\pm$ 145 2000
Sn	< 521.2 150	< 641.9 300	< 593.4 150	< 621.4 150
Ba	7304 $\pm$ 419 L	7172 $\pm$ 441 L	11131 $\pm$ 614 70	12202 $\pm$ 665 50

Table 12. Concentrations (ppm) comparison of NAA results with SQSA results for Jabal Hamra (samples were obtained at ground level)

Samples Elements	W-16	W-18	W-20	W-23
As	743.5 ± 42.0[a] N[b]	779.6 ± 51.7 N	1311 ± 51 N	1212 ± 54 N
Mo	40.85 ± 19.24 N	32.39 ± 16.99 N	22.96 ± 15.11 N	55.19 ± 20.52 N
Sb	8.155 ± .507 N	6.992 ± .439 N	8.024 ± .451 N	9.612 ± .533 N
La	1643 ± 12 G(1000)[e]	1473 ± 10 G(1000)	1090 ± 8 G(1000)	1762 ± 12 G(1000)
W	< 7.289[d] 70	< 6.850 100	< 5.474 70	< 7.975 70
Sc	.1601 ± .0246 20	.1665 ± .0215 15	.1535 ± .0215 15	.2050 ± .0232 20
Cr	203.8 ± 5.5 200	242.7 ± 6.0 300	247.3 ± 6.2 200	269.9 ± 6.5 300

[a]NAA results.

[b]SQSA results, N: Not detected at limit of detection, or at value shown.

[c]G: Greater than 10% or greater than value shown.

Table 12. continued

Fe	67096 70000	$\pm$ 855	64747 70000	$\pm$ 819	71377 70000	$\pm$ 901	58417 70000 $\pm$ 746
Co	1.548 N	$\pm$ .141	1.334 N	$\pm$ .149	1.453 N	$\pm$ .133	1.814 N $\pm$ .142
Ni	< 107.7 N		138.4 N	$\pm$ 59.8	< 110.1 N		125.8 N $\pm$ 57.9
Zn	651.6 N	$\pm$ 23.2	515.3 N	$\pm$ 18.6	982.0 N	$\pm$ 33.7	679.8 N $\pm$ 23.9
Sn	< 426.1 150		< 393.8 100		< 412.8 100		< 431.0 150
Ba	6329 300	$\pm$ 351	5337 150	$\pm$ 300	5729 150	$\pm$ 320	7824 300 $\pm$ 420

Table 13. Concentrations (ppm) comparison of NAA results with SQSA results for Umm Al-Birak (samples were obtained at ground level)

Samples Elements	W-24	W-26	W-30	W-33
As	< 7.506[a] N[b]	< 5.891 N	44.01 $\pm$ 2.11 N	44.21 $\pm$ 2.3 N
Mo	51.37 $\pm$ 23.1 5	152.92 $\pm$ 28.4 100	22.33 $\pm$ 11.08 L[c]	73.37 $\pm$ 18.2 50
Sb	1.027 $\pm$ .309 N	< .7698 N	< .7504 N	1.033 $\pm$ .269 N
La	796.0 $\pm$ 5.8 1000	316.1 $\pm$ 2.5 500	451.9 $\pm$ 3.4 5000	859.1 $\pm$ 6.2 G(10000)[d]
W	11.95 $\pm$ 3.49 N	< 5.898 N	< 5.821 N	11.13 $\pm$ 3.25 N
Sc	.5314$\pm$ .0295 N	.2612 $\pm$ .0241 N	.3521$\pm$ .0192 N	.6396$\pm$ .0228 N
Cr	265.1 $\pm$ 6.2 200	117.4 $\pm$ 3.9 100	146.3 $\pm$ 3.9 100	105.5 $\pm$ 3.1 100

[a] NAA results. <: peak not seen, ∴ a detection limit was calculated, so sample has less than this amount.

[b] SQSA results, N: Not detected at limit of detection, or at value shown.

[c] L: Detected, but bellow limit of determination.

[d] G Greater than 10%, or greater than values shown.

Table 13. (continued)

Fe	22300	+ 382	22015	+ 361	16753	+ 281	23231	+ 343
	20000		30000		20000		20000	
Co	1.284 +	.137	1.398 +	.109	1.371 +	.096	4.261 +	.146
	N		N		N		N	
Ni	< 112.6		< 115.7		< 79.9		89.68 +	40.34
	7		L		N		L	
Zn	312.1 +	12.7	451.5 +	16.5	148.1 +	7.0	244.1 +	9.7
	200		500		L		L	
Sn	< 396.9		< 342.6		< 269.3		< 282.0	
	50		150		50		70	
Ba	3399	+ 219	1875	+ 142	3398	+ 196	3776	+ 214
	L		30		N		N	

Table 14. Concentrations (ppm) comparison of NAA results with SQSA results for Jabal Sayid (samples depth ranges form 18 - 27 m)

Samples Elements	W-39	W-42	W-45	W-48
As	< 13.26 [a] N [b]	< 12.30 N	< 4.545 N	< 5.406 N
Mo	156.23 + 78.5 N	232.47 + 104.5 N	< 19.55 N	16.68 + 8.66 L [c]
Sb	2.146 + .508 N	< 1.489 N	< .6111 N	< .6677 N
La	2357 + 17 G(1000) [d]	1518 + 11 G(1000)	5.989 + .439 L	143.0 + 1.4 500
W	< 12.64 N	< 11.94 N	< 4.555 N	< 5.153 N
Sc	2.120 + .061 N	.4794 + .047 N	17.36 + .29 20	.1541+ .0143 N
Cr	283.7 + 8.5 200	249.2 + 8.4 300	110.7 + 3.3 150	214.9 + 4.7 300

[a]NAA results, <: peak not seem, .˙. a detection limit was calculated, so sample has less than this amount.

[b]SQSA results, N: Not detected at limit of detection, or at value shown.

[c]L: Detected, but below limit of determination.

[d]G: Greater than 10%, or greater than value shown.

