

GLOBAL JOURNAL OF ENGINEERING SCIENCE AND RESEARCHES DETERMINE GOLD KARAT USING XRF

Maram Albagir Aljaily Alshiekh^{*1} & Sawsan Ahmed Elhourri Ahmed²

^{*1}Sudan University of Science and Technology – College of Science – Department of Physics – Khartoum – Sudan

²University of Bahri - College of Applied & Industrial Sciences-Department of Physics - Khartoum – Sudan

ABSTRACT

In this work we used X-ray fluorescence (XRF) technique to analyze gold specimens and also to identify gold karat; which depends on the internal construct of ten homogeneous samples. Results shows that (XRF) analysis in the region 0.2 Å, three layers were observed in XRF spectrum of all gold samples, top layer Au, Ag and Cu with a different ratios, calculated by stocks instead of grams. Gold alloys are rated using the 24 point karat system, 24 karat gold is pure (100% Au) it includes 1000 stocks of gold. Alloys values were recorded for different gold karats.

I. INTRODUCTION

Gold is a chemical element with symbol (Au) and atomic number 79 makes it one of the higher atomic number elements that occur by nature effect. It melts at 1064°C and boils to a vapor at 2856°C. Gold is a very dense material it has a specific gravity or density of 19.3, this means 1cm³ weighs 19.3 grams while 1cm³ of water only weighs 1 gram. Its rarity and unique properties make it one of the most prized and useful metal, it has many applications in medicine, jewelry, electronics, and other fields. Also it considers as a perfect metal to execute tests according to its superior malleability and ease of fabrication, excellent corrosion and oxidation resistance. It often alloy with other metals in order to improve its durability and malleability, commonly it mixed with copper, platinum or silver [1]. Fire assaying is an old method used to determine gold karat by cupellation and parting. First, the sample portions are weighed, and wrapped in a predetermined amount of lead foil, lead is used as a collector, and the lead oxidizes is absorbed into the copal in a high temperature leaving a precious metal bead. The bead is then dissolved in aqua regia (HCl: HNO₃ solution in volume ratio of 3:1) for analysis by a chemical way. Due to the long time which this method takes in measuring and the defects which causes; we prefer to use x-ray analysis [2]. A general definition of non destructive testing (NDT) is an examination, test, or evaluation performed on any type of test object without changing or altering that object in any way, in order to determine object characteristics such as size; dimension; configuration; or an internal structure, including alloy content, hardness, grain size, etc. NDT is divided into various methods, each based on a particular scientific principle: Visual inspection, Ultrasonic testing, liquid penetrate testing, Radiographic tests, and Magnetic particle testing [3]. X-ray fluorescence spectroscopy (XRF) is one of radiographic NDT method used to analysis gold specimens by getting the incorporated element under investigation to fluoresce and measure the intensity and characteristic energy of the emitted x-rays spectrum.

II. X-RAY FLUORESCENCE

X-rays emitted by sudden stoppage of the fast moving electrons. It can also be scattered with and without losing part of their energy (coherent or Raleigh-scatter and incoherent or Compton - scatter). When an electron from the inner shell of an atom is lost due to some sort of excitation, it is replaced with an electron from the outer shell; the difference in energy is emitted as an x-ray photon which has a wavelength that is characteristic for the element (there could be several of characteristic wavelengths per element). Analysis of the x-ray emission spectrum produces qualitative results about elemental composition of the specimen. Comparison of spectrum of the specimen with spectra of standards of known composition produces quantitative results (after some mathematical corrections for absorption, fluorescence and atomic number) [4,5]. When x-ray beam passes through matter, some photons will be

absorbed inside the material or scattered away from the original path. Most modern stand-alone x-ray spectrometers use x-ray excitation sources rather than electron excitation. All conventional x-ray spectrometers comprise three parts: the primary source unit, the spectrometer itself and the measuring electronics. An x-ray detector is a transducer for converting x-ray photon energy into easily measurable and countable voltage pulses. All detector types work through a process in which interaction between the entering x-ray photon and the active detector material produces a number of electrons [6].

