CHAPTER III

EXPERIMENT

3.1 Equipments

3.1.1 Neutron Source: Thai Research Reactor-1/Modification-1 (TRR-1/M-1) which is a swimming pool type designed to operate at a maximum power of 2 MW but routinely operate at 1 MW and has been operated for 5-6 hours a day, five days a week.

3.1.2 Counting equipments, composed of:

3.1.2.1 A Ge (Li) co-axial detector (ORTEC, Model 8001-2021 ORTEC) has a nominal active volume of 26 cm^3 and a resolution (FWHM) of 2.1 KeV at 1332 KeV of Co-60.

3.1.2.2 A ORTEC 4096 channel pulse height analyzer (Model 7030) with mini computer.

3.1.3 Mechanical shaker: Shaker with shaking heads, for four separatory funnels from Arthur H.Thomas Company, Model 8292-B10.

3.1.4 Laminar flow ultra clean work station (Dexon, Model HT 53E-830)

3.2 Sample Collection and Sampling

3.2.1 Sample Collection

About 180 head hair samples were collected from men, who entered into the priesthood on April, 1980. Each whole head hair was shaved off by a clean razor (very sharp) and stored in a separate clean plastic bag with identification, and sealed. Informations of the individual donor i.e age, field of study or occupation, residence, hair treatment and date of collection, were recorded.

3.2.2 Sampling

Owing to the unforseen circumstances e.g. shut down of the research reactor, the delay of a newly designed irradiating facility, a malfunction of detector etc , only 50 samples could be analyzed. Consequently, samples were selected in such a way that they would represent all occupations or subjects of study, age and residence. The selected samples for study were divided into five groups, according to occupation or field of study, that is:

a) Private businessman.

b) Farmer and officer working in Maize Research Center.

c) College student in the field of engineer.

d) College student in the field of Science and applied science, including chemistry, pharmacy, zoology, fishery, forestry and agricultural science.

e) College student in the field of Social Science, including political sciance and forensic science.

A list of selected samples together with their occupation/subject of study, residence , age and character of hair

Table 3.1 A list of selected samples together with their occupation/subject of study, residence, age and character of hair.

Occupation/Subject of study	Age (Years)	Residence	Character of hair
Private businessmen			
F-21	27	BK.13	S
F-25	25	BK.12	M
F-26	19	BK.12	L
F-27	22	BK.12	M, P
F-28	18	BK.12	M
F-29	18	BK.12	M Soft
Farmer and officer working in Maize Research Center			
C-25	21	Ayutthaya	M
D-10	20	Pathum Thani	L
B-11	32	Nakhon Ratchasima	S
C-28	23	Nakhon Ratchasima	S
College student in the field of Engineering			
A-4	22	BK.11	S, VP
B-5	21	BK.26	L
B-22	24	BK.6	S
B-24	21	BK.11	L

Table 3.1 (contd.)

Occupation/Subject of study	Age (Years)	Residence	Character of hair
College student in the field of Engineering			
E-8	20	BK.6	S
E-25	21	BK.10	M
College student in the field of Science & Applied Science			
a) Chemistry			
D-22	21	BK.9	M
F-5	21	BK.19	S
b) Pharmacy			
A-28	21	BK.3	L
C-1	20	BK.5	L
C-2	22	BK.5	L
C-16	22	BK.5	M
D-2	22	BK.4	S
E-1	20	BK.6	L
E-13	20	BK.4	L
E-26	20	BK.5	L
c) Zoology			
A-22	24	BK.5	Very L
A-26	20	BK.21	M, Thick
E-20	23	BK.9	M, Hair-oil

Table 3.1 (contd.)

Occupation/Subject of study	Age (Years)	Residence	Character of hair
d) Fishery			
D-7	21	BK.27	L
B-10	21	BK.7	M
F-2	21	BK.6	M
F-6	20	BK.14	L
e) Forestry			
B-15	20	BK.9	L
f) Agricultrual - science			
D-17	21	BK.9	L
College student in the field of Social science			
A-3	23	BK.23	M
A-23	20	BK.22	M
D-6	21	BK.24	M
B-13	21	BK.24	S
B-18	24	BK.24	L
B-23	21	BK.21	S
C-15	21	BK.24	L
C-19	22	BK.11	L
C-21	22	BK.11	L
D-23	24	BK.11	M
E-6	20	BK.24	M
E-7	22	BK.21	M
E-29	21	BK.21	L
F-11	22	BK.24	S
F-13	22	BK.10	L

S-Short hair (length < 1")
M-Medium hair (3"< length > 1")
L-Long hair (length > 3")
P-Permanent wavy hair
VP-Very permanent wavy hair

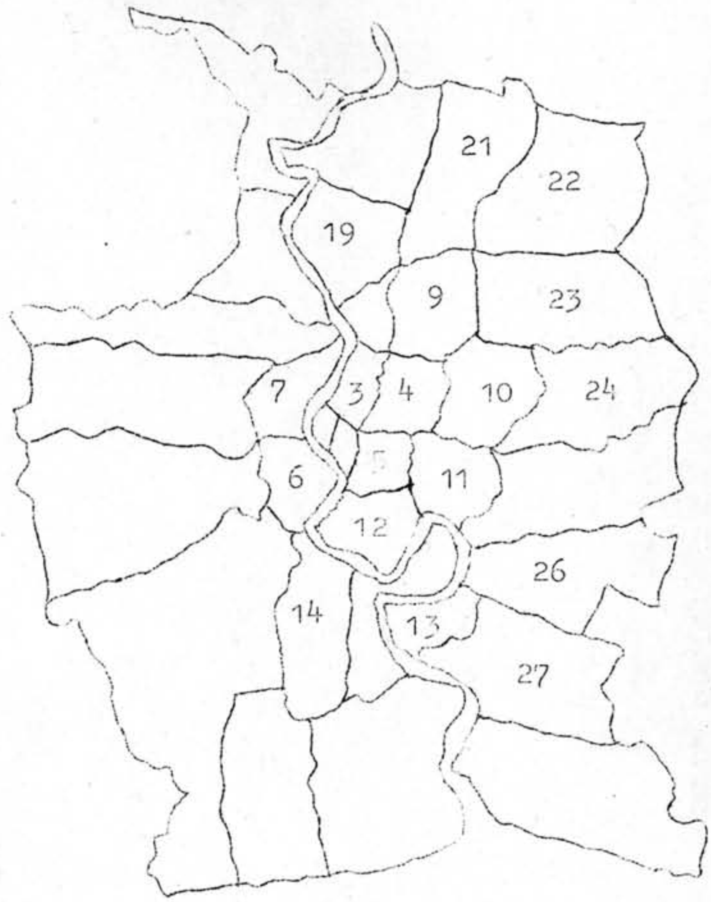


Fig.3.1 The distribution of residence of selected hair samples

3 - BK.3 Dusit
4 - BK.4 Phaya-Thai
5 - BK.5 Pathum Wan & Bangrak
6 - BK.6 Thonburi, Bangkok Yai, Khlong-San
7 - BK.7 Bangkok Noi
9 - BK.9 Bang Khen
10 - BK.10 Bang Kapi & Huay Khwang
11 - BK.11 Phra Khanong
12 - BK.12 Yan Nawa
13 - BK.13 Phra Pradaeng
14 - BK.14 Rat-Burana
19 - BK.19 Nonthaburi
21 - BK.21 Min Buri
22 - BK.22 Ram Inthra
23 - BK.23 Bang Kapi
24 - BK.24 Khlong Chan
26 - BK.26 Bang Na
27 - BK.27 Samut Prakan

