



**Al-Quds University**  
**Thesis Approval**

Upgrade of BASEMA XAFS/XRF Beamline at SESAME and Application to  
a Scientific Case

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## Abstract

BASEMA (Beamline for Absorption Spectroscopy for Environmental and Materials Applications) is the x-ray absorption fine structure (XAFS) and x-ray fluorescence (XRF) beamline under construction at SESAME.

This work is concerned with the beamline upgrade from the existing optical components of ROsendorf Beamline (ROBL) donated to SESAME by ESRF-France. The Conceptual Design Report (CDR) for BASEMA focused on improving the performance of the beamline (beam size and flux) proposing adequate optical components for the design and manufacture.

Two cases for the optical design were studied and our recommendation is to use the 2/1 demagnification setup where the distance from the source to the first mirror is twice the distance from the second mirror to the sample. This conclusion was based on simulations that showed the benefits of this designs over the 1/1 configuration, mainly in the lower cost, easy manipulation, high flux, and larger radii.

In addition to the development of the beamline, an application study was driven in parallel. We completed a work initiated by Dr. M. Harfouche in France few years ago that used XAFS to study the local structure in a number of zircon samples around Zr, U, and Th. Our continued work focused on studying reproducing the Zr, U, and Th data in addition to studying for the first time the local structure around lead (Pb) for the same samples considered as analogues to ceramics used to store Long Lived and Highly Active (LLHA) wastes.

Manipulating waste is dangerous and harmful which brought us to use natural analogues to study the structural behavior around actinides within the natural analogues assuming they mimic the matrix used for storing LLHA wastes. The low activity in natural samples is hopefully compensated by their important age factor (one billion years).

The conclusion of our measurements is that replacements destroy the crystalline structure but the size of the cell remains nearly the same which results in a stable trapping of the actinides within the amorphous structure. It is not clear however, if this structure can handle higher doses suggested by the industry for practical storage of waste.

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## **1.1 Introduction and Definitions**

A synchrotron is a cyclic particle accelerator originating from the cyclotron in which the guiding magnetic field (bending the particles into a closed path) is time-dependent, being synchronized to a particle beam of increasing kinetic energy.

The synchrotron is one of the early accelerator concepts that enabled the construction of large-scale facilities, since bending, beam focusing and acceleration can be separated into different components.

The first electron synchrotron was constructed by Edwin McMillan in 1945. The production of the most intense X-ray beams available in the world today is the result of a century of discoveries in the field of X-rays. These beams can only be provided by third-generation synchrotron facilities.

### **How is Synchrotron Light produced**

To gain synchrotron light, electrons have to be accelerated to near light speed. Inside an electron accelerator, magnetic fields force the electrons into circular orbits. The electrons react to this force with the emission of electromagnetic radiation, or Synchrotron Light.

### **The light spectrum and flux optimization**

The spectrum of the synchrotron light is shifted towards short wavelengths due to the Doppler effect of a moving light source.

To make an efficient Synchrotron Light Source one arranges many magnets into a storage ring, where the high energy electrons can circulate for hours. undulators have periodic arrays of magnets with alternating magnetic field polarity. The alternating fields force the electrons to oscillate around a straight trajectory which in turn concentrates the light into discrete wavelengths, and a brilliant beam. Contrary to X-rays, produced in a conventional X-ray tube, the intense synchrotron light beams are sharply focused like a laser beam.

### **Bending magnets**

The traditional source of synchrotron radiation was the bending magnets (used to deflect the electrons from a straight path). When the electrons are deviated by the bending magnets, they emit light tangential to the curved trajectory. The radiation emitted is fan shaped.

## **Insertion devices**

In third- generation machines, the most important radiation emitters are especially designed magnetic systems called insertion devices placed in the straight sections of the storage ring. There are two types: wigglers and undulators. Both are made of a succession of small magnets with alternating polarity. The alternating magnetic fields force the electrons to oscillate around a straight trajectory. The light cones emitted at each bend superimpose, consequently increasing the light intensity. ( [www.esrf.eu](http://www.esrf.eu))

### **Wigglers**

The intensity increases proportionally with the number of magnet and the energy spectrum is continuous.

### **Undulators**

Undulators have more magnets and the light cones from different bends overlap and interface with each other. The magnets of the undulators are mounted on jaws that can be opened or closed. The high-intensity peaks can be adjusted to the required wavelength.

### **Beamlines**

The synchrotron light is guided tangentially away from the storage ring through beam lines to different experimental hutches. Each experimental group has control over its own line and can select its own wavelength.

