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# Elemental Analysis Procedure using the EDXRF 'Epsilon5' Spectrometry System of Chemistry Laboratory of Atomic Energy Centre, Dhaka

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***Abstract:*** *Upgraded and highly précised spectrometry system is essential for precision, qualitative and quantitative elemental analysis in scientific research as well as industrial applications. EDXRF spectrometer 'Epsilon 5' is a sophisticated, fast and highly précised spectrometry system of Chemistry Laboratory of AECD that is being used for elemental analysis of huge variety of samples from the beginning of its commissioning at February 2009. A huge variety of environmental, industrial and biological samples can be irradiated by the X-ray beam for the identification of elements present in the sample and their concentrations. Skilled knowledge on working procedure and safety systems are the primary requirements to apply the spectrometry system for elemental analysis of solid, loose & pressed powder, metal, foils, filters and liquid samples without any change of their formation. The system is truly able to project the elements and their concentration from Na to the rest of the periodic table. HPGe X-ray detector (PAN-32), a airlock and sample changer, $LN_2$ system are equipped in 'Epsilon 5' spectrometer. X-ray tube, High Tension generator, vacuum system, X-ray safety and protection system, sample handling mechanism, measuring chamber, etc. are within the basic cabinet. Automatic Robotic system is used for sample loading and unloading. All of the functions are controlled by the software 'Epsilon 5' designed and installed in the computer provided with the system.*

***Key words:*** *Beryllium, HT, MCA, X-ray, spectrometry, analytical, HPGe, particle,*

## I. Introduction

In Bangladesh pollution is increasing day by day due to industrialization, urbanization, motorization and agricultural development using artificial fertilizers. Maximum development works are being done without having any proper wastages management system. So the factors involved in the environmental degradation are raising a lot [2, 3, 5, 7]. In this circumstances, no way to avoid the identification of the pollution level of soil, water, crops, air etc. that would help us to take the precaution against it and for the betterment of living beings. EDXRF is one of the best techniques that could be applied for the elemental analysis of a huge types of solid, powder and liquid samples as they are as in the form. A hydraulic press pellet maker is used for 25mm dia and 2mm thick pellets formation according to the robotic sample handling mechanism. All of the operations and controlling systems like pellet handling in X-rays excitation chamber, X-ray tube activation, data spectrum formation and display etc. are performed accurately by the software package designed and interfaced within the 'Epsilon 5' EDXRF spectrometry system.

## II. Major parts and their responsibilities

'Epsilon 5' EDXRF spectrometer has been fabricated as extremely flexible with the external peripherals according customers requirements. This microprocessor controlled spectrometer system is operated from a computer connected via a UTP and USB interface (figure 1). *UTP= Unshielded Twisted Pair, *USB= Universal Serial Bus. The main parts of Epsilon 5 spectrometer and their brief description are –

**Spectrometer**

Spectrometer is an analytical measuring tool used for elemental analysis of different type of samples. The spectrometer beam path should be kept at <10 pa vacuum condition. Gas consumption is .5 L/m+1 L/sample. Cabinet temperature is $35^0$ C. Maximum target is 15 and primary beam filter is 5 (Max.), all are in µm thick within 100-500 µm thick. X-ray Tube, Detector, Counting Electronics, HT generator, Measuring Medium, Optical Path, Measuring Chamber, sample Changer and the Control Panel are included within the spectrometer.

**X-Ray Tube**

The spectrometer is fitted with a Gd anode tube. This is a side window tube whose power supply rating is 25 – 100 kV, 0.5 – 24mA and 600 watts. It needs the internal water cooling.

**HPGe Detector**

High Purity Germanium X-ray detector model: PAN-32 has been used in this spectrometry system [8]. The detector window is made of 8 µm thick Beryllium and the energy range is 0.7 – 100 keV. Counting capability is $10^5$ CPS. This high quality solid state detector with 20 litres $LN_2$ dewar should fill up with $LN_2$ after every 8 days uses. X-ray photons emitted from the targets are detected, converted into voltage pulses and finally projected as the data spectrum with the help of ADC, MCA and the data acquisition software 'Epsilon 5'.

