A FULLY AUTOMATIC SYSTEM FOR INAA

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ABSTRACT

A fully automatic system at HWRR of CIAE for INAA is described. All processes of the system, for example, change, transfer and irradiation of sample, data collection and treatment and so on are automatically controlled by an IBM- PC/XT micro- computer. Its applications are as follows: fully automatic determination of uranium content by delayed neutron counting method; fully automatic multielement INAA for short, middle and long lived nuclides.

Keywords: Instrument neutron activation analysis Fully automatic Micro-computer

I. INTRODUCTION

The INAA method is widely used in many scientific and technical fields and more and more samples are determined with it in China. Measuring a lot of samples with INAA by man is a tense, tired and tedious work and the operator may receive large radiation dosage from the activated samples. In order to quickly, safely and inexpensively analyse great number of samples, we have built a fully automatic system at HWRR of CIAE for INAA. The IBM- PC/XT micro- computer not only automatically controls all processes of the automatic system, but also can automatically analyse gamma spectrum and calculate the element contents.

The automatic system is stable, reliable and convenient for use. It can save a lot of time and man power, increase the working efficiency of the instruments, reduce the radiation dosage received by the operator and quickly give the analytical results.

II. FULLY AUTOMATIC SYSTEM FOR INAA

The block diagram of the fully automatic system is as Fig.1. It mainly consists of an IBM- PC/XT micro- computer, a pneumatic transfer system, detectors and the electronic circuit.

1. Pneumatic transfer system

The pneumatic transfer system mainly consists of air compressor, oil- water separator, tank, automatic sample changer, selector of sample storage container, diverter, three- way pipes, irradiation tube, counting ends, photo- detectors, solenoid valves, indicators and intermediate relay group and so on. The parts are connected with polyethylene plastic tubes with inside and outside diameters of 19 and 24 mm, respectively.

The capsule of sample is made of phenolic resin bakelite which has good mechanical strength and can resist high temperature. The inside and outside diameters of the capsule are 14 and 17 mm, respectively. Its length is 43 mm. The capsules can be used repeatedly. Firstly the samples are put into the polyethylene sample bottles and weighed and then loaded into capsules one by one. The maximal weight of the sample is about 2 g. The automatic sample changer is made of stainless steel[1], it is placed in the measurement laboratory. The sample magazine of the automatic sample changer is made of plexiglass; it can hold 50 samples. The counting end for DNC is made of stainless steel tube. The counting end for gamma spectrum analysis is made of plexiglass, it can be moved up or down by an electric lifter. The distance between the determined sample and the surface of Ge(Li) detector can be changed from 2 to 100 cm.

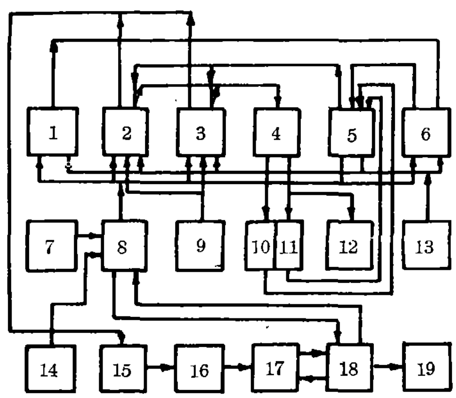

Fig.1 Block diagram of the fully automatic system

1. automatic sample changer 2. BF_3 proportional counter group for counting delayed neutrons 3. Ge(Li) gamma ray detector 4. selector of sample storage container 5. diverter 6. heavy water research reactor 7. low voltage power 8. indicators and intermediate relay group 9. high voltage power 10,11. polyethylene plastic spiral tube 12. lead chamber for storing radioactive samples 13. air compressor, oil—water separator and tank 14. photo- detectors 15. preamplifier 16. amplifier 17. S- 85 multichannel analyzer 18. IBM- PC/XT micro- computer 19. 2024 printer

The irradiation tube consists of three high pure aluminum tubes, which are connected with screw nuts. It is easy to repair or replace the irradiation tube, which is installed in one of the vertical channels of the graphitic reflector of HWRR. The sample irradiation position in the irradiation tube is at the half height of the reactor core. When the reactor power is 10 MW, the thermal neutron flux is about $2\times10^{13}n \cdot cm^{-2} \cdot s^{-1}$. The ratio of thermal to fast neutron flux is about 500. The distance from the automatic sample changer to the sample irradiation position is about 70 m, the time for capsule passing through it is about 4 seconds. The diverter is made of stainless steel. The diverter, plastic spiral tubes and lead chambers are placed in the reactor hall. The functions of the diverter are as follows: transferring activated sample to the counting end in the measurement laboratory with fresh and no active compressed air; turning round the capsule to make the measuring geometry identical and sending the sample to counting end after preset cooling period.