## **Utilization of Synchrotron Light**

With light beams of a long wavelength one can probe the surfaces of new structures, whereas with the penetrating X-rays one examines the bulk properties of novel materials.

Among the many research topics are:

- Magnetic properties of surfaces, leading to compact magnetic data storage systems.
- High resolution microscopy on surfaces.
- Surfaces with low friction.
- Catalytic surfaces and tracing minuscule contaminations on surfaces.
- Solar cells and new materials relevant for environmental or energy issues.
- High temperature superconductors.

- Spectroscopic analysis of atoms.
- 3d-imaging (micro-tomography) of biological materials.
- The structure of protein crystals. This is important for the development of new drugs and for understanding the human genome.
- Determination of crystal structures for mineralogy, chemistry, catalysis, etc.

( [www.esrf.eu](http://www.esrf.eu) )

## 1.2 History and Evolution of Synchrotrons

### 1.2.1 History of Synchrotrons

**1895** – G. C. Roentgen discovered mysterious rays capable of passing through the human body. He called them X-rays because of their unknown nature. Their major application became medical radiography.

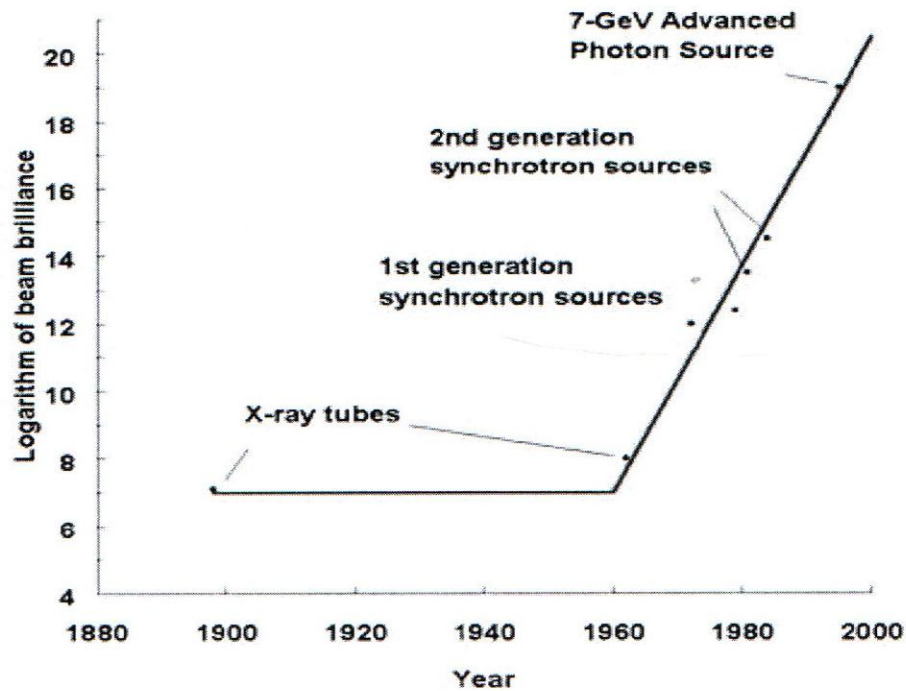
**1912**- M. Von Laue, W. Friedrich and P. Knipping obtained the first diffraction pattern of a crystal using X-rays. This opened the way to using X-ray for investigation the microstructure of materials.

**1913**- W. D. Coolidge invented the X-ray tube which is still used today.

**1970's** – Synchrotron centers are built for academic research into material.

**1980's** – Synchrotron radiation is used for industrial research.

**2000's** – Synchrotron radiation facilities become reference tools for industrial research and quality control in many areas: pharmaceuticals, Specialized chemical...etc. Figure 1.1 shows the beam brilliance increase over time and synchrotron generations.



**Figure 1.1** *The brightness of light beam produced by first, second, and third generation Synchrotrons (www.ESRF.eu).*

### 1.2.2 Synchrotron Accelerators: Pre Injection, Booster, Storage Ring

The Synchrotron machine is mainly divided into 3-4 parts, 1-2 accelerators, a storage ring, and beamlines. Acceleration can be achieved through 2 or three stages and the storage ring can act as accelerator or only store the beam for usage. Beamlines provide stations for experimental testing of various beam wavelengths or intensities. Figure 1.2 shows a typical synchrotron layout.

