

or: Looking inside the ATOM!





Humans love to test things. Indeed it is probably a survival response. Humans and animals do this everyday if they realize it or not. Who has not lifted an obect to determine its heft, touched to evaluate texture, examined it visually very closely, sniffed to determine clues as to freshness and chemical content, pushed, scratched, kicked or knocked on, even tasted something?

Follow any shopper in a clothing store, shoe emporium or food market to see it in action. The term "kicking the tires" describes an action that is so common as to be cliché.

Amateur scientists have a particular desire or need to test things a bit further.

Materials testing and measuring comes in two basic forms, Destructive and NONdestructive.

Collectors of a variety of items must determine: hardness (Mohs scale, Brinell Hardness Number etc), density (specific gravity), weight, volume, color, thermal conductivity, magnetism or suscepibility, chemical composition, heat of fusion, melting point, temperature, strength, durability and so on.

Most of these testing activities can be carried out non-destructively, such as by weighing or measuring with a ruler. Other parameters are by nature destructive, one cannot determine melting point without melting something! Not all items lend themselves to destructive testing, one example comes to mind of a young scientist who wanted to determine of the family silverware was real silver or just silver plated (-sorry about that mom!).

Moviegoers know about RADAR and SONAR, both used to help locate and identify objects at a distance by "pinging" them with an energy pulse, then watching for a return signal, radio frequency waves for RADAR and sound waves for SONAR.

By a different mechanism, we can "ping" materials with an external energy, be it charged particles, X-Rays, Gamma Rays etc. thereby eliciting a response. Not an echo in this case but a completely new X-Ray energy, which happens to be specific for each element on the Periodic Table.

Here's how it works- atoms have a positively charged nucleus surrounded by shells of negative electrons. These electron shells are arranged in a certain order for each different element. In a stable atom, the number of positive charges is neutralized by the same number of negative charges. Under some circumstances, an inner electron can be caused to gain EXTRA energy, making it now unsuitable to fill its former spot in the intricate balance of the mother atom. This electron goes rogue, leaving behind a void or hole where it was, which must be filled and quickly. Fortunately there are a lot of other electrons around willing to take up residence there!

Upon settling in to its new home the donor electron must somewhat adjust its energy level to fit in with the neighborhood. THE EXCESS ENERGY IS GIVEN OFF AS AN X-RAY.

This X-Ray has an energy level distinct for each shell and each element; this is the unique "characteristic" signal we look for after "pinging" an element. Each and every energy signature is specific to the origination element. They are known as "Characteristic X-Rays".

When the K shell is the one excited, there are two possible sources for the electron to fill the hole. The first and slightly more likely donor electron is from the adjoining L shell, and the energy difference is called the K-alpha (Ka) energy. It is also possible for the donor electron to come from the M shell, in which case it is called K-beta (Kb), and gives a slightly higher net energy release.

When the ejected electron is from a different shell, say the L shell, the same convention is applied, L-alpha (La) if filled from the next outermost shell, L-beta (Lb) for the second outermost shell and so on.

For our purposes you need to understand these major points:

• Each atom of a given element has a specific and unique number of protons in the nucleus.

That number, called the Atomic Number, (Z) is what determines which *ELEMENT* the atom is.

All atoms of the same element have the same number of protons. If for any reason the number of protons changes, the atom is now a different element. Simple.

Mnemonic: "proton<u>Z</u>"

- Although the number of protons in an *ELEMENT* is fixed, the number of neutrons can vary. Add the number of neutrons and the number of protons to derive the approximate Atomic Mass number (A). Different mass numbers of the same element are called *ISOTOPES*. Mnemonics: "protons <u>A</u>nd neutrons"
- Electrons "orbit" the nucleus in discrete "energy shells". Starting nearest the nucleus is shell K (2 electrons max), then L (8), M (18), N etc. as needed to accommodate the correct number of electrons. <u>*Each shell has a particular associated ELECTRONIC BINDING ENERGY*</u>
- Electrons in lower shells (nearer to the nucleus) have and need less energy than farther away ones.
- A result of "pinging" an atom with external energy, an inner electron can absorb some of that energy, leave its shell, be replaced by a different electron, which gives up its excess energy as a Characteristic X-Ray. Very specific energies depending on the original energy of the donor electron. We measure the energy level of those X-Rays to determine what element caused them.
- The technique, no matter what form of energy starts the process, is called XRF for X-Ray Fluorescence. Sometimes the whole term EDXRF for Energy Dispersive X-Ray Fluorescence is used. And finally: when the excitation is by way of alpha particles, the techniques is called APXS for Alpha Particle X-Ray Spectroscopy.
- Our home made apparatus uses variously: beta particles (PIXE), alpha particles (APXS) and X-Rays or Gamma Rays (EDXRF). The best results are with alpha particles for surface elements plus more penetrating Gamma Ray sources for the deeper elements. It so happens that the radioactive source in a cheap ionization smoke detector has both alphas and Gamma Rays.

The following chapters discuss the hands-on, DIY building procedures to create a viable demonstration apparatus, which with the application of different heads, can detect and identify stable as well as radioactive elements.

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Chapter 1

Beta Particle Excitation

IDENTIFYING ELEMENTS VIA XRF> X-RAY FLUORESCENCE the natural characteristic X-Rays unique to each element on the Periodic Table of the Elements

by George Dowell

For a long time XRF or X-Ray Fluorescence has been used to identify elements by exciting them with an external form of energy (X-Rays) and reading the resulting "Characteristic X-Rays" subsequently given off by those elements when they relax back to stability. Commercially, detection is done with extremely expensive, usually cryo-cooled, detectors.

