

## CHAPTER 2 EXPERIMENTAL

### 2.1 Apparatus and Material

#### 2.1.1 NAA apparatus

##### 1) Nuclear Reactor Facility

The renewed Triga Mark III nuclear research reactor (TRR-1/M1), operated by the Office of Atomic Energy for Peace (OAEP), Bangkok, Thailand, with the neutron flux at the irradiation position;

Thermal neutron flux  $8.7 \times 10^{11} \text{ n cm}^{-2} \text{ s}^{-1}$  (A4 core tube)

Epithermal neutron flux  $1.0 \times 10^{10} \text{ n cm}^{-2} \text{ s}^{-1}$  (Ca3 core tube)

##### 2) $\gamma$ -Spectroscopy, manufactured by Oxford Instrument Inc., Nuclear Measurement Group, U.S.A., consisted of ;

Tennelec coaxial type HPGE detector (22% relative efficiency, FWHM of 1.64 keV at the 1.3 MeV peak of  $^{60}\text{Co}$ ) and relevant electronics;

- a) High voltage power supply
- b) Preamplifier and amplifier
- c) Multichannel analyzer
- d) Data processing system, with the program Assayer from the software package OxfordWin of 16K MCA 8192 channel.

##### 3) Polyethylene irradiation container ("rabbit") and vials

##### 4) Agate mortar

##### 5) Sieve, 325 mesh

#### 2.1.2 Chemical and material

1) Certified reference material, SRM 2710 (Motana soil) certified value of arsenic in motana was  $626 \pm 38 \text{ mg Kg}^{-1}$ , manufactured by the Nation Institute of Standard and Technology (NIST), U.S.A

2) Arsenic trioxide,  $\text{As}_2\text{O}_3$ , MW 197.84, AR Grade, Merck, Germany