Table 14. (continued)

Fe	55772 ± 773 50000	42700 ± 669 30000	30283 ± 438 30000	24836 ± 377 30000
Co	1.854 ± .287 N	1.453 ± .262 N	8.226 ± .2164 7	1.562 ± .043 15
Ni	466.2 ± 182.1 N	307.7 ± 132.4 N	< 93.89 7	< 60.33 7
Zn	2093 ± 71 1500	908.4 ± 33.4 L	114.5 ± 5.7 N	637.1 ± 22.2 1000
Sn	< 819.5 150	< 685.9 200	< 291.7 L	< 222.5 30
Ba	59112 ± 3024 70	43592 ± 2243 200	228.1 ± 61.1 300	1857 ± 123 20

42 the SQSA results were higher than the NAA results by about 50 ppm, for sample W-45 the SQSA result was less than 1 ppm, while the NAA result was about 4.5 ppm, and for sample W-48 the results were in close agreement.

For Jabal Tawlah samples the U concentration provided by DGMR were about half of the NAA result for sample W-1 and were below the NAA detection limit for the other three samples. The SQSA results were also below the NAA for the three samples. Apparently the presence of U and Th at the other two locations, Jabal Hamra and Umm Al-Birak was not investigated at this time by DGMR. The U presence at Jabal Sayid was not investigated.

The NAA method sought the presence of the following elements: Na, As, Br, Mo, Sb, La, Sm, Yb, W, U, Sc, Cr, Fe, Co, Ni, Zn, Rb, Sn, Cs, Ba, Ce, Nd, Eu, Tb, Hf, Ta, Th, and Lu. (Arranged in order of increasing Z, for 6 and 25.6 days delay time).

The SQSA method sought the presence of the following elements: Fe, Mg, Ca, Ti, Mn, Ag, As, Au, B, Ba, Be, Bi, Cd, Co, Cr, Cu, La, Mo, Nb, Ni, Pb, Sb, Sc, Sn, Sr, V, W, Y, Zn, and Zr.

It will be noted that there are a number of elements common to both lists and the concentrations of only these can be compared.

From Table 10, it will be observed that the results obtained by different techniques are consistent for Jabal Tawlah and Jabal Sayid samples.

From Tables 11-14, it can be seen that:

- There is a consistency (considerable similarity) in the results of NAA and SQSA for five elements: La, Cr, Fe, Co, and Zn coming from all four areas. For example, the Fe

concentrations determined by the two techniques have the same order of magnitude.

- The SQSA did not reveal the elements As and Sb in any samples.
- There is inconsistency (considerable variation) with six elements: W, Mo, Sc, Ni, Sn, and Ba.

Although the NAA results are higher than SQSA results in most cases, the SQSA results are sometimes higher (e.g., Cr in samples W-42 and W-48 at Jabal Sayid and in samples W-18 and W-23 at Jabal Hamra).