Fluorescence occurs when an orbital electron of a molecule, atom or nanostructure relaxes to its ground state by emitting a photon of light after being excited to a higher quantum state by some type of energy. The atom emits x-ray Fluorescence in the region 0.2–20 Å. Fluorescence is the emission of light by a substance that has absorbed light or other electromagnetic radiation. In most cases; the emitted light has a longer wave length, and therefore lower energy than the absorbed radiation. When the absorbed electromagnetic radiation is intense, it is possible for one electron to absorb two photons; these two absorption photons can lead to emission of radiation having a shorter wave length than the absorbed radiation. The emitted radiation may also be of the same wave-length as the absorbed radiation, termed (resonance fluorescence) [7]. The fluorescence quantum yield gives the efficiency of the fluorescence process; it is defined as the ratio of the number of photons emitted to the number of photon absorbed. Fluorescence has many practical applications, including mineralogy, geology, chemical sensors, cosmic ray detection, and practical analysis [8, 9]. The main principle of x-ray fluorescence spectroscopy (XRF) is based on the fact, that atoms, when irradiated with x-rays, radiate secondary x-rays – the Fluorescence radiation. X-rays need energy to pass through the material to be analyzed and to reach the detector or photographic plate.

On this basis XRF-analysis is possible because:

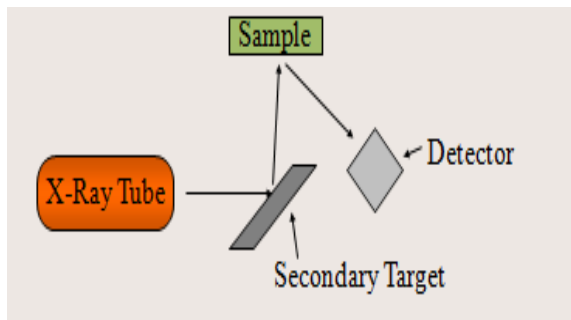
- The wavelength and energy of the Fluorescence radiation is specific for each element.
- The concentration of each element can be calculated using the intensity of the Fluorescence radiation.

The optimum energy for x-ray beams depend on interaction cross section and on the nature (density) of material. When an atom absorbs an incident photon which has an energy that exceeds an electron binding energy, it may result in the emission of a so called photoelectron from the current shell of the atom. This photoelectric absorption process leaves a vacancy in the electron shell. It will immediately be filled by another electron, usually coming from an outer shell and thus less tightly bound, with a subsequent rearrangement of shell electrons. As energy will be liberated when a vacancy is filled, it may be emitted as electromagnetic radiation (a photon) or be transferred to an electron with less binding energy which is ejected from the atom. The probability of having photons emitted is represented by the fluorescence yield. It can be found tabulated among spectroscopic data.

The emitted photons will have an energy representing the difference between the discrete energy levels of the shells involved. These emitted x-ray energies are characteristic of the involved element, enabling identification. Another element will have a different set of possible characteristic X-rays. When characteristic x-rays like these are captured by a detector and the energies identified, it will reveal the presence of the element. The number of detected photons, or counts, is proportional to the concentration of the element in the measurement volume. [10]

High-energy photons induce ionization of inner shell electrons by the photoelectric effect and thus electron vacancies in inner shells (K, L, M ...) are created. The creation of a vacancy in a particular shell results in a cascade of electron transitions, all correlated with the emission of photons with a well defined energy corresponding to the difference in energy between the atomic shells involved. The family of characteristic x-rays from each element including all transitions allows the identification of the element. X-ray Fluorescence (XRF) device which we used; is one of Oxford instruments productions. X-strata-920 works by exposing a sample to be measured to a beam of primary x-rays. The atoms of the sample absorb energy from the x-rays, become temporarily excited and then emit secondary x-rays. X-strata-920 is a non destructive analyzer present rapid analysis of solids. It generally used for jewelry and alloys analysis in which it present precious metal alloy assay, karat analysis and material identification. This device contains Powerful Smart-link software with pre-loaded calibration parameters for easy setup. The picture below shows

x-strata-920 with its annex computer.