Table 3.2 Distribution of residence of the samples

Residence	No.of sample	Identification No.
BK.3	1	A-28
BK.4	2	D-2,E-13
BK.5	5	C-1,C-2,C-16,E-26,A-22
BK.6	4	B-22,E-1,E-8,F-2
BK.7	1	B-10
BK.9	4	B-15,D-17,D-22,E-20
BK.10	2	E-25,F-13
BK.11	5	A-4,B-24,C-19,C-21,D-23
BK.12	5	F-25,F-26,F-27,F-28,F-29
BK.13	1	F-21
BK.14	1	F-6
BK.19	1	F-5
BK.21	4	A-26,B-23,E-7,E-29
BK.22	1	A-23
BK.23	1	A-3
BK.24	6	B-13,B-18,C-15,D-6,E-6,F-11
BK.26	1	B-5
BK.27	1	D-7
Ayutthaya	1	C-25
Pathumthani	1	D-10
Nakhonratsima	2	B-11,C-28

Table 3.3 Distribution of age of samples

Age (Years)	No.of sample	Identification No.
18	2	F-28,F-29
19	1	F-26
20	11	A-23,A-26,B-15,C-1,D-10,E-1,E-6,E-8,E-13, E-26,F-6
21	16	A-28,B-5,B-10,B-13,B-23,B-24,C-15,C-25, D-6,D-7,D-17,D-22,E-25,E-29,F-2,F-5
22	10	A-4,C-2,C-16,C-19,C-21,D-2,E-7,F-11,F-13, F-27
23	3	A-3,C-28,E-20
24	4	A-22,B-18,B-22,D-23
25	1	F-25
27	1	F-21
32	1	B-11

was shown in table 3.1. Figure 3.1 illustrated the distribution of residence of selected hair samples. In addition, grouping of sample according to residence and age was shown in table 3.2 and 3.3 respectively.

3.3 Sample Preparation

3.3.1 Washing Procedure

Each sample was first washed three times with 200-500 cm^3 of tap water to remove soap on hair which was used on the shaving step and washed again three times with 200-500 cm^3 of demineralize water. In each washing, hair sample was swilled and transfered from one washing to another little by little by using a clean aluminium tong (12"length). After washing, individual hair sample was air-dried on a clean aluminium tray (8"x12") at room temperature in a laminar air flow fumehood. (Fig.3.2)

Each dried hair sample was divided into two portions. The first portion, about 1-3 g , was kept in a clean plastic bag and named unwashed sample. The latter, about the same weight, was washed by using the IAEA's recommended procedure (3). The detail can be briefly described as follows:

Hair sample was put into a clean pyrex separatory funnel (250 cm^3) and washed with 200 cm^3 portions of reagent grade acetone and successively, distilled-demineralized water, distilled-demineralized water, distilled-demineralized water and reagent grade acetone, decanting off the washing liquid after each 10-minute washing. The mechanical shaker (Fig.3.3) was used throughout