The typical gamma-ray spectrum for samples H-1 and L-1 are shown in Figs. 19-22 for the two activity measurements. One can notice from the figures that even after taking the sample mass differences into account the H-1 count level is higher than that for L-1 indicating that there is a difference in the concentration of elements in them.
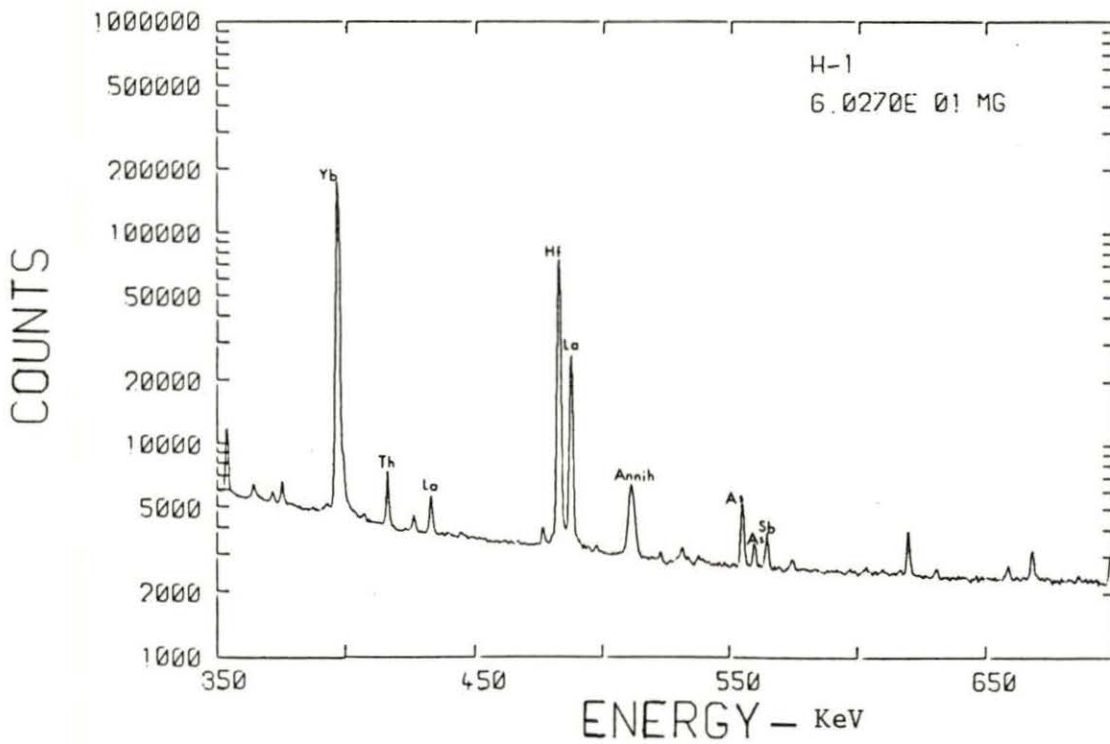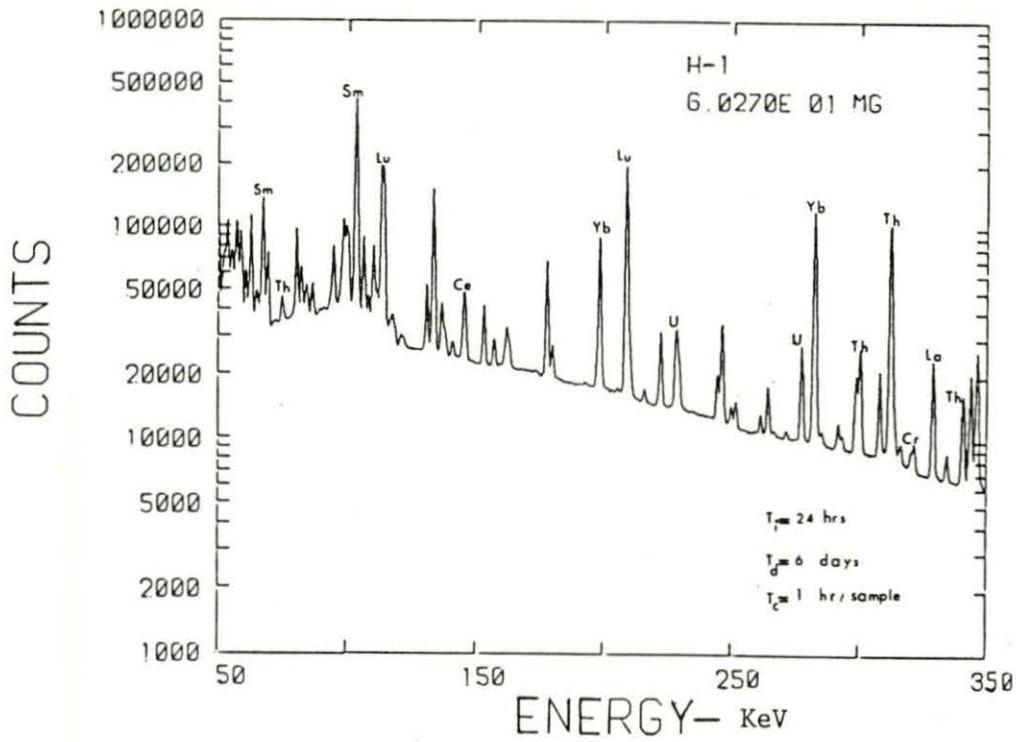
Fig. 19. Typical γ-ray spectrum for sample H-1
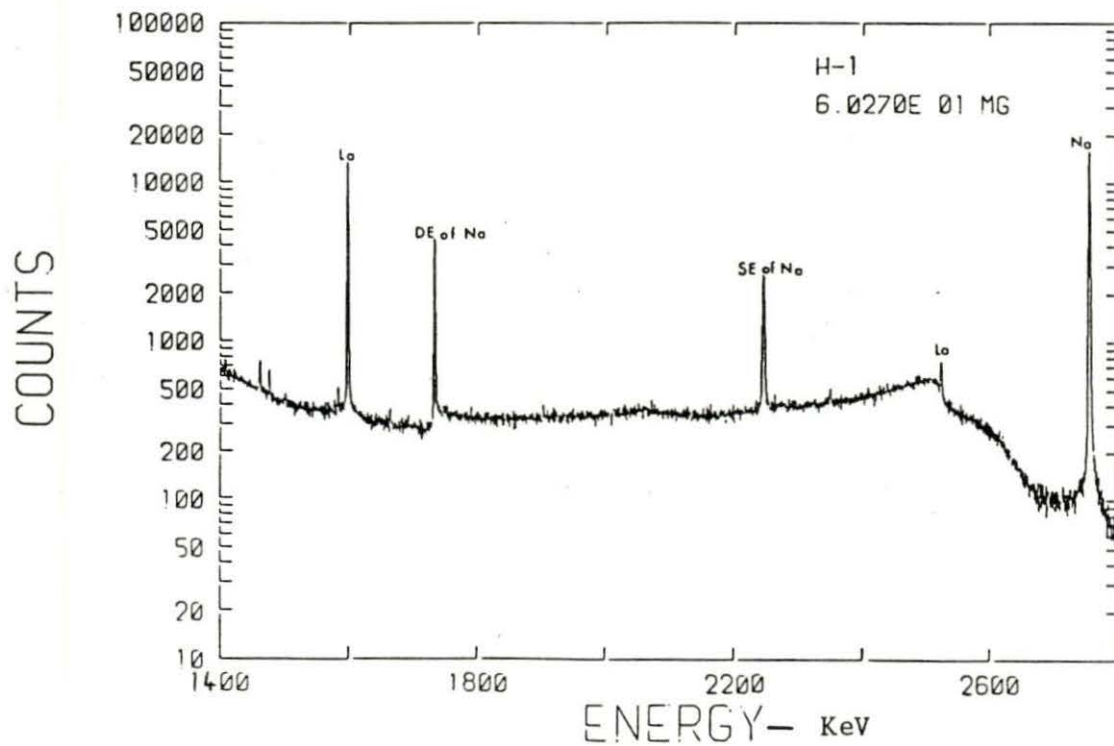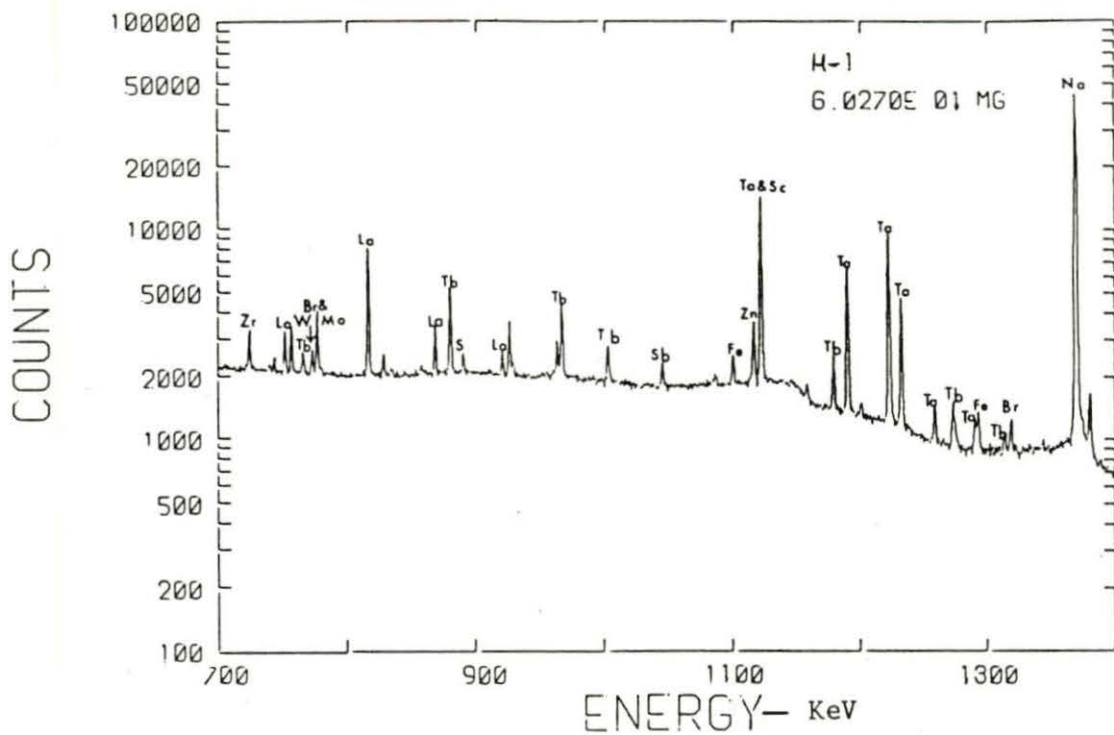