Similarly, the same process occurs in a Scanning Electron Microscope using beams of electrons as the exciting energy. Micro beams allow very precise areas of a sample to be analyzed, one by one. The technique is named Scanning Electron Microprobe.

Since we don't have a handy X-Ray micro beam or Scanning Electron Microprobe here in the Home Lab, much less an HPGe probe cooled by liquid nitrogen, we use what we have: inexpensive, small sealed exempt quantity radioactive test sources, mostly by Spectrum Techniques - and a proportional type or scintillation type detector fed into a student grade MCA- in this case we use a UCS series MCA by Spectrum Techniques. My favorite excitation source has been Kr-85 but most will work, even Sr-90 or Cs-137.

Exciting the elements has never been a problem. Measuring the resultant K shell X-Rays has been a challenge however, due to the rather low energy range, 2 to 75 keV for elements between aluminum and lead.

Lead (Pb) has always been the easiest to identify and we have done so in lead crystal glass and many other objects using our amateur methods.

Most scintillation probes will give a decent spectrum at 75 keV, even as low as 20-30 keV depending on the housing material. Tin / Lead solder gives a nice double peak, one from tin, the other from lead. Peak amplitude (corrected for detector efficiency) is an indication of alloy percentages.

Thick section crystals as we all know by now detect higher energy gammas and X-Rays rather well, and even respond well to low energies if the housing material allows them to get inside to the actual crystal. A disadvantage to using a thick crystal, even one with a thin beryllium window is of course the over response to higher energy stray radiation.

A solution is to use a thin crystal with a thin window. Higher energies will pass right through while lower ones will be efficiently absorbed. Indeed this is the design of all LEG or Low Energy Gamma probes, such as the Ludlum 44-3 or the FIDLER. Such probes are automatically physically tuned to the 14-17 keV X-Ray energies of Plutonium, seeking which is their prime usage in industry and military. Almost always such probes are used in SCA / Windowed metering circuits, thereby further peaked electronically at 10-20 keV.

Such a thin crystal, typically only 1 mm thick, is sensitive enough but does not provide the required response to give a really nice looking spectrum on an MCA.

All available LEG's have been tried (NaI(Tl), CaF2, CsI etc.), all work but none do the job with sufficient precision using our amateur methods.

Home Lab experiments over the years have indicated that a sodium iodide, thallium activated crystal of about 4mm thickness, inside a beryllium windowed housing SHOULD give ideal results under our circumstances.

Scionix made one to my specs and the results are amazing. Scionix Model GEO 4mm! FWHM about 9.3% @ 122 keV Co-57.

FIG.1> Setup showing Beta particle excitation sample and sensor arrangement:

FIG.2> Results showing specific energy signature for Cobalt alone:



FIG.3> Setup using small piece of Indium solder FIG. 4> Results of the scan for Indium



Historically, one of the nifty lab items we have used in the Home Lab for selective energy filtering is a metal disc, 2 3/4" diameter and 0.85mm thick, removed from an old Ludlum made uranium enrichment analyzer. The material it was made from was never specified, nor could Ludlum identify it after so many years. I assumed it was either tin or cadmium, since its qualities were similar to either. Weight/density and hardness testing leaned more towards cadmium. Since cadmium is a somewhat toxic metal and this filter never had a warning label or anything else to alert an operator, tin would be a more likely choice. Anyhow to be safe I labeled it as Cd and always handled it accordingly. Still it has bugged me not knowing for sure.

Today I know for sure. It is cadmium; nearly pure the best we can tell. We have metallographic samples of 0.9999 pure elements, one of which is cadmium. By shining the rays and particles from the Kr-85 source onto the various metals, a signature is developed using the Scionix probe into an MCA, for each element. The filter- "Device Under Test" or DUT matches perfectly the 0.9999 Cd metal sample.

FIG.5> Results of .999 pure cadmium element test. Peaks coincide exactly with pure cadmium element:

<u>FIG.6> Results of unknown metal filter test</u> showing unique energy lines of cadmium



FIG. 7> Setup using old US dime coin to test for silver: <u>F</u>.

FIG. 8> Results of a test of pure tin element (Sn)



Observe the specially made Periodic Table of the Elements we had made (Thanks AtomicDave!). Instead of protons and neutrons these charts give the characteristic XRF energies of the two K shell and two L shell electrons.

Our method of beta excitement works mostly on the K shell, so the energy read with our probe is roughly an average of both the K shell energies together (favors lower energy about 5/7). Each element can therefore be identified very easily by comparing it to a known sample. Furthermore, some alloys, like Bronze can be identified by a double peak, one from copper the other from tin. Two elements that are right next to one another on the chart will merge usually, but either in pure form can easily be identified. See Appendix 1, and these XRF CHARTS LINKS:

8.5" X 11" Size: http://www.qsl.net/k/k0ff/XRF%20Periodic%20Table/PeriodicTable3.pdf

11" X 17" Size: http://www.qsl.net/k/k0ff/XRF%20Periodic%20Table/PeriodicTable11x17.pdf

24" X 26" Wall Chart Size:

http://www.qsl.net/k/k0ff/XRF%20Periodic%20Table/PeriodicTable24x36.pdf

Using beta particle excitement will identify only the surface elements, not what is beneath. An example is a modern USA penny. Once made from mostly all copper, today they are made of zinc, with a very thin copper flashing. If you remove the copper from one side and analyze each side separately, the copper and zinc are easily discerned even though they reside each other on the chart. If one were using X-Rays to excite, they would penetrate further into the clad coin, and would give a merged spectrum or two peaks.