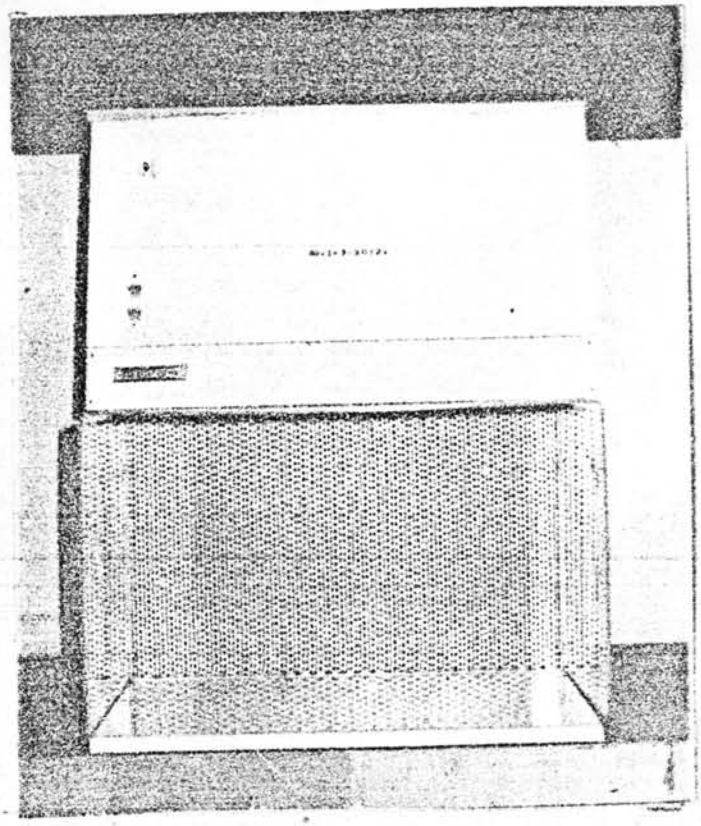


Fig. 3.2
Laminar flow ultra clean work station

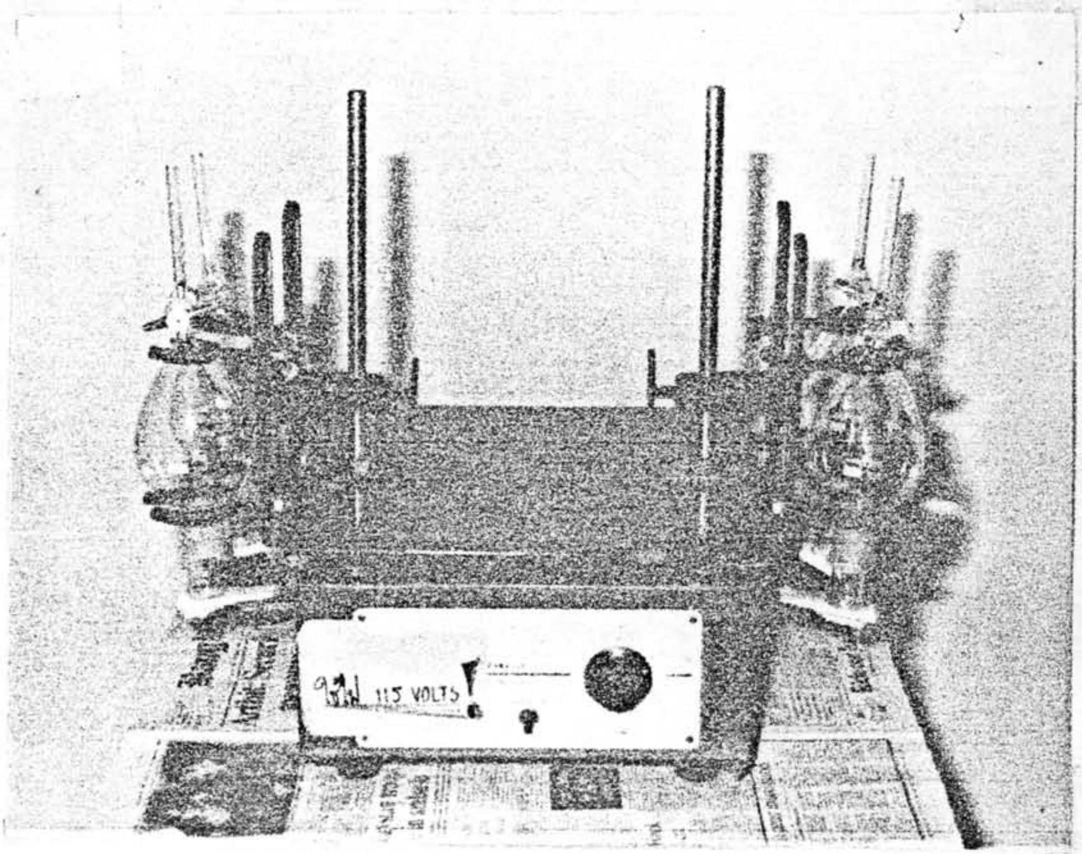


Fig. 3.3 Mechanical Shaker

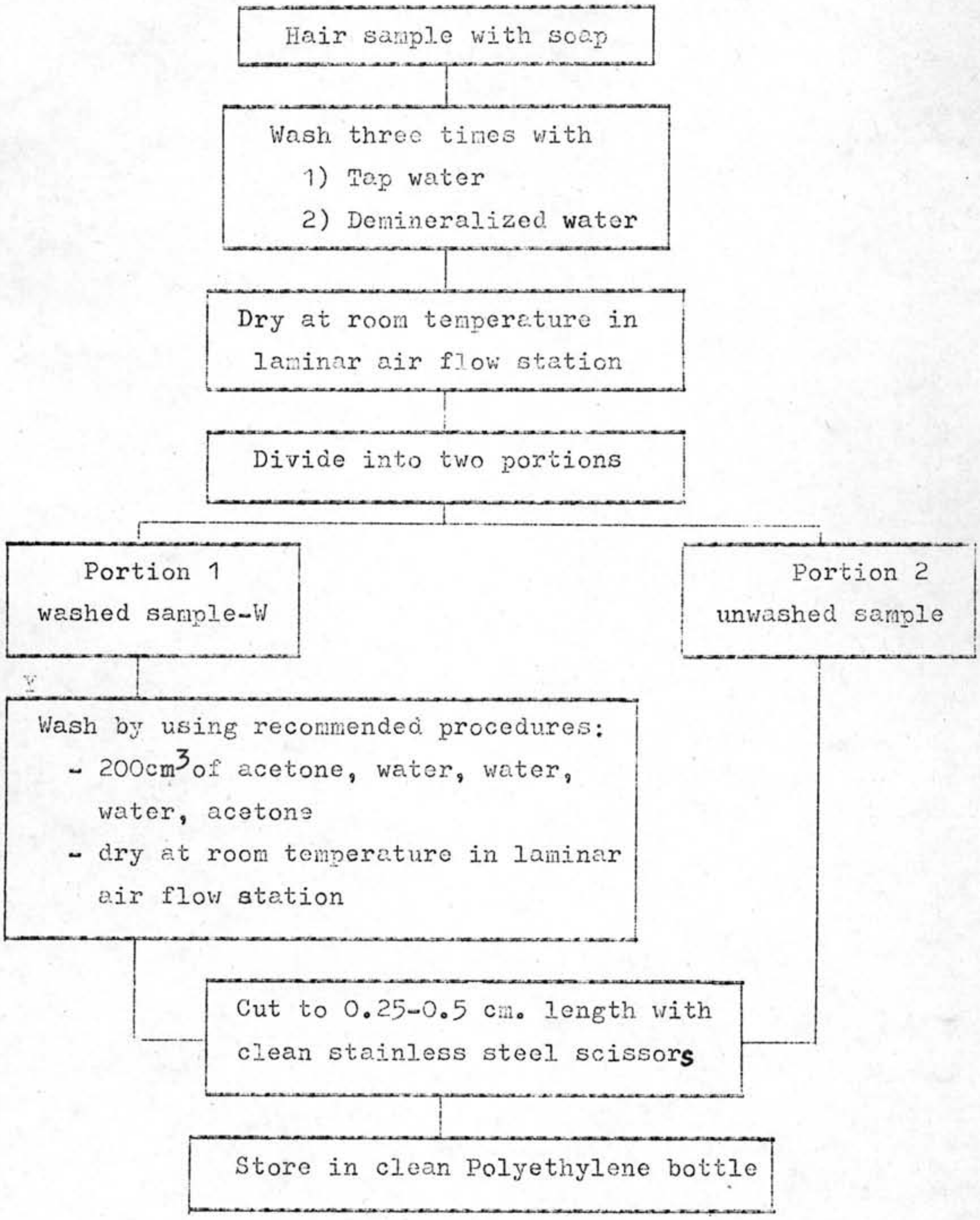


Fig.3.4 Flow chart of sample preparation

this washing step. After washing, hair sample was air-dried on the clean-aluminium tray at room temperature in the laminar air flow station. The sample was stored in a clean plastic bag and named washed sample.

3.3.2 Cutting

Both types of sample were cut separately to a 0.25-0.5 cm length with clean stainless steel scissors in a laminar air flow station. The hair was stored in a clean dry polyethylene bottle with identification. The sample preparation scheme was shown in the form of flow chart in Fig 3.4.

3.4 Qualitative Analysis

The determination was divided into three groups according to the half life of activated radioisotopes i.e short, medium and long-lived radioisotopes respectively.

3.4.1 For short-lived nuclides.

About 400 mg of hair sample was put into a clean dry polyethylene vial of ca. 1 ml capacity with a snap cap, heat-sealed, and placed in a Rabbit container. Sample was irradiated in the pneumatic transferring tube system of TRR-1/M-1 for 2 minutes at a flux of 1.8×10^{12} n $cm^{-2} sec^{-1}$. After cooling for some time, the irradiated sample was counted by ORTEC Ge(Li) co-axial detector coupled with a 4096 channel pulse-height analyzer (ORTEC) for 2 minutes. The spectrum was identified and recorded. An example of spectrum was shown in Fig.3.7.