Fig. 19. continued
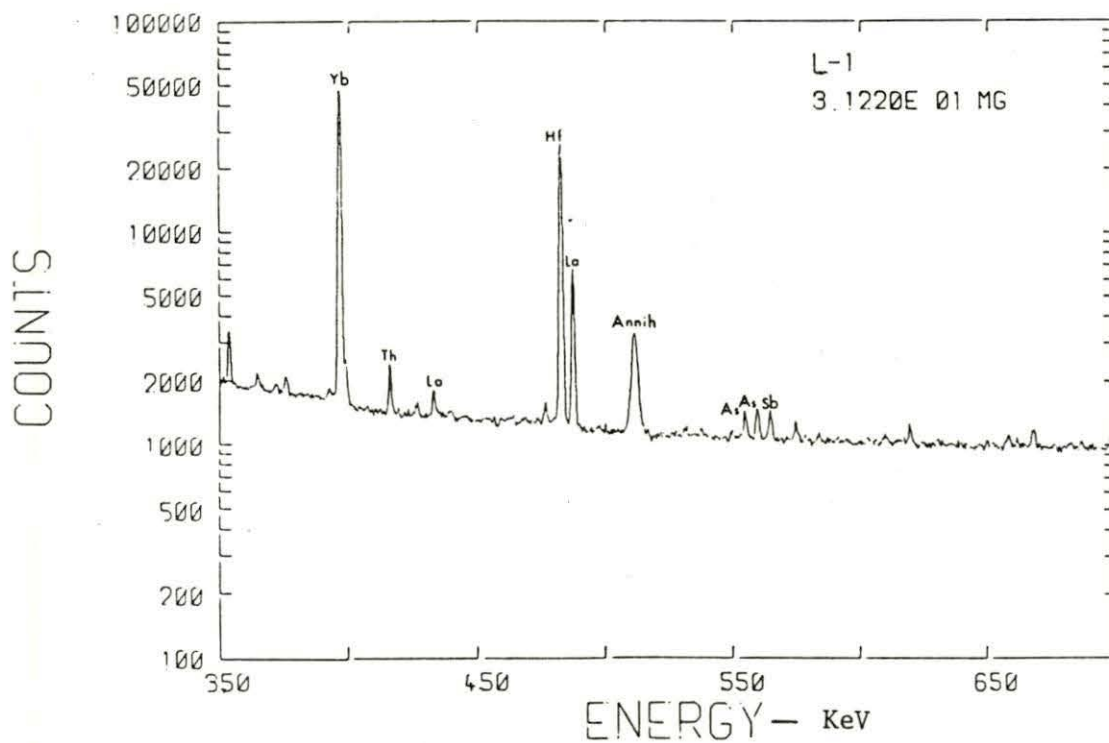
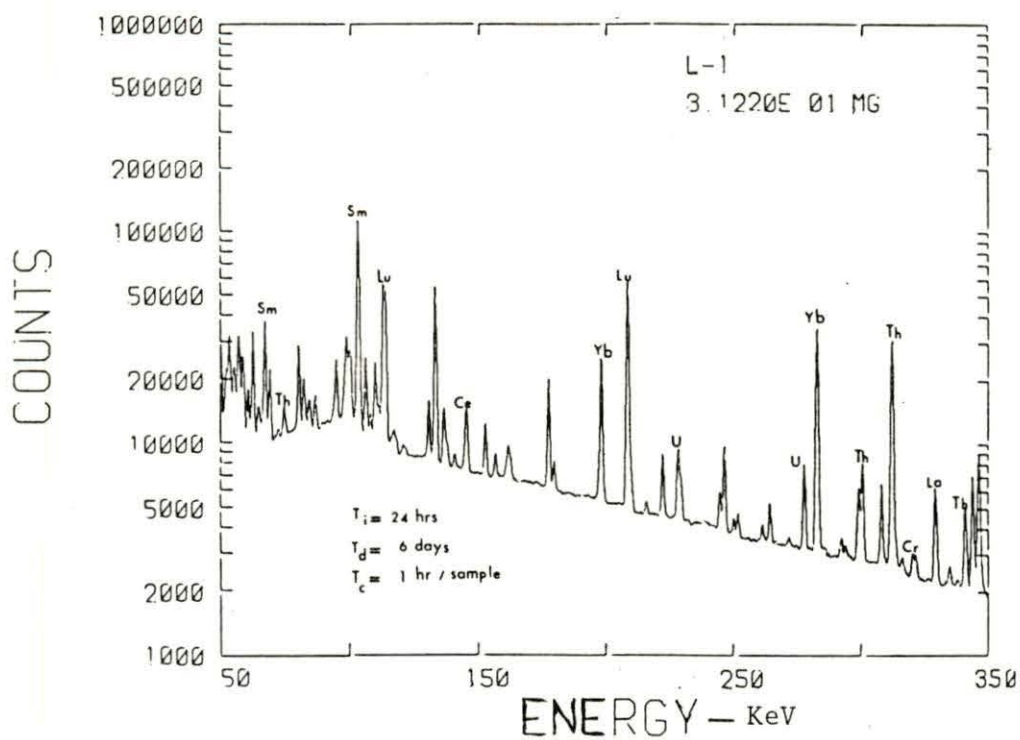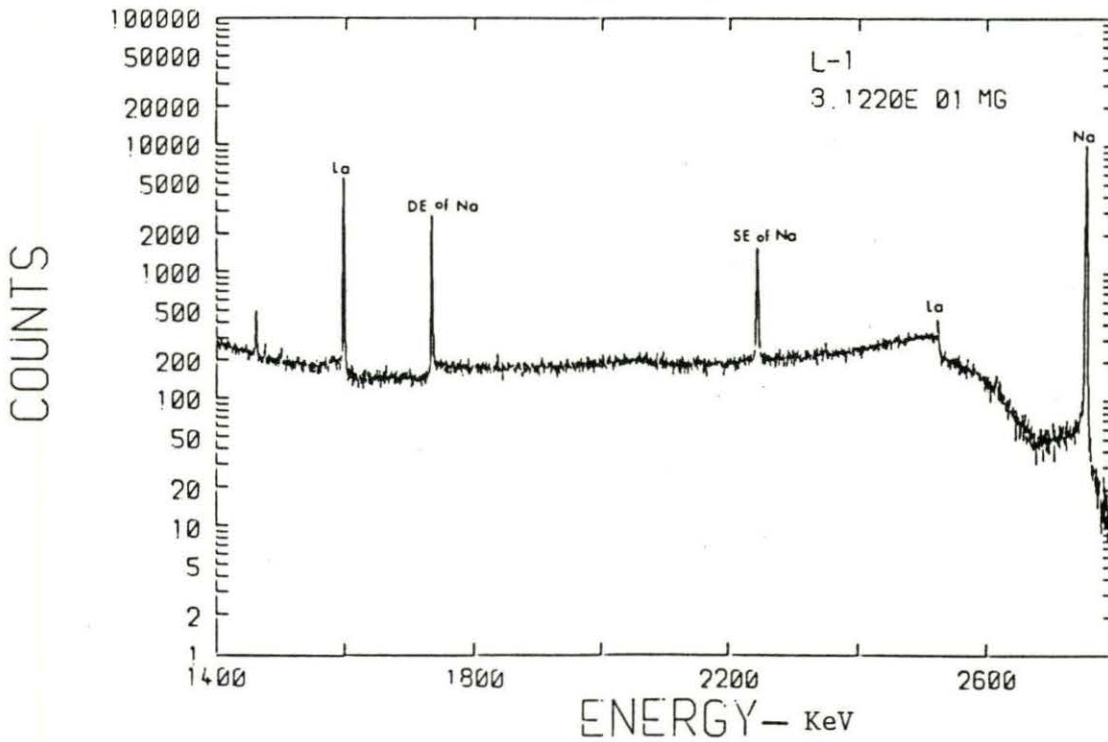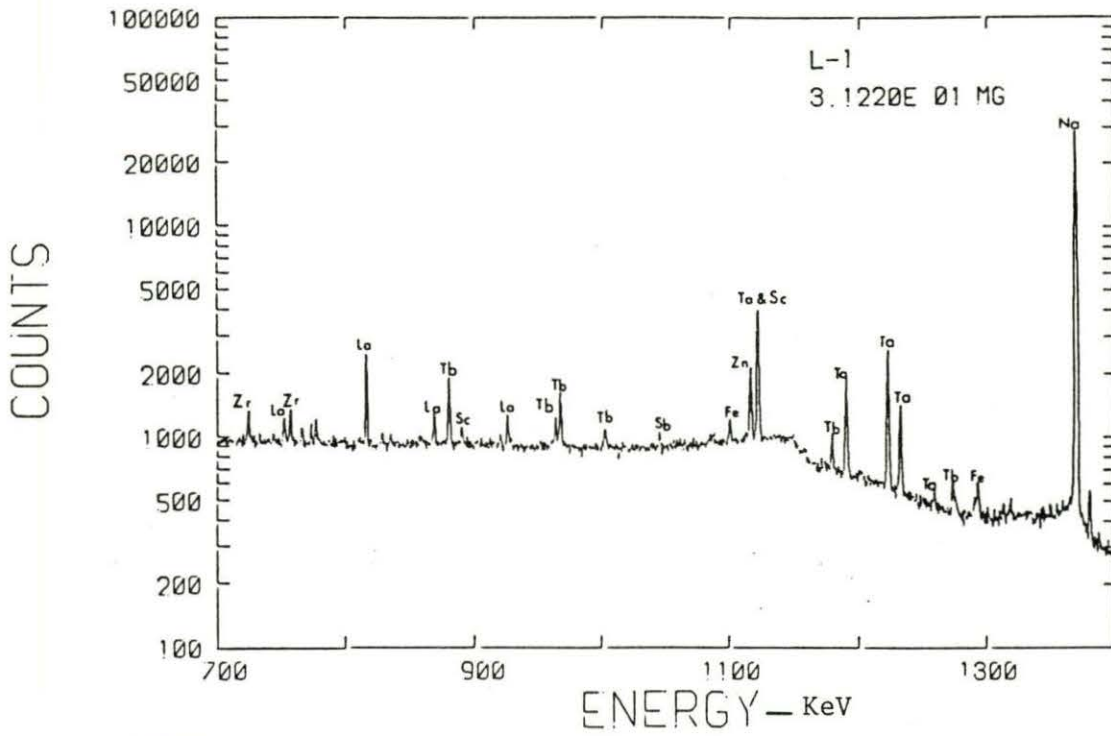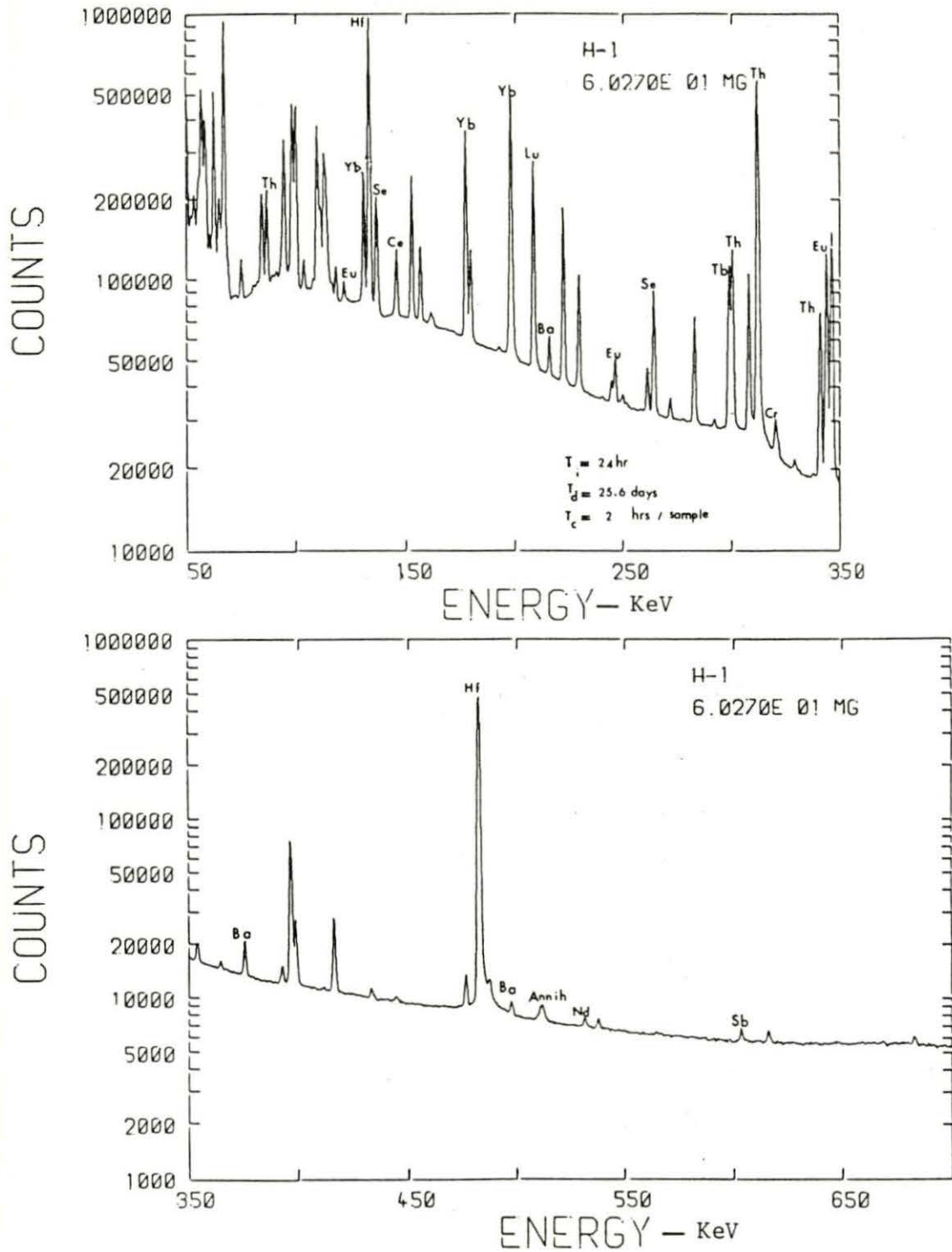Fig. 20. Typical γ-ray spectrum for sample L-1