2. Detectors and electronic circuit

The detector of DNC consists of 12 BF_3 proportional counters. The outside diameter and the length of the counter are about 40 mm and 65 cm, respectively. The proportional counters are symmetrically plunged into a paraffin cylinder and connected in parallel way. The paraffin cylinder is the moderator of delayed neutrons. Its inside and outside diameters are 13 and 55 cm, respectively. Its height is about 70 cm. There is a lead cylinder in the paraffin moderator which is used to reduce the gamma ray strength. Four cadmium foils with the thickness of about 1 mm are surrounded outside of the paraffin cylinder to absorb neutrons. The efficiency of the detector is about 11%.

The detector for gamma spectrum analysis is a Ge(Li) detector. Its effective volume and relative efficiency is about 120 cm^3 and 30%, respectively. The resolution for 1332.4 keV gamma ray of ^{60}Co is about 2.2 keV. The detector is put into a lead chamber with a thickness of about 8 cm to reduce the background. The amplifier and multichannel analyzer is Canberra 2021 spectroscopy amplifier and S- 85 multichannel analyzer.

3. IBM—PC/XT micro—computer

The IBM- PC/XT micro- computer in the fully automatic system undertakes four responsibilities.

1) It is a programmed controller The computer controls all processes of the automatic system, for example, change, irradiation, transfer and store of sample etc. Two interface hardwares are added in the computer. One of them is the output interface, it has 14 output channels and every channel controls one executive part, for example solenoid valve, through a intermediate relay. Another interface is the input interface, it has 8 input channels and every channel input a photo- detector signal. According to the input signals the micro- computer can judge if every process is correctly completed. These two interfaces have subroutines written in MACRO ASSEMBLY language. They can be called by the main program written in FORTRAN- 77 language. According to the needs of the measurements the process control programs and input signals can be placed in proper places in the main program, so the use of the system is very reliable, flexible and convenient.

2) It is a clock and timer By calling the GETTIM and GETDAT subroutines of IBMFOR library through software the computer can automatically record sample irradiation end time or gamma spectrum data collection starting date and time, so the system can automatically do decay time correction. The executive time of every process is also set up through calling the GETTIM subroutine. This method is not only convenient, flexible, exact, reliable and cheap, but also stable.

3) It automatically controls S-85 MCA The interface hardware between the computer and S- 85 MCA is modified from the original 8673 board of the S- 85 MCA, it conforms to the RS- 232C series communication standard. The subroutines of this interface are written in Fortran- 77 language. The functions of these subroutines are similar to those of FT- 8673 software package.

4) It can automatically treat a large amount of data The computer can automatically receive the original gamma ray spectrum data from S- 85 MCA and store them in the disc with cooling time, energy calibration coefficients etc. It can also automatically analyse the gamma spectrum, calculate the element contents with relative comparing method and print the analytical results on paper. The analysis program is written in Fortran- 77 language. The method of treatment of data and the mathematical model of the analysis program are similar to that of the Spectran- F gamma ray analysis program. The standards for relative comparing are chemical standards, standard reference materials or both of them.

After preseting the analysis parameters, the automatic system no longer needs any dialogue between the computer and man and any manual operation, except the operator loads samples into the magazine of the automatic sample changer in time.

III. APPLICATIONS OF THE FULLY AUTOMATIC SYSTEM

1. Automatic determination of uranium contents by DNC

When the sample weight is 2 gram, reactor power is 10 MW, irradiation, cooling and measurement time are 60, 10 and 60s, respectively, the detectable limit of natural uranium is about 0.01 ppm. The background counts of the empty capsule and polyethylene sample bottle are about 50. When determining large number of geological explorative samples, the irradiation, cooling and measurement time are 30, 10 and 30s, respectively. In the above conditions if the natural uranium content of sample is 1 ppm, the relative error of the determined result is less than 5%. While counting a sample, another one is irradiated in the reactor, so the automatic system can determine about 80 samples every hour.