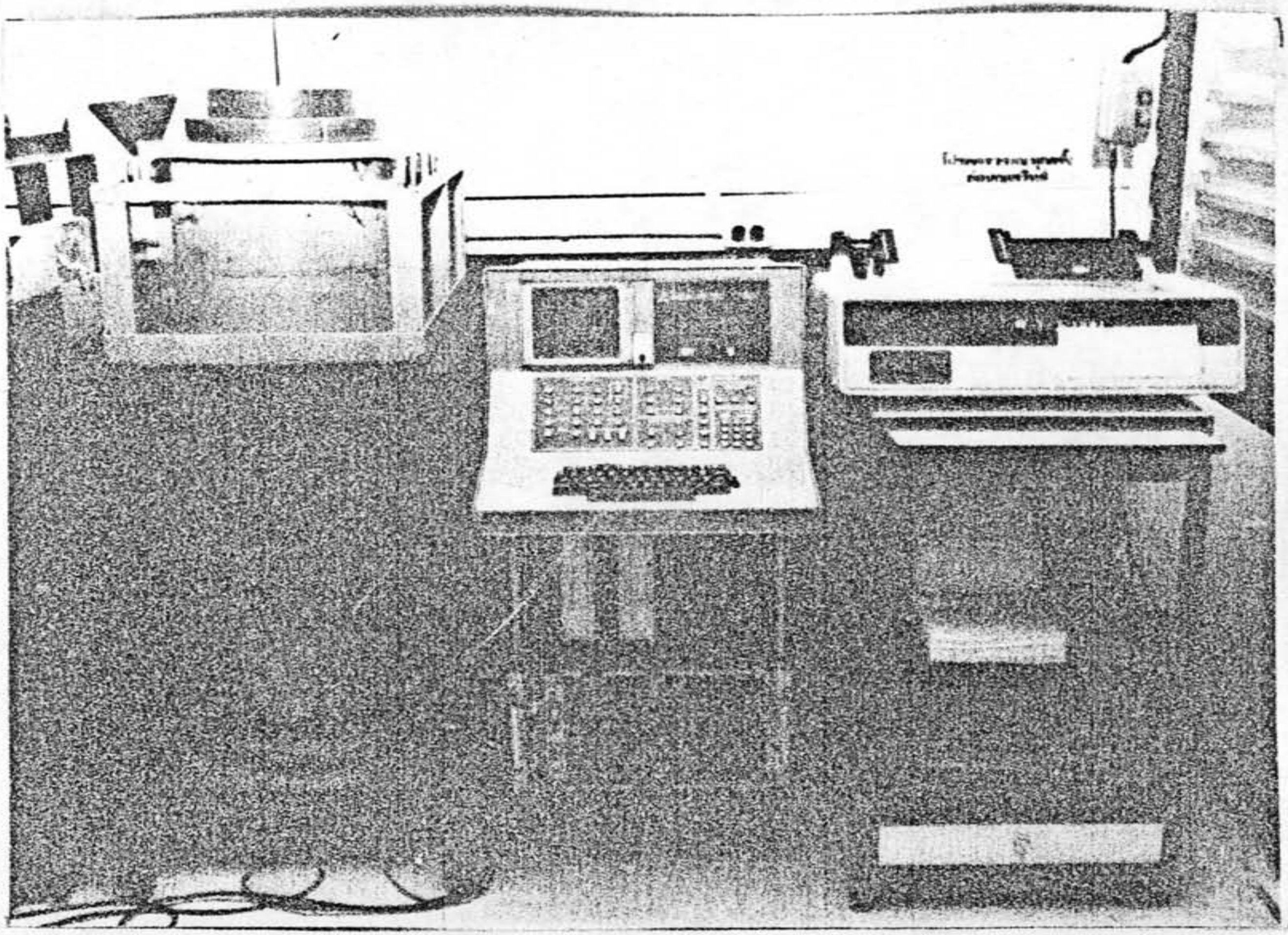
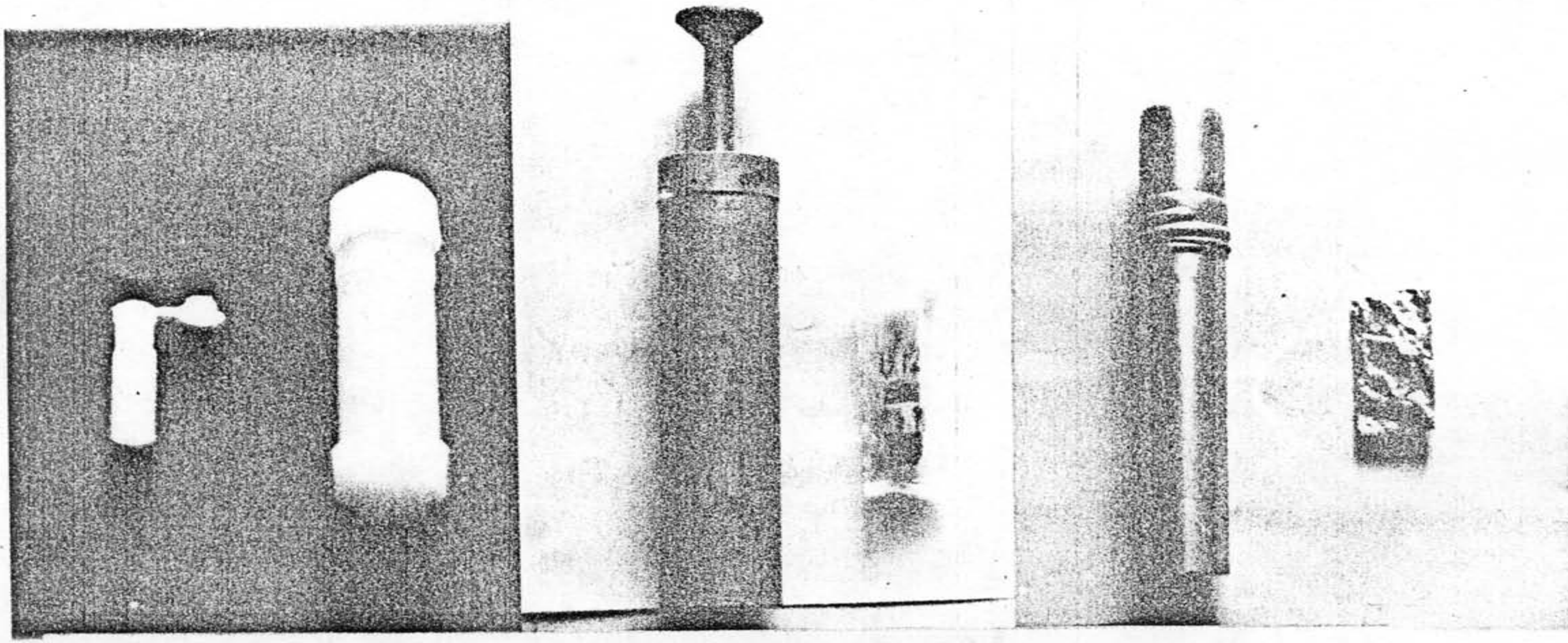