Fig. 20. continued

Fig. 21. Typical γ-ray spectrum for sample H-1

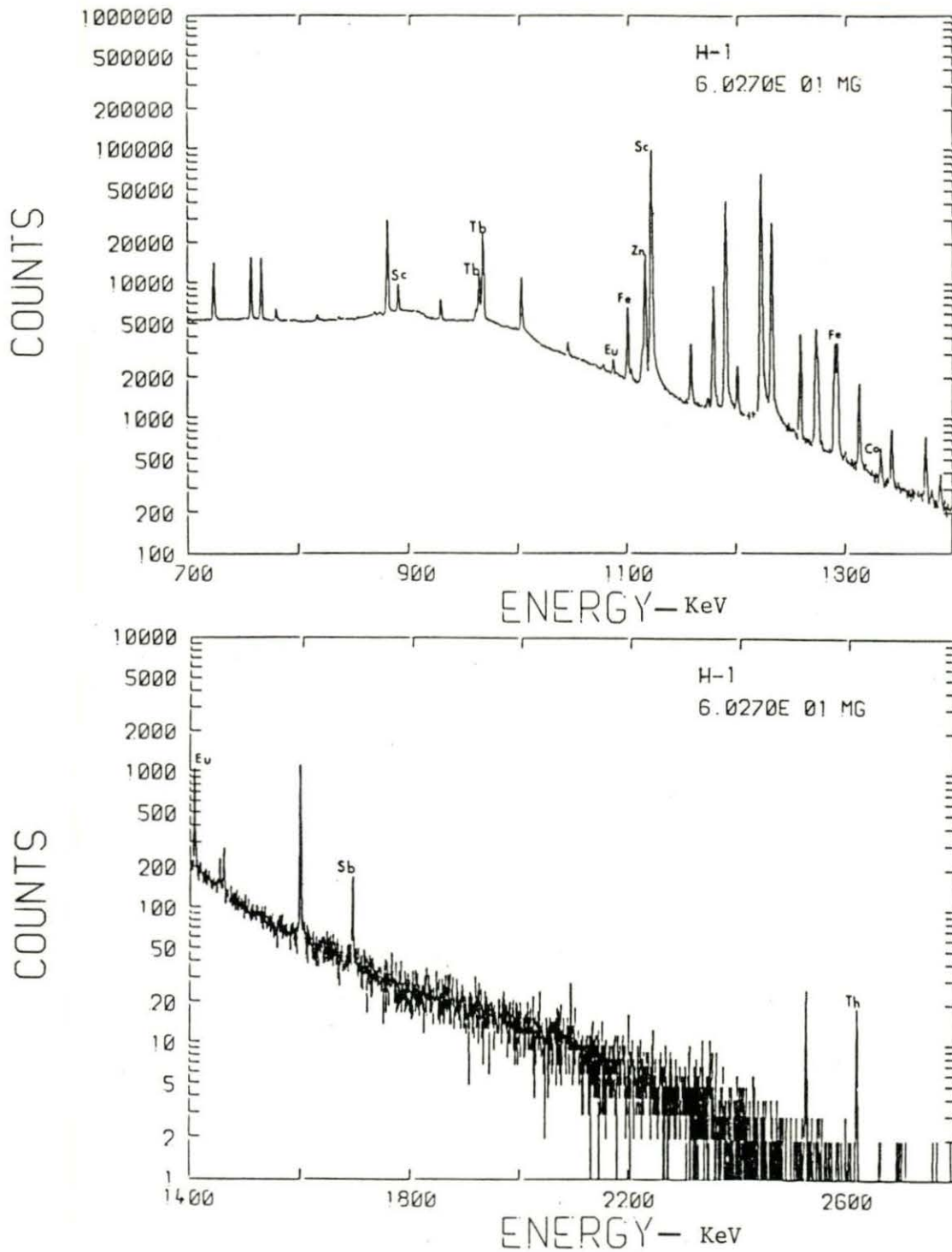Fig. 21. continued