**Counting Electronics**

A digital analyzer has been used to perform the energy dispersion as well as photons counting purposes. The MCA has the signal processing capability upto 16000 channels.

**High Tension Generator**

High Tension power supply is the most important unit of 'Epsilon 5'. The X-ray tube needs the power to be activated that supply is provided by the HT unit. The key switch of HT is on the control panel. By turning 'ON' HT a quarter turn clockwise, the HT power supply automatically switched to 25 kV /10mA. The HT can be switched up to 100 kV by the step of 1 kV and the current can be increased up to 24mA by adjusting with the step of 0.1mA. The indicator (X-ray ON Lamp) shows whether the X-rays are being emitted or not. The HT switch is a key operated and the key switch is a part of the safety circuit and enables the HT Generator.

**Measuring Medium**

In 'Epsilon 5' Energy Dispersive X-ray Florescence spectrometry system irradiation and measurements are done in vacuum. For the analysis of liquid and loose power samples and low Z elements (Z<12) Helium (He) is required that improves the sensitivity of Na →K. Nitrogen ($N_2$) gas is used for the analysis of elements whose Z>15. So, He and $N_2$ options are fitted in this spectrometry system.

**Measuring Chamber**

Measuring chamber consists of Cap, Airlock, Beam Stop and Measurement Chamber. 2 gm of fine powder is pressed by hydraulic pellet maker for the pellet of 25mm diameter. Pellets are set manually on the tray and kept inside the measuring chamber. Holders are also set in the chamber. Automatic sample changing mechanism is applied for handling the holders and samples in and out of the irradiation chamber.

**Sample Changer**

The 'Epsilon 5' spectrometry system has a fully integrated X-Y sample changer that can accommodate maximum 133 samples. The sample changer is operated via a graphical user interface that is configured with drag and drop operations. Different type of sample holders is used for solid, liquid, loose & pressed powder, and filters. The solid pellets are normally 25mm, 32mm and 41 mm in dia. And the thickness is 2mm→30mm; sample weight maximum 500gm. Trays can accommodate different quantity of sample depending on the sample types. These are 8 solid or liquid sample-holder/tray, 8 steel rings(51.5mm)/tray, 21 un-mounted samples(25mm)/tray, 12 samples(32mm)/tray and 10 samples (41mm)/tray.

The sample changer can be used in normal loading and direct loading system of un-mounted samples to a holder in the measuring position. The sample changer is operated by the 'Epsilon 5' software package delivered with the system. The sample changer is calibrated the fine tune, the sample changer movements of the gripper so that samples are correctly transported to the spectrometer loading position.