Fig.3.5 A ORTEC Ge(Li)co-axial detector and a ORTEC 4096 channel pulse height analyzer with mini computer



(a) (b) (c)

Fig.3.6 Container for irradiation :

a) polyethylene vial and Rabbit container

b) quartz ampoule and aluminium container

c) aluminium-foil sheet and aluminium container

3.4.2 For medium and long-lived nuclide

About 400 mg of hair sample was encapsulated in a clean dry quartz ampoule and placed in a aluminium container. Sample was irradiated in a wet facility of TRR-1/M-1 for 15 days (about 90 hrs.) at a flux of $2x10^{12}$ n $cm^{-2}sec^{-1}$. After an appropriate time of cooling, the irradiated sample was transferred into a clean dry polyethylene bottle and counted on the Ge(Li) detector coupled with a 4096 channel pulse height analyzer for 1000 and 3000 seconds for the investigation of the medium-lived and long-lived nuclides, respectively. These spectrums were identified and recorded in which the individual example was shown in Fig.3.8 and 3.9 respectively.

In case of the determination of Potassium, since the half life of K-42 is not too long (about 12 hours) and could not be determined by the method described above, the technique must be modified as follows:

About 400 mg of hair sample was wrapped with aluminium and placed in a aluminium can. Sample was irradiated in Rotating Specimen (Lazy Susan) facility of TRR-1/M-1 reactor for 2 days (10-12 hours) at a flux of $4x10^{11}$n $cm^{-2}sec^{-1}$. After cooling for about 18 hours, the irradiated sample was unwrapped and transferred into a clean dry polyethylene bottle and counted on Ge(li) detector for 1000 seconds.

3.4.3 The Interpretation of Gamma-Ray Spectrum

Since a radionuclide is characterized by its half-

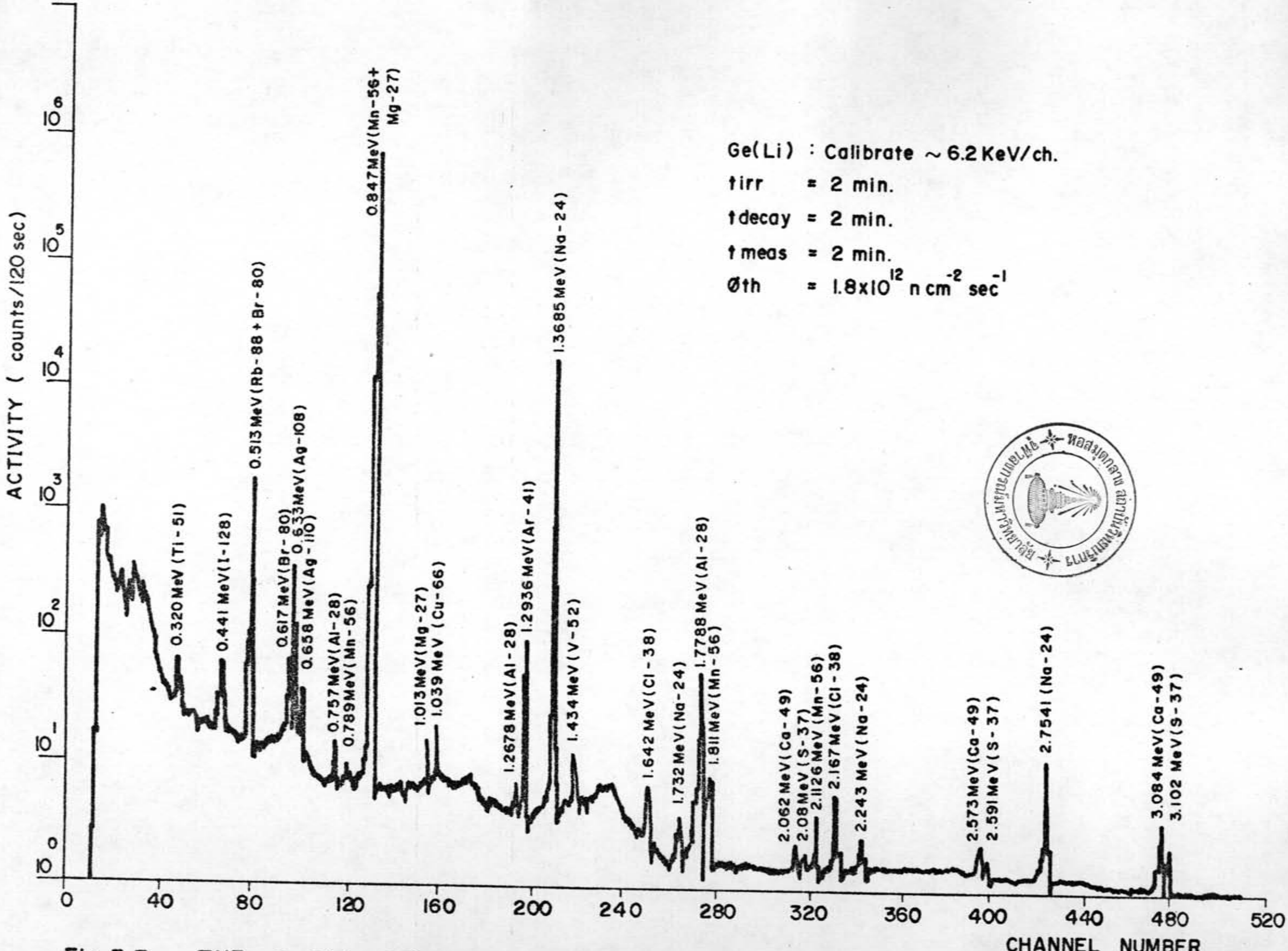


Fig. 3.7 THE γ-RAY SPECTRUM AFTER 2 MIN. (Short-lived isotopes)