This series of experiments were designed as proof-of-concept, not as a superior invention of a working apparatus. No one would build such a machine when better ones are available, (at a somewhat elevated cost today of about \$30,000 USD! see Niton XRF)

More advanced experiments following this same basic technique will be shown soon, with which we have identified all the elements from Calcium through Bismuth.

George Dowell

Chapter 2

Refining the Adaptor for Simultaneous Surface and Subsurface Alpha Particle plus X-Ray Excitement

Alpha Particle X-Ray Spectroscopy X-Ray Fluorescence in the Home Lab or: Fun with Quantum Physics

by George Dowell

My recent CSL article (see above, Part 1)

introduced you to X-Ray Fluorescence - the method of exciting an element's atoms and reading off the Characteristic X-Rays given off when the atom de-excites back to a stable state.

FIG. 1: XRF via beta particles, also known as PIXE (Particle Induced Radiation Emission):



Any number of methods may be used to excite ordinary atoms to fluoresce in this manner. Just about any energetic charged particle will do it, as well as ionizing electromagnetic photons, such as Gamma Rays and X-Rays. The first article in this series used small radioactive beta sources to provide that excitation. The only requirement is that the energy imparted to the target atom exceeds the binding energy of the particular electron shell involved. This binding energy is unique for each and every element and each and every electron shell within that element.

The process involves an inner electron being first ejected from the atom buy the added energy, then being replaced in its shell by a donor electron. When we measure the resulting XRF, what we see is the difference energy between the kinetic energy state of the donor electron, minus the binding energy of its new orbital shell. Therefore each and every element has a "Characteristic X-Ray" signature for each of its electron shells.

When a pure beta source is used to excite, the beta particles penetrate the target's surface only slightly, therefore betas are used in such applications as coating thickness analysis, and surface analysis.

If we were to use X-Rays, they of course penetrate deeper, causing elements beyond the surface of the target to become excited. Sometimes, if those atoms are no too deep in the target, their resulting fluorescent X-Rays will break through the surface, allowing them to be measured. A rule of thumb is, the higher the Atomic Number the more self-shielding is an issue.

Introducing APXS, *Alpha Particle X-Ray Spectroscopy* - a form of XRF using alpha particles to excite the target atoms. Traditionally element analysis used heavy lab equipment, X-Rays and liquid nitrogen cooled sensors. Today's world required portable, lightweight equipment designed to be used away from the Home Lab, out in the field. No better example of remote operation is Mars! Mars Rover Curiosity uses XRF with excitation provided by alpha particles, a relatively new technology

Curiosity uses some pretty strong, military grade radioactive sources, like Curium, which are unobtainable to Citizen Scientists.

NASA REFERENCES: http://mars.jpl.nasa.gov/msl/mission/instruments/spectrometers/apxs/ We do however have a pretty potent alpha particle source not only available but downright cheap! Ionization type Smoke detectors, presently available at Wal-Mart for less than \$4 USD will supply the only excitation needed. They contain less than one microCurie of Am-241, in a safe configuration, that can be owned without license and disposed of in any trash can when finished.

Still we caution young students to seek adult supervision. We should note that devices containing these elements are NRC controlled, and cannot be sold or resold without a specific license (so don't try to make and market them).

IMPORTANT NOTE: Depending on the country you live in, these may or may not be available, plus other restrictions might apply.

Am-241 gives alpha particles and some low energy photons, both of which combine to make an excellent excitation source, exciting the surface with the alpha particles and deeper with the photons. This is a distinct advantage as we will see in later experiments. From one to eight smoke detectors are required, but no more are ever needed. Because the X-Ray source is so weak, for elements above Lu, we rely on their L shell XRF lines instead of their K shell lines. A few really massive elements require we look at their M shell lines.

Before we go into the adaptor containing the exciters, let's talk about the sensors and the one selected for use in this project.

Sensors specifically designed to measure very low energy X-Ray and Gamma Ray photons have to have some pretty unique qualities. First they have to have a housing, or at least a "window" made of very lightweight material. Anything too robust will simply block the desired photons by absorption. Next they have to be sensitive to the energy range in question, and finally they must give a signal output that is proportional to the amount of energy detected. After all, we are judging our results by the energy given off an atom, so we must be able to measure that energy.

Geiger Counters are well known, and some even will respond to low energy photons, but DO NOT give any energy information. All the ''clicks'' heard in the speaker are the same, no matter what sort of radiation caused them, .Geiger Counters are ruled out for XRF.

Another sensor called a Proportional Detector DOES give energy information, and have been used in commercially made XRF machines for a long time. They have some disadvantages, namely:

1) They are VERY expensive.

2) They require THOUSANDS of Volts to operate.

3) Their output signals are VERY WEAK, requiring expensive and fragile preamplifiers to be used

before the MCA.

On the plus side, they do operate at room temperature, so don't need liquid nitrogen. For this reason alone proportional detectors have been used before superior but not more affordable technology has recently become available.

Next we will examine the scintillation detector. These types of sensor work by emitting a flash of light from a crystal when radiation strikes it. These small flashes are picked up these light pulses, convert them to photoelectrons, then greatly amplify those photoelectrons. Some have an amplification factor of one million or more. Once amplified, these electrical pulses are analyzed by our Multi Channel Analyzer, and their height determined individually. The height of these pulses is what contains the originating energy information- the higher the pulses in milliVolts, the higher was the originating radiation energy. This whole process is called PHA or Pulse Height Analysis, and is the basis for all the scans that I show related to radiation energy. On the scan, the bottom axis increases as per energy, while the vertical axis increases by number of pulses. Later on we will publish many tutorial that explain each and every aspect.