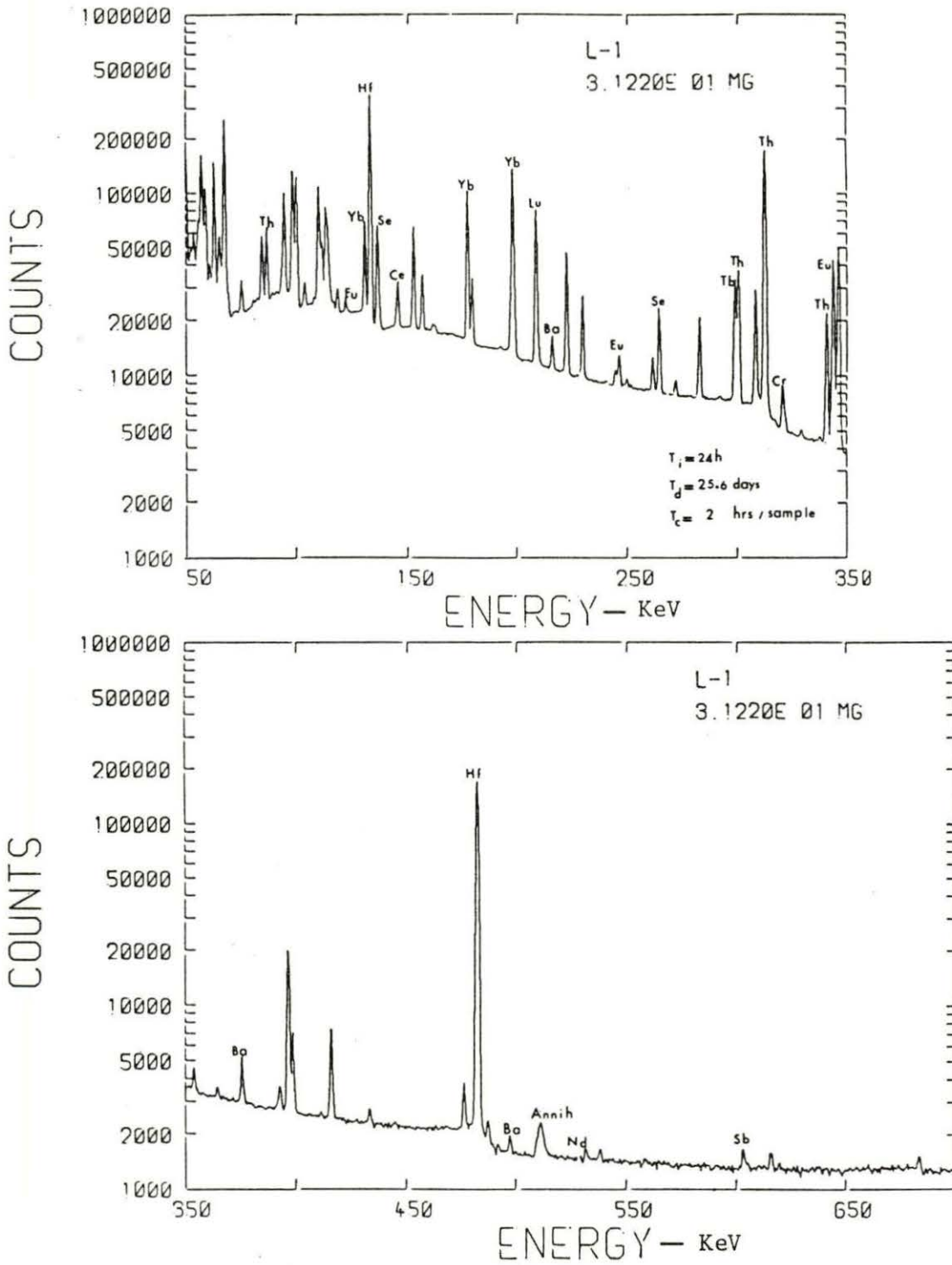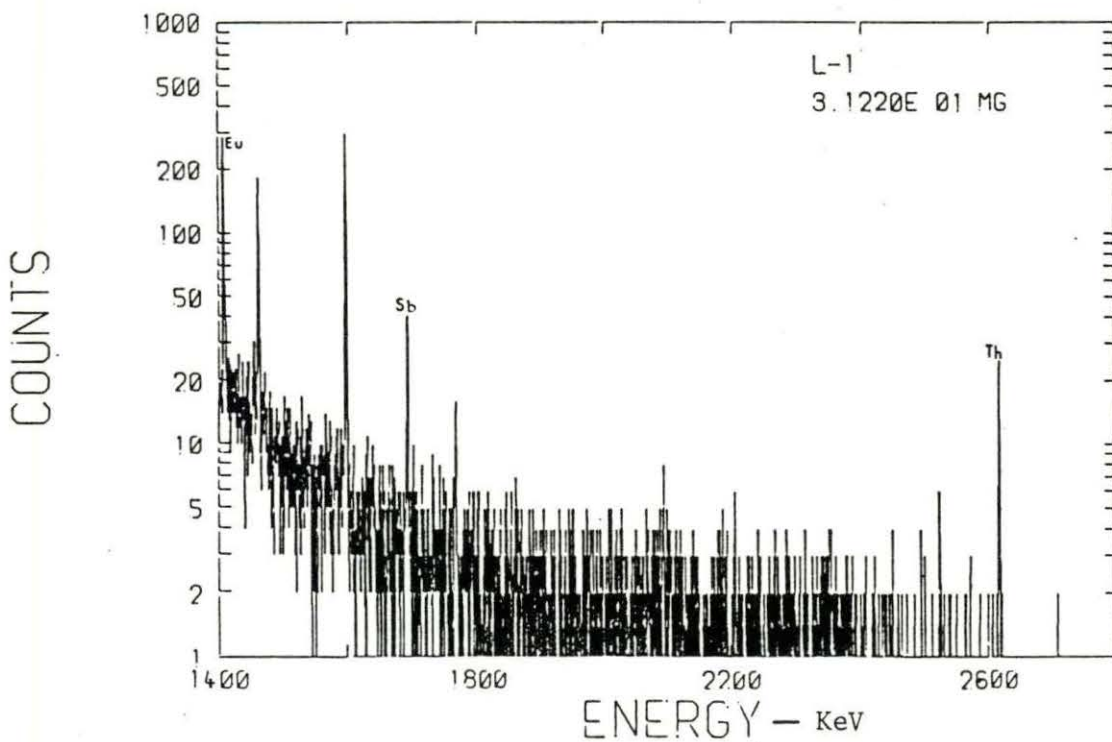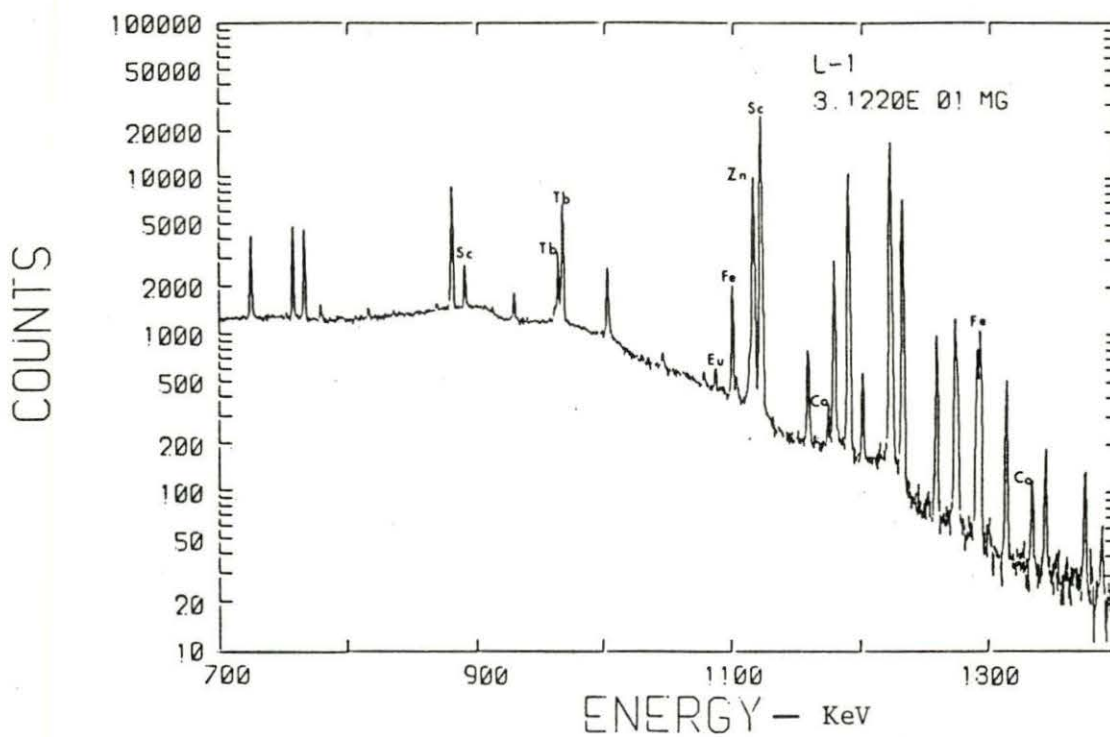
Fig. 22. Typical γ-ray spectrum for sample L-1