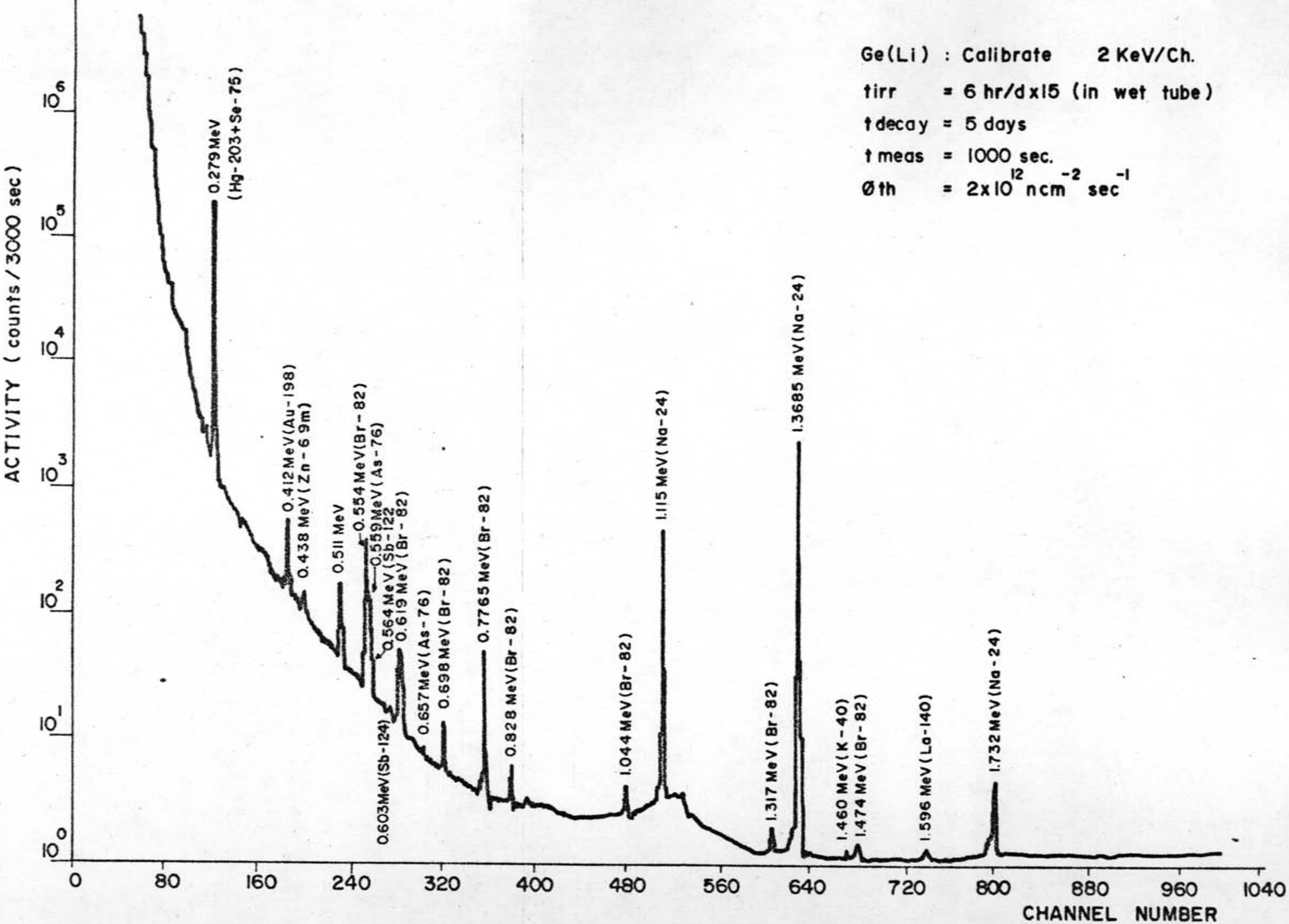


Fig. 3.8 THE γ-RAY SPECTRUM AFTER 5 DAYS (Medium-lived isotopes)

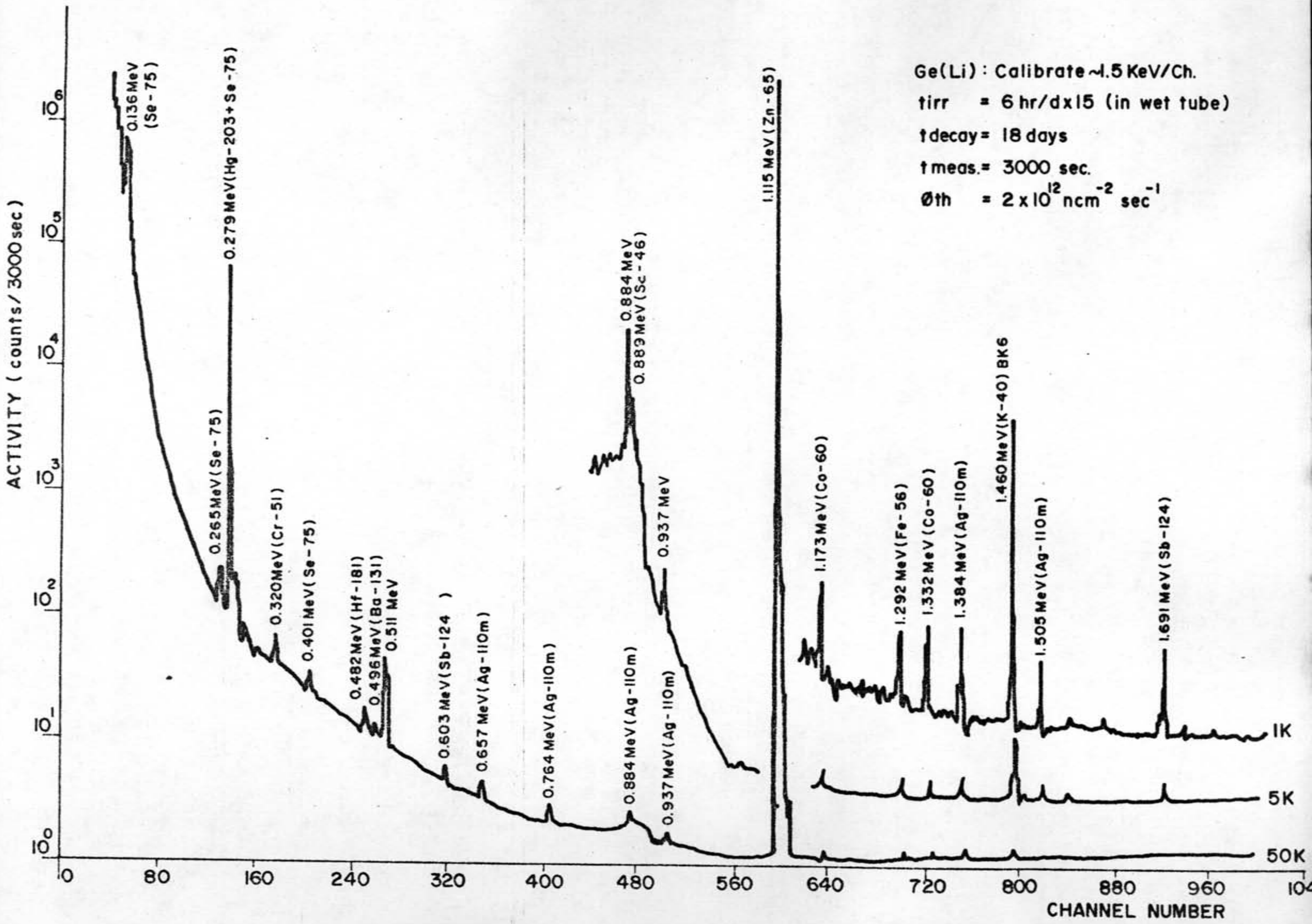


Fig. 3.9 THE γ - RAY SPECTRUM AFTER 20 DAYS (Long - lived isotopes)

life ($t_{1/2}$) and the emitted gamma radiation (s), the interpretation of gamma-ray spectrum involves:

1) locating photopeaks in the spectra,

2) determining photopeak energies from calibration curve which is done by plotting between the energies of measured gamma rays (vertical line) and numbers of channel in which γ-rays occurred (horizontal line),

3) calculating the half-life of each radionuclide by following the decay of radionuclide as a function of time.