Traditional scintillation detectors used a crystal of sodium iodide, activated with a small percentage of thallium, These are very efficient, but sodium iodide is hygroscopic, which means it absorbs moisture like a sponge. Sodium iodide left in the open will turn to slush overnight.

Recent developments in crystal technology have allowed a new crystal to become available, but at a cost. This one uses Cesium Iodide, also thallium activated. It does not share the same moisture issues as its sodium iodide cousins, and to add to its efficiency, it is also denser.

Therefore a really good scan can be made with a 1 mm thick CsI(Tl) scintillator sensor so long as the "window" allows the photons to enter the sensitive volume.

A really recent example of this technology was created by S E International, in the embodiment of their RAP-47 LEG Probe. The RAP-47 uses a select grade PMT, a 1'' X 1mm CsI(Tl) crystal and a thin aluminum window. At less than \$1000, these are a bargain, but check eBay for them at less than half that. Other suitable LEG probes are made by Ludlum Measurements and Technical Associates, but none of those use CsI(Tl) yet.

FIG.2>Rap-47 1" Diameter Low Energy Probe: FIG.3>RAP-47 1" thin aluminum window and protective cap:



THE RAPCAP APPARATUS

The RAPCAP is a home built XRF exciter made to clip on to the front of a RAP-47 or other LEG probe. Radioactive sources removed from up to 8 \$5 ionization type smoke detectors provide the alpha particles and low energy X-Rays. These are arranged so they point away from and perpendicular to the face of the sensor, and have a small layer of lead shielding immediately behind them to avoid direct rays for the source hitting the probe window.

This first version shown uses 8 such sources, mounted on a brass washer with 1/8" blind holes drilled around the periphery to accept the sources, to completely surround the target as it is placed immediately in front of the RAPCAP, with a space of 1/4" allowed between the sources and target. This small space is required so the excitation rays and particles have enough room to fully illuminate the target. Subsequent XRF rays FROM the target are collimated through a hole in the center of the RAPCAP and back to the sensor window. This "flat" version is best for flat targets, which need be no larger than 1" X 1".

FIG.4> RAPCAP MKI:



RAPCAP MKII

For tiny specimens, a second version of the adaptor head was developed, constructed and deemed successful. It has the excitation sources positioned at 22 1/2 degrees from perpendicular, thereby focusing the excitation onto a spot, just in front of the collimation hole. This version can analyze sub-gram sized samples, held in place by a fixture

(tweezers). Over a few week period of time, I have used the RAPCAP to analyze all applicable elements on the Periodic Chart from Calcium through Bismuth. Since a few elements within that range are naturally radioactive, these have been analyzed by the probe but without excitation (self-excited) - see notes.





RAPCAP STEP BY STEP CONSTRUCTION

Step 1) Have your mentor purchase several ionization smoke detectors and remove the sources, using approved safety methods. As few as 1 will work, no more than 8 are ever needed.

Step 2) Prepare a washer like base from brass, aluminum or other metal that is easy to work. If using a large brass washer, drill up to 8 evenly spaced 1/8" blind holes surrounding the central hole. The sources have a protrusion on the back that will fit perfectly into these holes. Secure them with epoxy, and then place an undrilled similar washer on the back to further shield the sensor. If using a machined brass adaptor, always place an extra lead shielding washer behind it. Hole size can vary, from 1/8" and upwards. The most amount of back shielding plus the least amount of radiation provide the cleanest peaks.

FIG.6>Holes in 1/8" lead shielding washers: Back thru hole is tapered to allow beam to spread:



Step 3) Attach the now modified probe to the classroom MCA* (set to read from 2 through 50 keV), and begin sampling elements!

I will be available for questions from teachers and mentors, and over the next few months will go through the Periodic Table of the Elements with you, one at a time. Some pretty cool elements can be found in everyday household items.

FIG.7> Finished Stable Element Analyzer Sensor- FIG.8> Analyzer port with radfilm covering:

ELMO= Elements Missouri:



FIG.9> Ready to analyzer silver content in a silver mica capacitor:



FIG.10> A typical display showing the presence of different elements:



FIG.11> Tin and lead components of 60/40 solder:





FIG.12> A red rock from New Mexico. Can it be iron ore?

<u>Testing for Mohs Hardness with points and</u> <u>Color Streak tests are inconclusive</u>



FIG.12A> APXS/XRF definitely show red rock does test positive for iron!



Lessons learned:

Short version:

Scintillation crystal probe do a fine job in this service, allowing identification of titanium and heavier when the probe is optimized.

No more than 8 uCi of source activity is needed. It is better to have 8 small sources surrounding the surface of the target than a single large source. As little as 1 uCi will work, it just takes longer.

Sources at 90 degrees to the surface give the highest results in terms of characteristic X-Rays but at a price, that being backscatter interference by the exciting energy.

Any extra energy hitting the probe other than the characteristic X-Ray will make identifying the element harder. i.e. pure elements analyze easier than alloys.

An attack angle of source to target of 45 degrees give good, clean usable results.

At least three factors determine the lower energy perceived by the probe.

1: Crystal characteristics- a cleaved crystal will detect smaller energies than a cut crystal.

2. Crystal housing window. Be is best, but the crystal still determines the ultimate low energy, even if NO WINDOW is used.