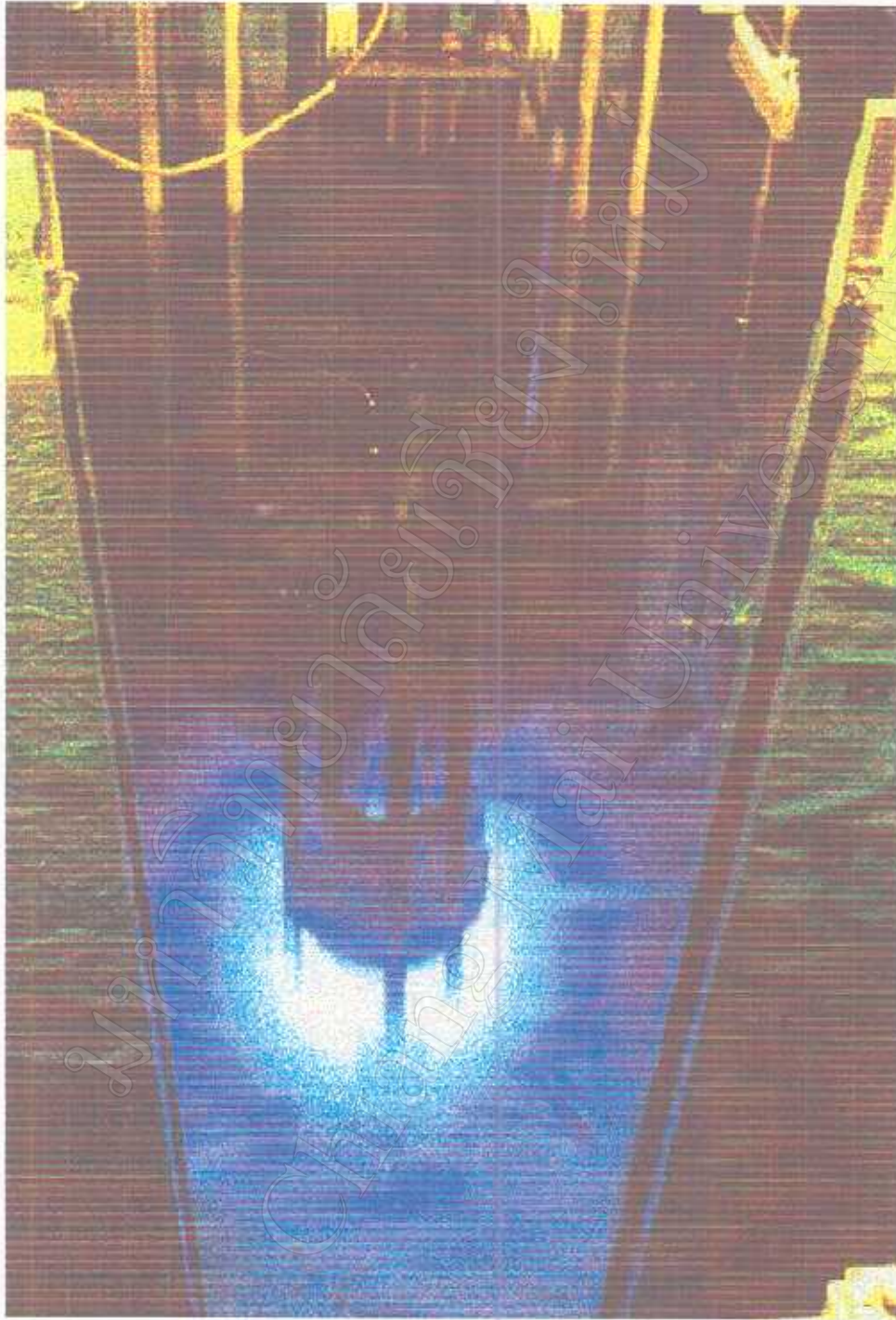
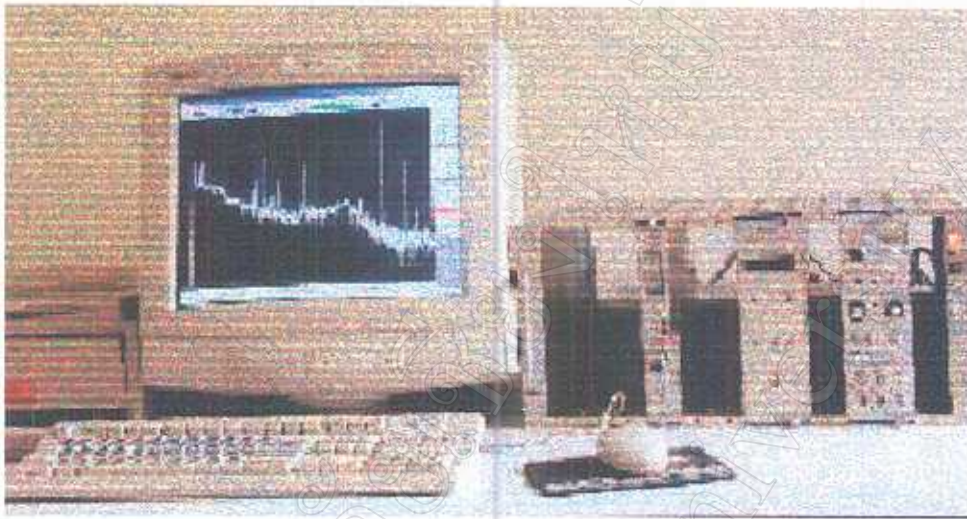


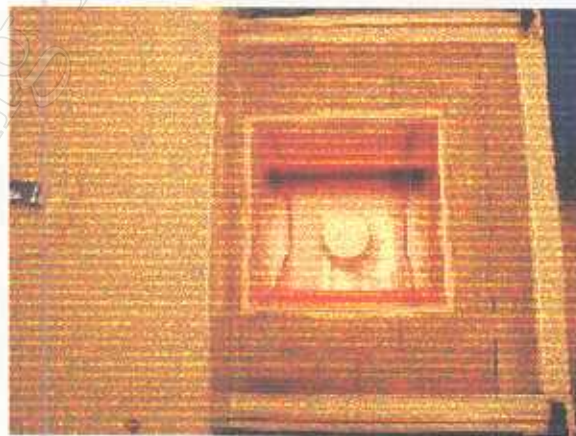
Fig 2.1 The renewed Triga Mark III nuclear research reactor (TRR-1/M1), operated by the Office of Atomic Energy for Peace (OAEP)



a) Relevant electronics of Tennelec coaxial type HPGE detector



b) Tennelec coaxial type HPGE detector



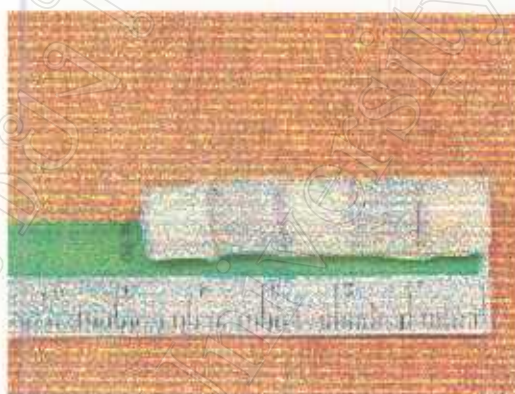
c) Tennelec HPGE detector (top view)

Fig 2.2  $\gamma$ -Spectroscopic system manufactured by Oxford Instrument Inc., Nuclear Measurement Group, U.S.A.