If the radionuclide emits more than one γ-ray per disintegration, both the γ-ray energies and their relative intensities, characteristic of radionuclide, are also calculated. If two photopeaks are overlapped, the activities of the interested photopeak can be determined by using spectrum stripping method. This method is performed by successive substraction of the various pure components until the photopeak of interest is free from interferring activities.

4) Comparing the energies of γ-radiation (s), half-life, relative intensities of each radionuclide to the value(s) which was reported in the literature.

3.5 Quantitative Analysis

The concentration of an element in a sample can be calculated by using a comparative method. In the comparative method an element X in sample and a known amount of the same element X as a standard are irradiated together and counted under exactly the same conditions by the same radiation detector. The equation by the comparative method is

$$\frac{\text{weight of element X in sample}}{\text{weight of element X in standard}} = \frac{\text{Ax* in sample } e^{\lambda t\text{sample}}}{\text{Ax* in standard } e^{\lambda t\text{standard}}} \quad (3.1)$$

Knowing the activities of x*, Ax*, in the sample and standard, the sample and standard decay time (t) and weight of X in the standard, the weight of element X in the sample is then calculated. The activities of x* is the net energy peak area which can be calculated by using a method described by Das and co-worker (65).

3.5.1 Standard Reference Material (SRM)

In comparative method as mentioned above, SRM was used instead of standard solution. In this study the IAEA Soil-5 and Lake Sediment (SL-1) and the National Bureau of Standards (NBS) Orchard Leaves (SRM1571), and Bovine Liver (SRM1577) were used in which their details were shown in table 3.4.

3.5.2 Irradiation, Counting and Calculation

Each weighted hair sample and all the Standard reference materials were individually enclosed in polyethylene vial or quartz capsule and the whole arrangement was put in the polyethylene container or in aluminium can. Sample and SRM were irradiated together and counting under the same configurations as shown in table 3.5.

Only the case of medium and long irradiation, a weighted pure copper sheet was attached to outside of the quartz capsule as a neutron flux monitor. After the irradiation was finished, both samples and SRM were separately transferred into the new clean dry polyethylene bottles and reweighed.

The concentration of each element in each hair sample was calculated by using the comparison method as in equation 3.1. In this method, the net peak area of a chosen gamma-ray energy of each chosen nuclide in sample and SRM were compared. The chosen gamma-rays were marked with an asterisk on the corresponding nuclides as in table 3.6.

3.6 The Reliability Test for Analysis Method.

The reliability in quantitative analysis is based on both precision and accuracy. Accuracy of a procedure can be evaluated by analysing elements in a well-homogenized geological standard. While the precision or the reproducibility of analysis is characterized by the standard deviation which can be evaluated by the replicate analysis of a homogeneous sample.

The reliability of the analytical methods used in this study was previously tested by analysing IAEA Powered Human Head Hair, HH-1. The result was shown in table 5.4.

Table 3.4 Standard Reference Materials (SRM)

Element	Concentration of element (ppm)			
	Soil-5	Lake Sediment	Orchard leaves	Bovine liver
Ag	(1.9±0.11)	—	—	—
Al	81900±2800	—	—	—
As	—	—	14±2	—
Au	—	—	0.004*±0.002	—
Ba	562±53	—	—	—
Br	—	—	(10)	—
Ca	—	—	20900±300	—
Cl	—	—	(700)	—
Co	14.8±0.76	—	—	—
Cr	28.9±2.8	—	—	—
Cu	—	—	12±1	—
Fe	—	—	300±20	—
Hg	—	—	0.155±0.015	—
Hf	6.30±0.38	—	—	—
I	(35)	—	—	—
K	18600±1500	—	—	—
La	28.1±1.5	—	—	—
Mg	—	—	6200±200	—
Mn	—	—	91±4	—
Na	—	—	82±6	—
Rb	—	—	12±1	—
Sb	14.3±2.2	—	—	—
Sc	14.8±0.66	—	—	—
Se	—	—	—	1.1±0.1
Sn	(4.2)	—	—	—
Ti	—	5170±370	—	—
V	—	170±15	—	—
Zn				130±10

*Value reported by Das and co-workers(65)

( ) Information value only

Table 3.5 Survey of irradiation and counting detail

Group of nuclides	Facility (Thermal neutron flux, in $ncm^{-2}sec^{-1}$)	Container	Time of irradiation	Time of cooling	Time of counting
<u>Short-lived</u>					
Ti-51, I-128, Br-80, Ar-41*, Mg-27, Cu-66, V-52, Cl-38, Al-28, Mn-56, Ca-49	Pneumatic ($1.8x10^{12}$)	polyethylene vial (ca 1cm^3 capacity) in Rabbit container (2.5cm i.dx8.0 cm)	2 min	2 min	2 min
<u>Medium-lived</u>					
Hg-197, Sm-153, *Np(U)-239, Au-198, Sb-122, As-76, W*-187, Br-82, Na-24, La-140, Zn*-69m, *Cu-64,	Wet tube ($2x10^{12}$)	quartz ampoule (1.2cm i.dx8cm) in aluminium container (4.0 cm i.d x 22.0 cm)	15days (90hrs)	5days	1000sec

Table 3.5 (contd)

Group of nuclides	Facility (Thermal neutron flux, in $ncm^{-2}sec^{-1}$)	Container	Time of irradiation	Time of cooling	Time of counting
K*-42	Lazy Susan ($4x10^{11}$)	Al-foil sheet (3.5 cm x 5.5cm) in aluminium container (2.2cm i.d x 10.5cm)	2 days (12hrs)	18hrs	1000sec
<u>long-lived</u>					
Ce-141, Ba-131, Se-75, Hg-203, Cr-51, Sb-124, Ag-110m, Sc-46, Rb-86, Fe-59, Zn-65, Co-60, Hf-181	Wet tube ($2x10^{12}$)	quartz ampoule (1.2cm i.d x 8cm) in aluminium container (4.0cm i.d x 22.0cm)	15days (90hrs)	18-21 days	3000sec

Table 3.6 Nuclear Data for the Element in head hair samples.