3. System, noise/PMT noise. This limits my system to 2 keV and above. Other systems will be different, perhaps vastly different.

HINT: Next time a battery goes dead in a device of yours, keep it because we will dissect it later for elements!!

*- If your classroom does not own an MCA, don't worry, we will be showing in weeks to come how to use a freeware program on an ordinary computer with a soundcard to fulfill all the functions of an expensive MCA. George Dowell

Chapter 3

THE CATALOG OF STABLE ELEMENTS ANALZED

This technique works from Ca to Bi, excells from Ti to Bi

The following catalog is set up so that each page is an element, and the page number corresponds to that elements Atomic Number (Z)

19 Potassium K

Ka=3.31 Kb=3.59



KI Tablets- Potassium Iodide



20 Calcium Ca

Ka=3.69 Kb=4.01





21 Scandium Sc

Ka=4.09 Kb=4.46





22 Titanium **Ti**

Ka=4.51 Kb=4.93





23 Vanadium V

Ka=4.95 Kb=5.43





24 Chromium Cr

Ka=5.41 Kb=5.95



25 Manganese Mn

Ka=5.90 Kb=6.49



MnO Paste with a little Zinc, from a D cell battery



26 Iron Fe

Ka=6.40 Kb= 7.06



27 Cobalt CO

Ka=6.93 Kb=7.65





28 Nickel Ni

Ka=7.48 Kb=8.26





29 Copper Cu

Ka=8.05 Kb=8.90





<u>30 Zinc Zn</u>

Ka=8.64 Kb=9.57



Zinc can, from a carbon-zinc D cell battery



31 Gallium Ga

Ka=9.25 Kb=10.26





<u>32 Germanium Ge</u>

Ka=9.89 Kb=10.98



High purity germanium detectors are used in many radioactive materials labs



33 Arsenic AS

Ka=10.54 Kb=11.73



Vintage rat poison, sealed in original container. XRF through the paper container.



<u>34 Selenium Se</u>

Ka=11.22 Kb=12.50





35 Bromine Br

Ka=11.92 Kb=13.29



Bromine is used in Printed Circuit Board material as a flame retardant- also used in child's pajamas.



36 Krypton Kr

Ka=12.65 Kb=14.11



A Krypton Gas proportional detector tube, with Beryllium window.


37 Rubidium **Rb**

Ka=13.39 Kb=14.98





<u>38 Strontium Sr</u>

Ka= 14.16 Kb=15.83



A Strontium Ferrite fridge magnet



<u>39 Yttrium Y</u>

Ka=14.96 Kb=16.79



A YAP Crystal (Yttrium Aluminum Perovskite activated by Ce3+)

used as a sensor in a scanning electron microscope.



40 Zirconium Zr

Ka=15.77 Kb=17.67



NPP Fuel Rod Cladding Tube- (Zirc4)



41 Niobium Nb

Ka=16.61 Kb=18.62





42 Molybdenum MO

Ka=17.48 Kb=19.61





43 Technetium Tc

Ka=18.41 Kb=20.59



Technetium



44 Ruthenium Ru

Ka=19.28 Kb=21.66



45 Rhodium Rh

Ka=20.21 Kb=22.72



Precipitate from a Rhodium plating solution



46 Palladium Pd

Ka=21.18 Kb=23.82





47 Silver Ag

Ka=22.16 Kb=24.94





48 Cadmium Cd

Ka=23.17 Kb=26.09





<u>49 Indium In</u>

Ka=24.21 Kb=27.27





<u>50 Tin Sn</u>

Ka=25.27 Kb=28.48





51 Antimony Sb

Ka-26.36 Kb=29.72





TOXIC SUBSTANCE-DO NOT HANDLE!

52Tellurium Te

Ka=27.47 Kb=30.99





53 Iodine I

Ka=28.61 Kb=32.29





54 Xenon Xe

Ka=29.80 Kb=33.64



A sealed Xenon gas proportional detector tube. XRF taken through its Beryllium window.



55 Cesium CS

Ka=30.97 Kb=34.98



Cesium Iodide crystal detector, XRF through its aluminum window.



56 Barium Ba

Ka=32.19 Kb=36.38



A Barium Fluoride crystal. Used as a scintillation sensor in PET Scanners in hospitals. Fluorine is invisible to our XRF sensor.



57 Lanthanum La

Ka=33.44 Kb=37.80





58 Cerium Ce

Ka=34.72 Kb=39.26





•

59 Praseodymium Pr

Ka=36.02 Kb=40.75





60 Neodymium Nd

Ka=37.36 Kb=42.27



A DEMAGNETIZED Neodymium magnet.

All magnetic materials must be kept away from the ELMO's sensor.



61 Promethium Pm

All Promethium isotopes are radioactive

Ka=38.65 Kb=43.95 La=5.43 Lb=5.96

Promethium Via K Line energies



A Prometium Needle from a Beta Thickness Gauge

Promethium via L Line energies



62 Samarium Sm

Ka=40.12 Kb=45.40





63 Europium Eu

Ka=41.53 Kb=47.03





64 Gadolinium Gd







<u>65 Terbium Tb</u>

Ka=44.47 Kb=50.39





66 Dysprosium Dy

Ka=45.90 Kb=52.18





67 Holmium HO

Ka=47.53 Kb=53.93





<u>68 Erbium Er</u>

Ka=49.10 Kb=55.69





<u>69 Thulium Tm</u>

Ka=50.73 Kb=57.58





70 Ytterbium Yb

Ka=52.36 Kb=59.35





71 Lutetium Lu

La=7.65 Lb=8.71

Analyzed via the L Line energies



eea * Towerse * Analyzed via the L Line energies*



72 Hafnium Hf

La=7.90 Lb=9.02

Analyzed via the L Line energies



Analyzed via the L Line energies


73 Tantalum Ta

La=8.15 Lb=9.34

Analyzed via L Line energies



A slug from a Tantalum capacitor

Analyzed via L Line energies



74 Tungsten W

La=8.40 Lb=9.67

Analyzed via L Line energies



*Analyzed via L Line energies



75 Rhenium Re

Ka=61.13 Kb=69.30

La =8.65 Lb=10.01

Analyzed via L Line energies



Analyzed via L Line energies

Rhenium dust.