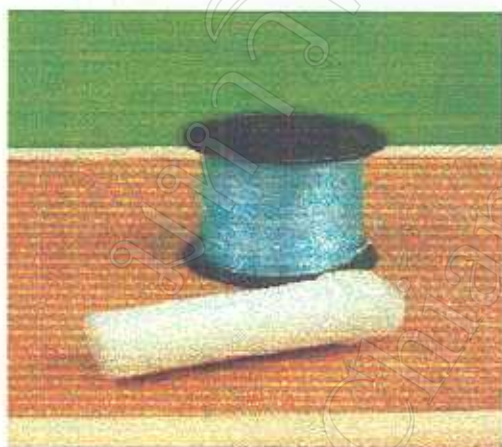




a) Polyethylene vials



b) Sample and standard vials



c) Polyethylene irradiation container  
("rabbit")



d) SRM 2710 (Motana soil)

Fig 2.3 Apparatus and material used in this study

## 2.2 Sample material

About 10 kg of natural agricultural soil samples from five different cites were collected from presumably polluted mining industrial areas in Ronpibul district. The samples were dried in air and reduced to about 100g weight by continued subdivision. The samples were ground and powdered in an agate mortar, sieved through 325 mesh to have uniform particle size and stored in polyethylene bags.

## 2.3 NAA experiment

### 2.3.1 Standard and soil preparation

About 350-400 mg of 5 candidates homogenized soil samples were accurately weighed and added to polyethylene vial and heat-sealed.

For standard, SRM-2710 (Motana soil) was rehomogenized by shaking for a few minutes and aliquots of 100 mg of standard were prepared in a similar way of samples.

### 2.3.2 Preliminary study

#### 2.3.2.1 Qualitative analysis

Five samples from different cites were preliminary analysis. The procedures of irradiation and elemental analysis by  $\gamma$ -spectrometry of short-, intermediate-, and long-life nuclides were applied in both A4 and Ca3 core tubes.

Five candidates sample vials of different cites were wrapped by paper tape, put in the rabbit and irradiated in both A4 and Ca3 core tubes in Triga Mark III nuclear research reactor.

The scheme of the proposed qualitative analysis of soil samples shown in Figure 2.4. [27].

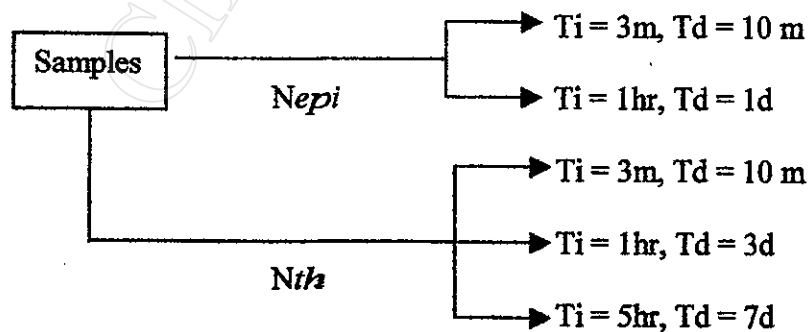


Fig 2.4 Scheme of qualitative analysis of soil samples. (*Nepi* = epithermal neutron, *Nth* = thermal neutron, *Ti* = irradiation time, *Td* = cooling time)

The irradiated samples and standard were allowed to decay for a predetermined times, transferred to clean counting container, then measured on HPGE detector at a distance of 5 inches from the head of the counter. NAA recorded spectra were evaluated with the program Assayer from the software package OxfordWin of 16K MCA 8192 channel. Various radionuclides were identified from their characteristic  $\gamma$ -ray energies.

The chemical composition of soil sample was investigated.

#### 2.3.2.2 Quantitative analysis

The standard and five candidate's samples from different locations were irradiated simultaneously in a rabbit. The standard and samples were placed in the rabbit, with 1 vial of each soil sample together with 1 vial of standard (shown in Figure 2.5(2)). They were irradiated for 5 hr in A4 core tube, cooled for 7 days, counted for 300s on HPGE detector at a distance of 5 inches from the head of the counter and corrections were made for decay. The photopeak area of 559.1 keV  $\gamma$ -ray from  $^{76}\text{As}$  was used to determine As concentration in the sample. Arsenic concentration was calculated by comparison of the photopeak area in sample with that of the standard using the same irradiation periods and  $\gamma$ -ray energies.