Element	Stable isotope	Nuclide produced	x-sect.[a] (σ, barn)	%Abun-[a] -dance	Half life[b]	Gamma - ray energy (MeV)[b] (Intensity: Number per 100 decays.)
Al	Al-27	Al-28	0.235	100.0	2.31m	1.7788*(100.0)
Br	Br-79	Br-80	8.5	50.52	17.6m	0.511 (5.01), 0.617 (7.2), 0.666 (1.10)
	Br-81	Br-82	3.0	49.48	35.4h	0.54434(72.5), 0.61910(39.6) 0.69836(28.0), 0.77650*(83.20) 0.82779(24.20), 1.04400(28.0) 1.31743(27.0), 1.47488(17.0)
Ca	Ca-48	Ca-49	1.1	0.185	8.70m	1.40890(0.62), 2.2290(0.26) 3.0844*(91.7), 4.07190(7.0)
Cl	Cl-37	Cl-38	0.4	24.47	37.3 m	1.64240(32.8), 2.16750*(44.0)
Cu	Cu-63	Cu-64	4.5	69.1	12.8h	0.5110(37.0), 1.34576(0.48)
	Cu-65	Cu-66	2.3	30.9	5.10m	0.8336(0.25), 1.03900*(9.00)
I	I-127	I-128	6.4	100.0	24.99m	0.44289*(17.50), 0.52662(1.68)
Mg	Mg-26	Mg-27	0.027	11.29	9.48m	0.84376(72.0), 1.01440*(28.0)
Mn	Mn-55	Mn-56	13.3	100.0	2.576h	0.8466(99.0), 1.8112*(30.0) 2.11260(15.50), 2.5230(1.50)
Ti	Ti-50	Ti-51	0.14	5.25	5.79m	0.32000(95.0), 0.92850(5.0)
V	V-51	V-52	4.9	99.76	3.75m	1.43420*(100.0)
As	As-75	As-76	4.5	100	26.3h	0.55910(44.6), 0.65710*(6.4), 1.21625 (3.70)

Table 3.6 (Contd.)

Element	Stable isotope	Nuclide produced	x-sect.[a] (σ, barn)	% Ablun-dance.[a]	Half life[b]	Gamma-ray energy (MeV)[b] (Intensity: number per 100 decays)
Au	Au-197	Au-198	98.8	100	2.697d	0.41180*(95.53), 0.67588 (0.83), 1.08768 (0.16)
Hg	Hg-196	Hg-197	800	0.146	64.1h	0.06881(36.1), 0.07735(19.5), 0.0779(12.63)
	Hg-202	Hg-203	4	29.8	46.59d	0.27917(81.5)*, 0.07287(6.4)
Na	Na-23	Na-24	0.40	100	15.00h	1.36855*(100), 2.75410(199.85)
Sm	Sm-152	Sm-153	210	26.63	46.8h	0.04090(16), 0.04154(30), 0.10412(13)
Sn	Sn-116	Sn-117m	0.006	14.24	14.0d	0.15840*(65)
Sb	Sb-121	Sb-122	6	57.25	2.70d	0.56408(63.0), 0.69276(3.27)
	Sb-123	Sb-124	3.3	42.75	60.20d	0.60271*(98.10), 0.64584(7.2) 0.72278(10.8), 0.96822(1.8) 1.04512(1.84), 1.36821(2.44) 1.69104(50.0), 2.09120(5.5)
La	La-139	La-140	8.9	99.911	40.27h	0.32875(21.3), 0.43255(3.1) 0.48703(45.7), 0.75179(4.5) 0.81580(23.6), 0.867861(5.6) 0.91960(2.5), 0.92525(6.8) 1.59620*(96.0), 2.52183(3.25)
W	W-186	W-187	40	28.4	24.0h	0.13424(10.1), 0.47948(26.6) 0.55147(6.0), 0.68570(32.0) 0.77284(4.8)

Table 3.6 (Contd.)

Element	Stable isotope	Nuclide produced	x-sect.[a] (σ, barn)	%Ablun-[a] -dance.	Half Life[b]	Gamma-ray energy (MeV)[b] (Intensity: number per 100 decays)
Zn	Zn- 68	Zn- 69m	0.1	18.56	13.8h	0.43890(100)
	Zn- 64	Zn- 65	0.46	48.89	245d	0.5110(3.10), 1.11552*(48.8)
K	K - 41	K - 42	1.2	6.77	12.36h	0.3129(0.3), 1.52470*(17.9)
Ag	Ag-109	Ag-110m	3	48.65	250.4d	0.65772*(94.4), 0.67758(11.1)
						0.68695(6.8), 0.70665(16.6)
						0.74426(4.5), 0.76392(22.5)
						0.81799(6.95), 0.88465(73.5)
						0.93745(34.3), 1.38424(25.9)
						1.50495(14.02)
Ba	Ba-130	Ba-131	8.8	0.1-0.13	11.5d	0.12373(32), 0.21601(21.0)
						0.37315(13.0), 0.49670*(41.0)
Ce	Ce-140	Ce-141	0.6	88.48	32.38d	0.14545*(49.30)
Cr	Cr- 50	Cr- 51	17	4.31	27.8d	0.32010*(9.80)
Co	Co- 50	Co- 60	19	100	5.263y	1.17323*(99.88), 1.33252(100)
Fe	Fe- 58	Fe- 59	1.1	0.31	44.6d	1.09927(56), 1.29158*(44.0)
Hf	Hf-180	Hf-181	10	35.22	42.5d	0.13305(41), 0.13625(6.9),
						0.34595(12.0), 0.48216*(83.0)
Rb	Rb- 85	Rb- 86	0.9	72.15	18.60d	1.0786*(8.8)
Sc	Sc- 45	Sc- 46	13	100	83.9d	0.88930*(100), 1.12050(100)

Table 3.6 (Contd.)

Element	Stable isotope	Nuclide produced	x-sect.[a] (σ, barn)	%Abundance.[a]	Half Life[b]	Gamma-ray energy (MeV)[b] (Intensity: number per 100 decays)
Se	Se-74	Se-75	30	0.87	120.0d	0.12110(16.5), 0.13590(58.0) 0.26450*(58.5), 0.2795(25) 0.40070(12.0)

* Energy chosen for quantitation

a Villforth, J.C. and Shultz, C.R. (eds) Radiological Health Handbook Revised Edition U.S. Department of Health, Education and Welfare Public Health Service, Maryland, 1970.

b. Erdtmann, G. and Soyka, W. "The Gamma-Ray Lines of Radionuclides, ordered by Atomic and Mass Number : Part I." J.Radioanal. Chem. 26(1975): 376.