76 Osmium Os

La=8.91 Lb=10.35

Analyzed via L Line energies



Analyzed via L Line energies



77 Iridium

La=9.19 Lb=10.71

Analyzed via L Line energies



Analyzed via L Line energies

Iridium heater wire from an Ion Gauge



78 Platinum Pt

La=9.44 Lb=11.07

Analyzed via L Line energies



Analyzed via L Line energies

Super fine Platinum wire from loop a hemispherical gas flow radiation detector. It's almost invisible.



$_{\underline{79} \; Gold} \, Au$

La=9.71 Lb=11.44

Analyzed via L Line energies



Analyzed via L Line energies

Yellow Gold tie pin.



80 Mercury Hg

La=9.99 Lb=11.82

Analyzed via L Line energies



81 Thallium Tl

La=10.27 Lb=12.21

TOXIC SUBSTANCE- Do not handle.

Analyzed via L Line energies



Sodium Iodide Thallium Doped Crystal sealed behind beryllium window



82 Lead Pb

La=10.55 Lb=12.61

Analyzed via L Line energies



Analyzed via L Line energies



83 Bismuth **Bi**

La=10.84 Lb=13.02

Analyzed via L Line energies



Analyzed via L Line energies



Chapter 4

Analyzing Radioactive Elements by Self-Excitation of XRF of their Daughter

A technique of using a radioactive isotope's own internally generated energies due to radioactive decay to excite XRF of the daughter element. Analysis is similar to APXS/XRF with the exception of the RAPCAP device being removed.

Then analysis is carried on as usual with the Low Energy Gamma scintillation probe.

Thorium

Large piece of Thorium Metal (RED)

XRF is self-excited as Thorium is radioactive.

Also shows Radon peaks (BLUE)



All via L lines

Part 5

Some practical things to do with APXS/XRF

20 gram MWA 869 stony meteorite. Low Iron and Titanium content. Barely magnetic.



Fe and Ti easily identified via their K lines



Solder-

60% Tin 40% Lead



Т

Lead on left via L Lines, Tin on right via K Lines.



Ore, Rocks and Minerals



The red rock contains iron!



Just a few of the Destructive Testing Methods normally used in geological classification:



Mineral sample marketed as "Smithsonite". Real Smithsonite will contain Zinc



High Zinc signature, unquestionably Smithsonite.



Testing a volcanic rock specimen.



Rock marketed as "fool's gold"



Mounted and testing:



Who's the Fool now?....or:"Yesterday I couldn't even SPELL Ytterbium!"

- > A sample from my late mother's collection, sold to her, marked as Fool's
- > Gold. I noticed none of the cubic crystal structure one would expect from a pure
- > sample. Under XRF, Iron- yes, but also rare earths.



Analyze and Identify Complex Metal Alloys

Fridge Magnets

May contain Barium Strontium, Neobdynum, Iron,



This one (above) contains Barium and Iron



Submitted to the Home Lab as a "Mystery Metal" test sample, this alloys was analyzed to contain Molybdenum, Rhenium and Tianium. After submitting our amateur analysis, the donor confirmed it to be exotic and rare MoReTi!!



(600 V DC Power Plant for Streetcars)



APXS/XRF proves it has a Silver coating over Copper.

The Alpha particle bombardment identified the surface Silver, while the X-Rays peered deeper inside to analyze the copper.

Modern US pennies show Copper over Zinc.

Iron-Nickel Meteorite. Beautiful Widmanstätten patterns.



Calibrating the system with pure spectrographic grade Iron and Nickel Standards



Testing Lab Apparatus (a.k.a. Treasure Hunting!)

Open style ion type vacuum gauge



BGO Crystals (Hospital PET-CT Scanner sensors)



Electronic vacuum tubes are sometimes made with exotic elements. Always save and disect an old tube.

<u>Caution: some components may be toxic, only disect tubes under the supervision of your mentor or teacher.</u>



Another ion vacuum gauge, this one a sealed, external, plug in type.





Testing a digital camera TFT LCD display for lead and rare earths.



Testing a CRT for rare earths.



Testing Paint



Modern Paint Tests POSITIVE for Titanium Pigment, NEGATIVE for Lead pigment



Vintage paint can contain Lead, Cadmium (yellow), Mercury (green), Selenium and other possibly toxic chemicals.



Dishes, Glassware, Household Items

Is it Lead Crystal or Common Flint Glass?



This stemware tests positive for Lead, proving it is real lead crystal



Ceramic dishware has shown an astonishing variety of exotic elements, both stable and radioactive.



Testing TOYS for Lead paint.



<u>NOTE: This is an educational lab demonstration exercise- don't use Home Lab equipment for Human</u> <u>Health certification!</u> Batteries.

This one contains Manganese, Zinc


This battery tested not so user friendly-

Mercury!