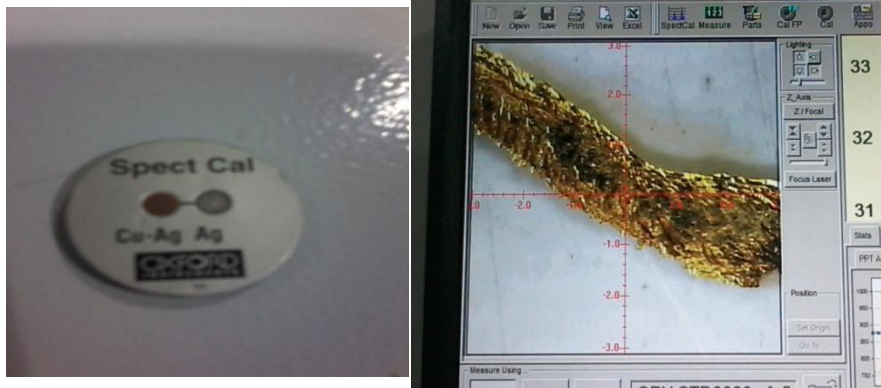
X-strata-920 XRF device the device analyze up to three layers, top layer Au (gold coating thickness), second layer Ag (silver coating thickness), the third layer Cu (copper coating thickness). It may analysis more layers such as palladium, platinum, nickel; if we have the identity standard disc of the required measurement.

III. GOLD TESTING

The famous and canonical types of gold karats in Sudanese standard and metrology organization are:

- 24 karat gold covers a highest ratio of gold; it's valued at around 999.9 gram from the total quantity 1000 stocks.
- 21 karat gold contains 875 stocks or grams of Au, the remaining stocks are copper, silver or zinc or other minerals. Most high quality gold jewelry is made of 21 karat gold.
- 18 karat gold contains about 750 stocks; it has a different color than 21 karat because gold ratio is less. Most rose gold and pink gold is also 18 karat.
- 14 karat gold contains about 583 stocks or grams and the remaining stocks are another metal.
- 9 karat gold has a lowest percentage of gold reach to less than 400 grams in a kilo gram; this is a cheapest type of gold jewelry. If the other metal in the alloy is copper, it will look a dull bronze color and if it is silver it will look white gold color.

After receipt gold species from goldsmiths in a Sudanese standard and metrology organization so as to determine gold karat and sealing their pieces; we chose several specimens from both jewelry and bullions [11]. Jewelry samples were put in specialize envelopes in which their data recorded, also we made gold bullions ready to tolerate x-ray testing, small chunks were taken from the whole pieces by a cutter machine (Electric mini drill), they put in same envelopes and sent to x-ray laboratory to start testing. Before x-ray start working, temperature and air moisture were registered by a digital thermo-hygrometer device, then we turned the device on and selected the application which can define the ratio of three elements, gold, silver and copper from the total ratio 1000 stocks. We started calibration operation to make sure that results or reading values are exactly correct [12, 13]. Calibration were done in two steps, the first step is an internal calibration in which a pure specimen of silver and copper submitted to small-spot x-ray for 240 second, it's reading became as a standard reference. The second step is an intermediate check, here the measured standard which define karat was calibrated (usually we check a pure specimen of 21 gold karat which contain 875 stocks of gold ratio); calibration time for this intermediate check was 60 second.



After calibration operation, we opened computer and x-ray producer device at first so as to give enough time to generate x-ray. gold samples were putted on XRF device; and then we choose a flat smooth surface of the specimen to illuminate x-ray on it, the radiation direction was dropped by using a class two focus laser which use here as a direction controller, XRF radiation is induced when photons of sufficiently high energy, emitted from an x-ray source, impinge on a material. The characteristic x-rays from each element including all transitions allows the identification of the element. Results displayed within seconds.