#### 2.3.3 Optimization conditions (irradiation and cooling times)

In this section, the position of the measurement was fixed in the procedure and all measurements were made at a distance of 5 inches from the detector end-cap.

Standard and sample soils No 5 were used to optimized conditions. Optimum conditions were determined by varying irradiation and cooling times in both A4 and Ca3 core tubes (the position of vials in the rabbit is shown in Figure 2.5(3)). The irradiation and cooling times used for measurement of arsenic were given in Table 2.1. The irradiated standard and samples were allowed to decay for a predetermined time, transferred to clean counting container and then measured for 300s by HPGE. In all the gamma-counting series, the maximum dead-times did not exceed 10%. Each same batch sample, after their complete decay, could re-irradiate in the next conditions and corrections were made for decay.

**Table 2.1** Irradiation and cooling times in this work for determination of arsenic

Position	Irradiation time	Cooling time
A4	5 min	3 hr, 21 hr
	10 min	1 hr, 4.6 d
	30 min	1 hr, 4.6 d
	1 h	45 hr, 3 d
	2 h	43 hr, 3 d
	4 h	70 hr
Ca3	30 min	4 d
	1 h	4 d

#### 2.3.4 Analysis validation method and flux correction

Five standard vials and five copper wires (served as to determine the flux gradients) were irradiated simultaneously in a rabbit. One cm of 5 copper wire pieces were attached to a thin cylinder of polyethylene at various vertical location in the rabbit (shown in Figure 2.5(4)). The arsenic concentrations in SRM-2710 (certified value of arsenic in Motana soil was  $626 \pm 38 \text{ mg Kg}^{-1}$ ), and all copper wires flux monitor were determined by 30 min irradiation, 1 day cooling, 300s counting, and the photopeaks of  $^{76}\text{As}$  at 559.1 keV were detected by HPGE detector at a distance of 5 inches from the head of the counter. Corrections were made for decay. The copper flux monitors were 2 day for cooling, 300s for counting and the photopeaks, correcting to time zero, of  $^{64}\text{Cu}$  were detected at 1,345 keV on the same detector [29].

#### 2.4 Analysis of soil samples

Five vials of samples and standard were placed in the rabbit; 3 vials sample sandwiches together with 2 vials standard (shown in Figure 2.5(5)). Each rabbit was irradiated for 30 min in A4 core or 1hr in Ca3 core, with 1 day cooling and counted of 559 keV the photopeak of  $^{76}\text{As}$  for 300s by HPGE detector correcting to time zero.

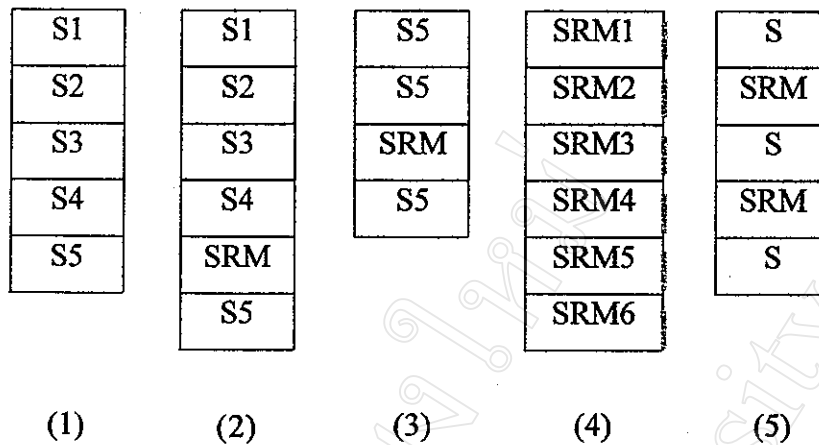


Fig 2.5 The positions of sample vials in the rabbits (S is sample vial, SRM is standard vial and  $\text{---}$  = copper wire)

### 2.5 $\gamma$ -Spectrum evaluation and calculation of results

The quantitative evaluations of the  $\gamma$ -spectra involving peak search, counting statistics, energy calibration, net peak count computation, and isotope identification were made by the Oxford Win-Assayer program.

The amount of arsenic in samples (ppm) was calculated as follows [29]:

$$\text{As (concentration)} = \frac{[\mu\text{g As in standard}](\text{photopeak area of sample})}{(\text{g of sample}) (\text{photopeak area of standard})}$$