Tested right through the sealed container.





But how much Mercury?



At LEAST a gram's worth of Mercury in one small battery!







Chapter 6 DIY Apparatus Projects

Try military surplus plutonium probes for APXS/XRF

Radiac sets (military Geiger Counters) often include interchangeable heads, for different types of radiation.

Often an alpha scintillator probe, an X-Ray Scintillation probe and a GM probe make up a complete "KIT".

It is that X-Ray Scintillation probe that we are interested in. They come in many different flavors depending on the era and country of origin.

Any of the thin crystals as used in military "plutonium probes" will work. Be sure to use them behind a very thin window to block the light but pass the low energy X-Rays. Aluminum foil is OK for this, or any very thin metal, lake even from a Pepsi can.

The crystals are usually made of 1 or 2 mm thick sodium iodide, CaF2, and many other crystal structures depends on the model and country of origin. One of the best for our hobby use is the DT-590A from the PDR-56 Radiac set:

This model comes with a CaF2 crystal of thin proportions, specifically designed to detect the 14-17 keV X-Rays from Plutonium.

While the CaF2 can be used directly for XRF, I have used the '590A as a test bed for many different types of crystals. The best one found for XRF work to date is a small, thin, square Sodium Iodide in a little 1" diameter capsule, removed from yet another vintage Radiac. To the best of my measuring ability (it's sealed of course) it is about 5/8" square and about 4mm thick. It has a beryllium entrance window and a glass face for the PMT end. The thicker than normal (most RADIACs and commercial LEG's use 1 mm thickness) to my eyes gives much better shaped peaks and therefore makes identification much easier.

In addition to the electronic mods to the DT-590A, today I improved the entrance window on the housing, based upon recent experiences. This is the part of the housing on the front that already has a light tight, thin black light blocking window, covered with a screen. Not to be confused with the beryllium window on the actual crystal's capsule.

A thin Beryllium disc would be the absolute best for light blocking and low energy passage, but there are some logistics problems with Beryllium. I used aluminum foil. Two layers, and it does a great job of light blocking while passing even lower energies that the original window material.

Attached is a 60 second scan of Zirconium metal, a really thin sheet the size of a postage stamp, mounted on the 8 pellet RAPCAP, placed on the '590's front screen.



Not bad for a hacked, homemade probe!

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Converting the DT-590A surplus scintillator to a Universal Probe

With emphasis on APXS/XRF applications.

The DT-590 Plutonium Probe is part of a military PDR-56F RADIAC set and is available as a separate part from time to time, check your favorite military surplus supplier.

The scintillator crystal that comes in the '590A is a very thin one, made for low energy X-Rays only, as from Pu-239. Since we don't have a lot of need for a Plutonium detector in these parts, it was decided to repurpose this sturdily made probe into a more or less universal detector of either low, medium and high Gamma energy ranges, depending on the sensor crystal installed.

As the project developed, it became obvious that this probe case and PMT could be used with a multitude of different crystals, for testing of those crystal and actual radiation detection use.

As mentioned, the original crystal is only 1/8" thick at most, and is the one of choice for our APXS/XRF experiments.

For other experiments where medium and high Gamma energies are encountered, there is room for at least a 2" long crystal if the small un-needed circuit board is first removed from the housing. Rewiring the base to a standard 950+ single coax feed like we use on all our probes was easy, and a BNC was added to the rear cap, once the handle fitting there was removed on the lathe (a hacksaw will suffice). Removing the original RADIAC electrical connections left a hole on the side, which was covered with a small metal plate attached with black epoxy. Be sure to use a light tight cover here (radfilm is ideal).

Inside the original probe is a circuit board which contains preamplification and energy discrimination functions that we do not need.

By removing the circuit board, space is made for different thickness crystals with the original total overall length retained by the use of blank spacer plates in its place.

Also on the lathe we turned the square block of plastic scintillator into a cylindrical shape, being careful not to scar up the polished ends. Turning the scintillator plastic on a lathe is very similar to any Plexiglas type product, you just have to use a sharp tool, and keep the speed low so friction doesn't melt the plastic and mess up your work.

Once the final shape was achieved, the thing went together just as if it had been designed that way from the beginning.

The front cap already had a metal screen "window" so I decided to add a nice Zinc Sulfide alpha screen at the very front of the plastic, hoping that the light generated by it would go through the plastic Gamma scintillator as if it were a light pipe. Works just fine. A thin Mylar light shield is installed just behind the metal screen. This crystal now provides alpha-beta-gamma-X-Ray detection like the expensive ones being sold on ePay, at less than \$100 USD investment.

I might add that this article was first published 8 or 10 years ago, being the very first "Universal Probe" anywhere.

Basically I wanted to report the project to stimulate ideas for others who may have one of these "X-Ray Probes".

Not only is it an excellent working probe, it makes a great test bed for experimental work with various crystals.

Since the original project, all sorts of crystals have been used in this rig including the super dense BGO and BaF2.





The original thin crystal that comes with the DT-590A. It is perfect for APXS/XRF and other low energy Gamma and X-Ray work.



2 layers of light blocking but very thin aluminized Mylar Radfilm covering/.



Original rubber gasket:



Rubber gasket fitted, the assembly must be 100% light-tight. Note on the screened cover cap the thin lead washer shield with small entrance hole which we added for our APXS/XRF work:



Back of cover cap demonstrating the protective plastic ring, metal spacer ring, screen, and lead shielding washer placement:



The completed DT-590A conversion, optimized for low energy X-Ray, and especially APXS/XRF work:



One version of the Plutonium Detector Crystal, this one is a Beryllium windowed Sodium Iodide type. Most of the different types work in our application. The requirement is they be thin.