IV. RESULTS

Jewelry Testing

Specimen Type	Au ratio	Ag ratio	Cu ratio	Gold Karat Type
Imported gold/bracelet	893.6	5.0	101.4	21 gold karat
Imported gold/pink/ ring	877.0	21.2	101.8	21 gold karat
Imported gold/ ring	851.8	16.2	132.0	18 gold karat
Local gold/necklace	584.3	126.2	289.5	14 gold karat
Local gold/necklace	886.0	4.7	109.3	21 gold karat
Local gold/bracelet	822.1	33.6	144.4	18 gold karat

Gold Bullions Testing

Specimen Type	Au ratio	Ag ratio	Cu ratio
Local gold/pure	999.9	0.0	0.0
Local gold	885.5	111.0	3.5
Local gold	912.8	82.9	4.3
Local gold	900.0	97.8	2.2
Local gold	873.4	121.3	5.4

V. CONCLUSION & DISCUSSION

XRF can be successfully utilized for analyzing materials which contain gold. Jewelry testing results explained three different types of gold karat. It had been noted that according to Sudanese Standard and Metrology Organization SSMO: (14 gold karats contain about 58.4 % of Au, 18 karat contains 85.1 % of Au, and 21 gold karat contains about 88.5 % of pure gold. Copper and silver ratio specifies gold karat, color, yellow and pink gold, red and

white gold. In jewelry results we noticed that usually copper ratio is higher than silver ratio in different jewelry karats, because of the cheap prize which copper possess. Meanwhile its color (near to gold); jewelers generally used plenty of it in jewelry. Gold bullion results showed elements ratio in five chunks, one of them is pure gold bullion which didn't contain alloys, the other bullions had high gold ratio. Goldsmiths concern about gold quantity in the bullion to re-create it by adding alloys until gold ratio reach to the closer karat of jewelry. X-ray fluorescence method saves wasting time, it shows testing results in 60 seconds only and allows to analysis around a hundred samples alternately. It should be put in mind that XRF is unable to see deep inside the metallic sample due to absorption and it doesn't characterize element isotopes from each other, it will give one analytical results. Application selection in x-strata920 device depends on the kinds of standard reference disks. In this experiment we used two standard references application which include two pure samples of alloys; silver and copper. If there is another alloy in the testing sample, as example: platinum, nickel, zinc....etc; the computer will not define it, its value will join to gold ratio spontaneously and increase it, this will affect on karat kind accuracy. To solve this problem we must possess at least the most adhesion alloys reference disks which are usually neglected, although its high cost (one alloy disk worth about 500 dollars); so as to comply accuracy results in calculating karat values

REFERENCES

1. *Neville F.Mott, H.Jones, The Theory of the Properties of Metals and Alloys, 2015*
2. *Bayley,J, 1995. Precious Metal Refining, in historical Metallurgy society (accessed january13, 2014)*
3. *Charles Hellier, Handbook of Non destructive Evaluation, McGraw-Hill, 2003*
4. *Marcelo Alonso, Edward J. finn, physics, 2013*
5. *Elizabeth M.Slyter, Henry S.Slyter, Light and Electron Microscopy*
6. *Kenneth S-Krane, Introductory Nuclear physics, 3th edition*
7. *C. Leroy and P. Rancoita, Principles of radiation interaction in matter and detection, 2nd Edition, 2014*
8. *Clark, George l, Applied x-rays, 2nd Edition, 1932, McGraw Hill company*
9. *Graham Hill, Chemistry Count, 2014*
10. *Lakowicz Joseph R, Principle of Fluorescence spectroscopy, 2010, 2nd Edition*
11. *Reinhold Klockenkamper, Total Reflection X-ray fluorescence analysis*
12. *X-strata-920 Brochure, Oxford Instrument*
13. *Sudanese standard and metrology organization library*