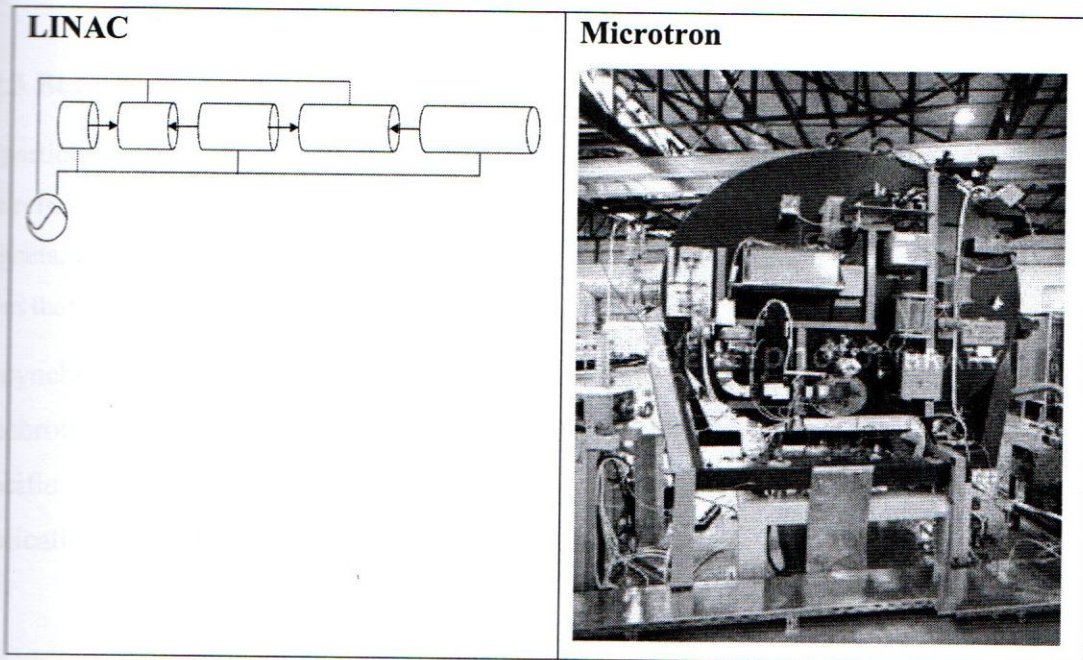


**Figure 1.2** *The layout of atypical synchrotron, showing a pre-injector, booster, storage ring, and beamlines (www.soleil.fr)*

### **Pre injection**

In 1928, R. Wideroe demonstrated that electrons could be accelerated through tube by applying a radio frequency voltage to separated sections of the tube so that the electrons felt an accelerating electric field when they passed the gap. If it was arranged so that the electrons arrived at the next gap at the right phase of the RF voltage (synchronized) they would be accelerated again, doubling the energy for every successful pass.

In a linear accelerator (LINAC) particles are accelerated in a straight line with a target of interest at one end. They are often used to provide low-energy kick to particles before they are injected into circular accelerators. The longest stand alone LINAC in the world is the Stanford Linear Accelerator, SLAC, which is 3 km (2 miles) long. SLAC is an electron-positron collider. Figure 1.3 shows LINAC and Microtron pre-injectors.



**Figure 1.3** Pre injection accelerators ([www.psi.ch/sls](http://www.psi.ch/sls)) and ([www.sesame.orj.jo](http://www.sesame.orj.jo)).

### **Booster**

A circular booster synchrotron is used in which the electrons receive a "boost" from an accelerating LINAC or Microtron each time they go around. In less than one second inside the booster, the electrons make millions of revolutions.

The Booster Ring increases the speed of the electrons close to the speed of light. There are two types of electro magnets shown in the figure 1.4 of the booster ring. The magnetic field is used to direct the electrons around the booster ring. The green quadruple magnets are used focus the bunches of electrons into a fine beam within the vacuum chamber.