Fig. 22. continued

CONCLUSIONS

- GS, as applied to the separation of heavy elements from the lighter ones, was found to produce an enhancement factor of two or more in heavy elements (e.g., Yb-175, sample H-26 and L-26; Table 3a, Fe-59 and Zn-65 sample H-26 and L-26, Table 3b).

- U and Th were found in all samples. U concentrations of up to 556 ppm were found at the Jabal Sayid location (2000 ppm is considered a good mining venture, whereas a concentration of 1000 ppm should be explored with care, and a concentration of 700 ppm is considered not to be economical to mine) [84].

- Th concentrations of up to 1043 ppm were found at Jabal Tawlah area (60,000 ppm is considered necessary for a good mining venture [84].

- Many rare earth elements were found including, La, Ce, Nd, Sm, Eu, Tb, Yb, and Lu, which are generally fouond in association with Th [84].

SUGGESTIONS FOR FURTHER WORK

- Belatedly, it was discovered that the samples from each of the four regions were obtained at different depths and thus, were not replicates. Continuing work should include a study of replicates at various depths.

- The analytical technique used in this work emphasized long half lives and thus excluded from considerations those elements with half lives less than about 15 hours. Further work would be directed toward shorter half lives.

- Additional analyses should be performed on more samples from all four areas but, particularly for the Jabal Tawlah and Jabal Sayid areas (i.e., Th and U "rich" areas).

- Inductively Coupled Plasma (ICP) technique should be investigated as a potential alternative analytical method.

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