During determining sample, many important messages, for example, number of the sample and capsule, whether the sample has left the automatic sample changer, whether the sample has arrived at the irradiation tube, elapse of irradiation time, whether the activated sample has left the reactor, arrived at the counting end, etc. are displayed on the terminal screen, so the operator can clearly know the working situation of the automatic system. The analytical results are safely stored in the disc. After all samples are determined, the computer can automatically calculate the natural uranium contents and print out the analytical report. So far we have determined about sixty thousand samples.

2. Automatic determination of long lived nuclides

After a long time irradiation and proper cooling the activated samples are put into the capsules one by one and then loaded into the magazine of the automatic sample changer. The processes for automatic determination of long lived nuclides are mainly as follows: emptying the counting end and reseting the photo- detector signals; transferring a sample from the automatic sample changer into the counting end; cleaning data and time in the memory of S- 85 MCA and then starting collection of gamma spectrum data; getting the date and time of data collection start and calculating the cooling time of the sample from the sample irradiation end time and data collection start time; getting the original gamma spectrum data of the last sample from the disc and then analysing it; the analytical results, for example, peak energies, areas and errors etc. are stored in another unformated direct file; deleting the analysed original gamma spectrum data file to save disc space; calculating the element contents with relative comparing method and printing the analytical report on paper; after the gamma spectrum data collection completed, transferring them from S- 85 MCA to computer; storing the original gamma spectrum data, cooling time and energy calibration coefficients etc in a formated sequential file in the disc. Above processes are automatically repeated until all samples are determined.

During the measurement, many important messages, for example, number of the sample, whether the sample has left the automatic sample changer and arrived at the counting end, gamma spectrum data collection start date and time, spectrum file number, the results of gamma spectrum analysis etc are also displayed on the terminal screen. When any trouble occurs in the automatic system, it automatically stops measuring and gives a light and sound signal to the operator, so the operator can find the problem in time. If we want to determine these samples again, the determined samples can be stored in a plastic spiral tube. For second round measurement the smples are sent from the diverter into the counting end one by one for measuring.

3. Automatic determination of middle lived nuclides

Firstly, the uranium contents of the samples are determined with DNC method. The irradiation, cooling and measurement time are 60, 10 and 60s, respectively. The irradiation end times of the samples are stored in the disc. After determination of uranium contents with DNC method, the samples are stored in a plastic spiral tube. After decay for 1 to 4 days, the samples are transferred from the diverter into the counting end one by one for measuring. The cooling time of the sample can automatically calculated from the irradiation end time got from the disc and gamma spectrum data collection start time got from the computer clock. The other processes for automatic determination of middle lived nuclides are similar to that of automatic determination of long lived nuclides. If we want to determine these samples again, the determined samples can be stored in another plastic spiral tube. The messages displayed on the screen are similar to those of automatic determination of long lived nuclides.

The advantages of this method are as follows: the accuracy, sensitivity and reliability of determination of uranium content with DNC method[2,3] is better than that with gamma spectrum analysis; the samples are not only used for DNC, but also used for automatic determination of short and middle lived nuclides, so we only need to weigh sample and input weight datum into computer one time for every sample; we can use the delayed neutron counts of uranium chemical standards to correct the change of thermal neutron flux; since the weight of sample is large, this method can reduce the influence of the sample inhomogeneity.

This method can automatically determine Sc, Cr, Ga, Fe, Co, Zn, Rb, Sb, Cs, Ce, Sm, Eu, Yb, Lu, W, Th, Ba, Br, As... element contents.

4. Automatic determination of short lived nuclides

We use fixed timing method[4] to automatically determine short lived nuclides. The sample with a weight of about 1 gram is sent from the automatic sample changer into reactor for 10 to 20s′ irradiation. The irradiation end time of the sample is stored in the computer memory, the activated sample is sent into the diverter. After 15 to 30 minutes′ decay the sample is sent into the counting end for measuring. The measurement time is 200 to 400 seconds. The real cooling time of the sample is automatically calculated from the irradiation end time and gamma spectrum data collection start time. The other processes and the messages displayed on the terminal screen for automatic determination of short lived nuclides are similar to those of automatic determination of long lived nuclides.

Because the sample cooling time is 3 to 5 times of the measuring period, so in the first 3 to 4 cycles no sample is measured. After 3 to 5 cycles there are irradiation, cooling and measurement of samples in every cycle, so the working efficiency of the system for determining short lived nuclides is high. The background of the empty capsule and polyethylene sample bottle is very low and we can firstly determine the background and then automatically deduct it in calculating the element contents.

This method can automatically determine Na, Cl, Mg, Mn, Al, Ti, V, Dy, Ca...element contents.

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