### **Storage Ring**

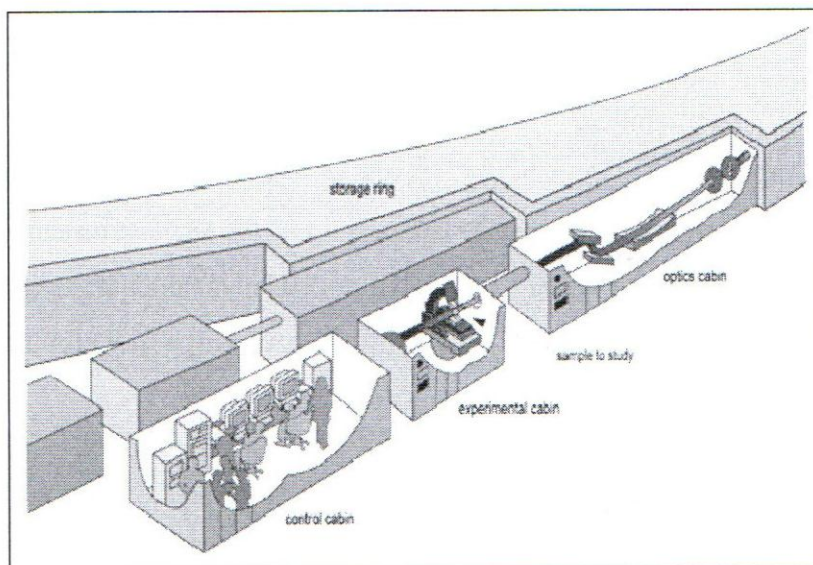
After reaching the target energy in the booster, an injection system transfers the beam to the storage ring. The process of transferring electrons from booster to the storage ring occurs approximately once per second for up to 600 cycles.

In the storage ring, the electrons usually circulate for four to twelve hours producing photons every time they pass through a bending magnet or other insertion devices like wigglers or undulators that are specialized to produce higher flux of certain frequencies. The ring is typically composed of a number of straight sections.

### 1.2.3 Beamlines

In particle accelerators, the accelerated particles are used to interact with targets. The beam line refers to a specific particle path, including the instrumentation segment (vacuum tube, magnets, and diagnostic devices). This part represents a sub-line diverted from the original main beam that can be a linear or a circular accelerator.

In synchrotrons, the beamline refers to the instrumentation that channels the produced synchrotron radiation from bending magnets or insertion devices in the storage ring to a specific experimental end station. Many types of beamlines are built for various applications. Figure 1.4 shows a typical light beamline.



**Figure 1.4** General layout of beamline hutches (optics, Experimental and control), in a synchrotron light source.

### 1.3 SESAME Synchrotron

SESAME is an international organization run under the auspices of UNESCO and it is being under construction in Allan-Jordan since 2008 and planned to operate by mid 2015. Its mission is to promote international collaboration in the Middle East and the Mediterranean region using synchrotron light for basic and applied research in physics, chemistry, biology, materials science, environmental, medical, and archaeological studies, and applications of relevance to the region. ([www.sesame.orj.jo](http://www.sesame.orj.jo))

In this work we present a development of a conceptual design of beamline for X-ray absorption Spectroscopy (XAS) using a superconducting bending magnet as an x-ray source at the 2.5 GeV SESAME storage ring. SESAME is open for members and international groups and offers research opportunities in a wide range of application for the academia and industry. Some special features of SESAME are:

- A wide spectrum of synchrotron light ranging from infrared light to hard X-rays.
- A mixture of straight sections (short, medium and long), allowing an optimal use of a variety of undulators with flexible polarization schemes, giving the possibility to rapidly change the polarization in the kHz range.

### 1.3.1 History of SESAME

SESAME (Synchrotron-light for Experimental Science and Applications in the Middle East) is a “third-generation” synchrotron light source under construction in Allan (Jordan). It will be the Middle East's first major international research centre.