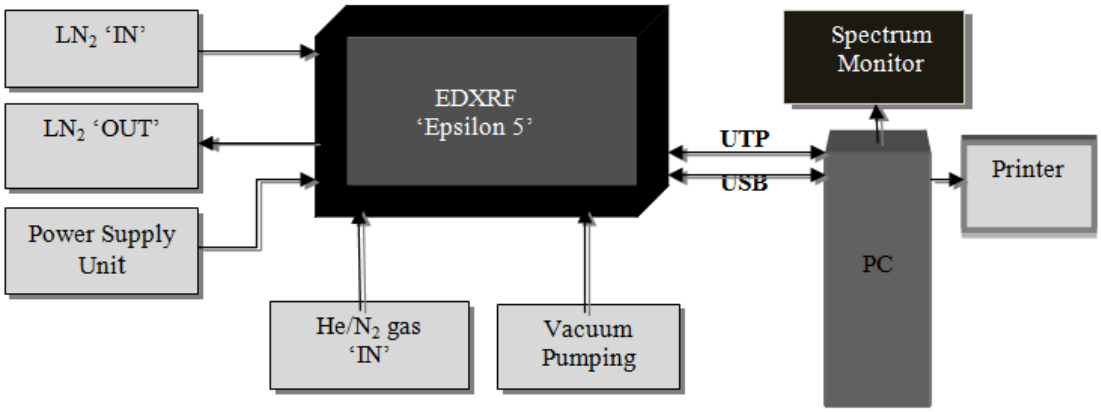


**Figure 1:** Basic Block Diagram of 'Epsilon 5' EDXRF Spectrometry system.

**Control Panel**

A control consol is located on the front of 'Epsilon 5' EDXRF spectrometer system. All the system hardware's control switches are fitted on this panel like 'Power ON' used to ON/OFF the Epsilon 5 spectrometer. HT key operated switch to activate the high tension power generator for the supply of the X-ray Tube. 'HT ON', 'X-ray ON' and 'Free Open' indicator are used for the Sample Changer.

## III. Working Procedure of 'Epsilon 5'

'Epsilon 5' EDXRF with its associated sub-systems is one of the excellent spectrometry systems that used for elemental analysis of different type of solid, powder and liquid samples. The optical path of the spectrometry system (figure 2) is important and essential to understand the basic working process. The X-ray Tube, Doubly Curved Crystal (DCC) Optics, Target Sample, Absorber/Filter, and the Detector with other electronics (preamplifier, amplifier, ADC, MCA) are mainly functioning through the optical path. The data acquisition setup has been incorporated in the system and all of the irradiation process is controlled by the software 'Epsilon 5' designed and installed in the computer by the manufacturer.

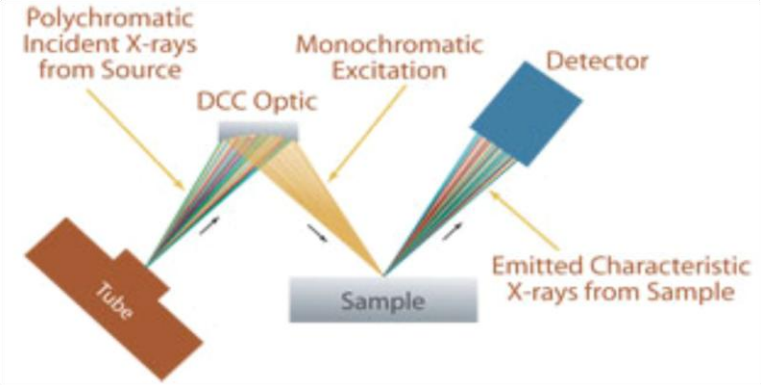


**Figure 2:** Optical Path of 'Epsilon 5' EDXRF spectrometry system.

The X-ray tube is activated by applying 25 kV/10mA power from the HT generator. The indication on the Control Panel shows that the X-ray emission is running and directed to the Doubly Curved Crystal (DCC) optics. DCC optics directs an intense micron-sized monochromatic X-ray beam to the target sample for enhanced elemental analysis. The target samples are exposed by the micron X-rays [2, 5, 10]. Electrons in the inner shells of the atom presence in the sample are given enough energy to cause them to be ejected, resulting in an unstable electron atomic configuration. Thus the electrons from higher shells in the atom then 'drop down' to fill these vacancies, and give off excess energy in the form of X-rays florescence. These X-rays florescence corresponds to the characteristics of the element and therefore can be used to identify elemental composition. These emitted characteristic X-ray photons are detected by the HPGe X-ray detector and converted into voltage pulses through the preamplifier, amplified by the spectroscopy amplifier and inputted to Multi-Channel Analyzer (MCA) through Analog to Digital Converter (ADC). The spectrum data is displayed on the computer monitor with other information. And by measuring intensities of characteristic X-ray lines, can be determined the concentrations of almost all elements in the target samples. The spectrum, projected elements with their concentration can be saved and printed as hard copy documents. The spectrum data of one of the sample is shown in figure 3.