**Figure 1.5** *SESAME site in Allan Jordan.*

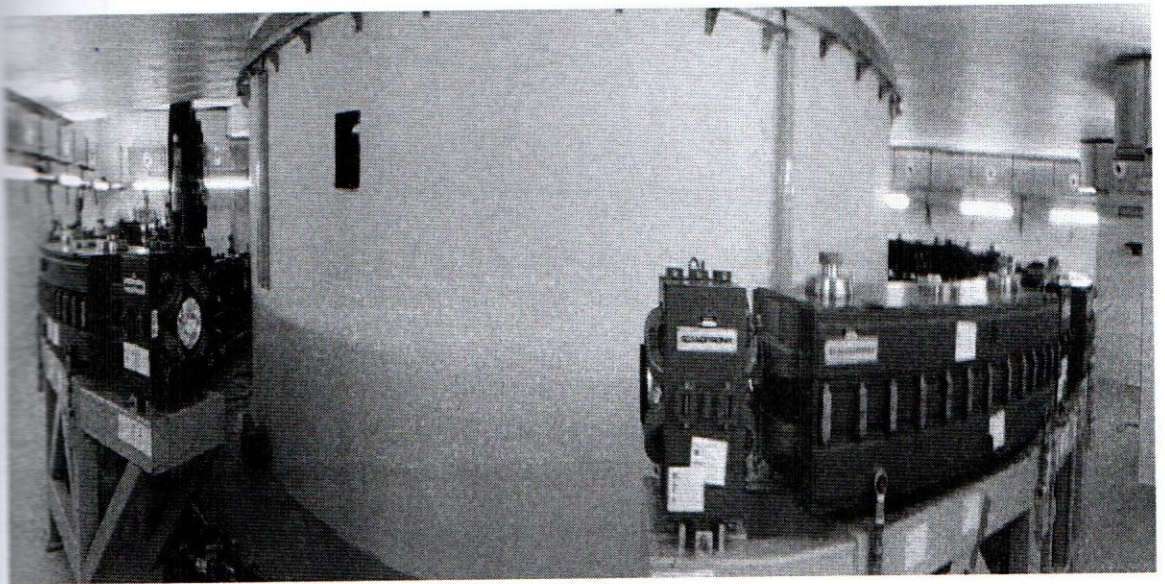
SESAME is the Middle East's first major international research centre in the making. It is a cooperative venture by scientists and governments of the region set up on the model of CERN (European Organization for Nuclear Research).

It is being developed under the auspices of UNESCO (United Nations Educational, Scientific and Cultural Organization) following the formal approval by the Organization's Executive Board (164th session, May 2002). It is an autonomous intergovernmental organization for the service of its Members who have full control over development, exploitation and financial matters.

SESAME is a third generation synchrotron light source. Located in Allan Jordan and hopefully will foster scientific and technological excellence in the Middle East and neighboring countries that will help prevent or reverse the brain drain by enabling world-class scientific research many subjects and build scientific and cultural bridges between various societies, and contribute to a culture of international cooperation in science.

### 1.3.2 SESAME Machine: Microtron, Booster, Storage Ring

The accelerator complex consists of a 22 MeV electron pre-accelerator (Microtron), an 800 MeV electron booster synchrotron, and a 2.5 GeV electron accelerator and storage ring. The Microtron and Booster Synchrotrons already exist, utilizing the donated BESSY I machine from Germany (figure 1.6). The 2.5 GeV Storage Ring is completely new.



**Figure 1.6** *Booster under assembly at SESAME.*

The electrons are emitted by the Microtron with energy of 22 MeV, and then accelerated in a booster until their energy reaches 800 MeV using pulsed electric field see figure 1.6.

The electrons are then transferred to the storage ring and accelerated to reach the energy of 2.5 GeV. Figure 1.7 shows the storage ring layout with outlets for beamlines.

## 4.4 Interpretation and Conclusions

This study shows that the actinides representing nuclear waste in zircon matrix are subject first, to an ionic substitution, where a Zr atom in the original zircon structure is replaced by the actinides. We found also that in the crystalline case zircon structure accepts the substitutes (U, Th) without major changes to the structure.

When the structure of zircon undergoes sever radiation damage, the local structure around the substitute (actinides) suffers from slight deformation mainly in the inter atomic distances where they become larger. This phenomenon was mainly observed in the semi-crystalline structures, thus an expansion of the local structure around actinides has been observed.

In the Metamict zircon the local structure around actinides looked much distorted and the distribution of atoms was highly disordered in contrast to the crystalline and semi-crystalline cases. Moreover, the U and Th occur for sure in zircon structure without forming independent clusters such as  $\text{UO}_2$ ,  $\text{U}_3\text{O}_8$ ,  $\text{ThO}_2$ , etc. This is due to the fact that Si next neighbors were detected in the short range order around the actinides (U, Th) as absorbing atoms in the EXAFS data.

One has to be careful while dealing with the lead case. Care should be taken during data acquisition where a use of a cryostat to freeze the thermal movements of the atoms can be a solution. Moreover, in the data analysis, a lot of care should be given to the choice of the models used for fitting EXAFS data.

One of the results of this study of lead in zircon shows that lead occurs in the structure of zircon with no lead agglomerations or clusters. This was evident in Metamict zircon from Madagascar where an Si atom next neighbor has been detected.

The conclusion of our measurements is that replacements can destroy the crystalline structure but the size of the cell remains fairly the same which can result in a stable trapping of the actinides within the sub crystalline or amorphous structure. It is not clear however, if this structure can handle higher doses suggested by the industry for practical storage of waste.