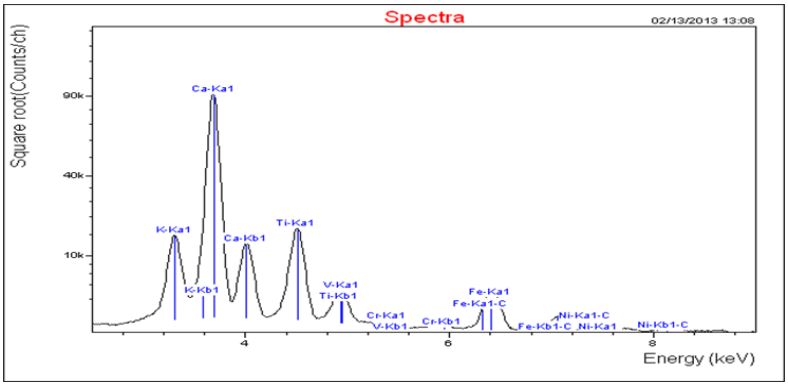


**Figure 3:** Spectrum of a sample irradiated by 'Epsilon 5' EDXRF Spectrometry system.

## IV. Conclusion

EDXRF 'Epsilon 5' is one of the best spectrometry system that used for scientific research and industrial applications, needed in industrial fabrications, agriculture, health and environmental development. This study will provide sufficient knowledge and be helpful to apply the system for elemental analysis of biological, environmental, health and industrial samples.

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## References

[1]. Eric, J. U. (1977). Trace Elements in Human and Animal Nutrition. (4th ed.). Dept. of Animal Science and Production, University of Western Australia, (Chapter 2, 6, 7, 13).
[2]. Islam, M., S., Hosain, M., Jolly, Y., N., Hossain, M., S., Akter, S., Kabir, J., (2015). Geochemical Analysis of the Reservoir Rocks of Surma Basin, Bangladesh. Geosciences, 5(1): 1-7.
[3]. Abedin, M., J., Akter, Shirin, & Arafin, S.A.K. (April 2015). Chromium Toxicity in Soil around Tannery Area, Hazaribagh, Dhaka, Bangladesh, and its Impacts on Environment as well as Human Health. International Journal of Innovative Research in Advanced Engineering (IJIRAE), Issue 4, Volume 2, ISSN: 2349-2163.
[4]. Huque, R., Munshi, M., K., Khatun, A., Islam, M., Hossain, A., Akter, S., Kabir, J., Jolly, Y., N., Islam, A. (2014). Comparative Study of Raw and Boiled Silver Prom fret Fish from Coastal Area and Retail Market in Relation to Trace Metals and Proximate Composition. International journal of Food Science, Volume, and Article ID: 826139.
[5]. Khan, F., E., Jolly, Y., N., Islam, GM., R., Akter, S., Kabir, J., (2014). Contamination status and health risk assessment of trace elements in food stuffs collected from the Buriganga River embankments, Dhaka, Bangladesh. International Journal of Food Contamination Springer Open journal 1:1.
[6]. Hossen, M. L., Islam, S. M. A., Abedin, M. J., Akter, S., Rasel, O. F., Ahsan, M. M., Khatun, R., & Monica, A. N. (2014). Elemental profile analysis of some traditional medicinal plants of Bangladesh using PIXE technique. Journal of Nuclear and Particle Physics, 4(5), 137-141.
[7]. Abedin, M. J. (2012). Study of the effects of industrial pollutants using ion beam analytical techniques, doctoral thesis, Jahangirnagar University,Savar, Dhaka.
[8]. Debertin, K., & Helmer, RG. (1988). Gamma- and X-Ray Spectrometry with semiconductor Detectors. Amsterdam: North Holland.
[9]. Friberg, L., Piscator, M., and Nordberg, G., in "Cadmium in the Environment." Chem. Rubber Publ, Co., Cleveland, Ohio, 1971.
[10]. Jolly, Y., N., Akter, S., Kabir, J., Islam, A., (August 2013). Health risk assessment of heavy metals via dietary intake of vegetables collected from an area selected for introducing a Nuclear Power Plant. research journal of physical and Sciences vol.2 (4), pp.043-051.