The Beryllium Window:

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	Legend							
[K AID	ha 1 K Beta 1							
E	lemental							
1 5	Symbol							
	Atomic						0.18 0.2	8
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Fe K							149 1.55	1.74
Fe L	CdL						AI	- Si
							13	14
4.51 4.93 4.95	5.43 5.41	5.95 5.90	10 000	/			Aluminum	Sili
Ti	VC	Mn	.49 6.40	07	.48 8.26 8.05	8.90 8.64	9.57 9.25 10	.26 9.89
22 2	3 24	25	re 26	Co	Ni	Cu Z	n Ga	
5 0.46 0.51	0.52 0.57 Chromiu	Manganese	Iron	Cobalt	28 Nickel	29 3 Copper 3		
17.67 16.61	18.62 17.48 1	9.61 18.41 20.5	0.70 0.72 09 19.28 21.66	0.77 0.79	0.85 0.87 0.	93 0.95 1.01	1.03 1.10	1.12 1.
Zr Nb	Mo	Tc	Ru	Dh	D.J	2.16 24.94 23.17	26.09 24.21	21.21
	42	43	44	45	Pa	Ag		n
2.12 2.17 2	26 2.29 2.4	Technetium	Ruthenium	Rhodium	Palladium	Silver	Cadmium	ndium
63.21 57.52 65	21 59.31 67.2	3 61.13 69.30	2.56 2.68	2.70 2.83	2.84 2.99	2.98 3.15 3.1	13 3.32 3.29	3.49
Ta	W	D	11.40	04.07 73.55	66.82 75.74	68.79 77.97 70	0.82 80.26 72.8	6 82.5
73	1 74	Re	Os	Ir	Pt	Au	Hg	TI
Tantakan	14	75	76	77	78	79	80	81
8.15 9.34	8.40 9.67	Rhenium	Osmium	Iridium	Platinum	Gold	Mercury	Thalliur
95.81 108.41	98.45 111.30	101.01 114.18	8.91 10.35 103.65 117.15	9.19 10.71	9.44 11.07	9.71 11.44	9.99 11.82	117.65
Pa	II	NI	111.15	100.00 120.16	109.10 123.24	111.90 120.401	114.15 120.04	
91	00	10 P	^u Pu	Am	Cm	Bk	CI	E
Protectinium	15mm	B-193 / 1.7	94	95	96	97	98	1 9
15.29 16.20 +	Cranium I	Neptunium	Plutonium	Americium	Curium	Berkelium	Californium	Eine

Si Al 14 13 Phe Silicon Aluminum 10.98 9.57 9.25 10.26 8.26 8.05 8.90 8.64 6.49 6.40 7.06 6.93 7.65 7.48 Ge Ga Ni Mn Fe Co Cu Zn 32 25 26 27 28 29 30 31 Manganese 0.64 Germanium Cobalt Nickel **Copper** 0.93 0.95 Zinc Gallium Iron 0.72 0.77 1.10 0.70 0.79 0.85 0.87 1.01 1.19 1.03 1 12 19.28 21.66 20.21 22.72 21.18 23.17 26.09 24.21 27.27 25.27 28.48 Rh Tc Ru S Cd In 43 44 45 50 49 8 Technetium Ruthenium Rhodium nium Indium Tin 2 56 2.68 2.70 2.83 3.49 3.44 3.66 3.29 3.32 69.30 62.99 71.40 64.87 73.55 82.56 74.96 84.9 80.26 72.86 Os Re Ir TI Pb g 75 76 77 81 82 80 Rhenium Osmium Iridium Thallium Lead Platin Mercury 9.19 10.55 8.91 10.35 9.44 10.27 9.99 11.82 12.21 10.71 11. 114.18 103.65 117.15 132.78 120.60 106.35 120.16 109.10 123.24 111.90 126.40 114.75 129.54 117.65 Pu Fn Cm Cf Es Am Bk 100 94 98 99 95 97 96 Ferm unium Plutonium Einstienium Americium Curium Californium Berkelium 21.17 16.38 14:28 18.28 16.02 18.83 14.62 19.97 15.66 20.56 14.96 19.39 15.31 45.40 53.93 49.10 43.95 40.12 52.18 47.53 41.53 47.03 42.98 48.72 44.47 50.39 45.99 Ho Sm Eu Gd Tb Dv 67 66

The Sodium Iodide Crystal. A little yellowing is acceptable.

The correct crystal will be thin:



References:

PMT Data Sheet:

http://www.qsl.net/k/k0ff/DT-590%20Probe%20Mods/R980_R980-01_TPMH1277E01.pdf

Original PC Card-

Layout:

http://www.qsl.net/k/k0ff/DT-590%20Probe%20Mods/TM-11-6665-245-34_30_1.jpg

Schematic:

http://www.qsl.net/k/k0ff/DT-590%20Probe%20Mods/TM-11-6665-245-34_32_1.jpg

Original Probe Mechanical Drawing:

http://www.qsl.net/k/k0ff/DT-590%20Probe%20Mods/TM-11-6665-245-34_30_1.jpg

Radiac Set AN/PDR 56F Manual:

http://www.qsl.net/k/k0ff/DT-590%20Probe%20Mods/an_pdr_56f_repair.pdf

Have Fun

Geo

Using the RAPCAP Apparatus on any Low Energy Gamma Spectrometer















As few as one source will give results.

The minimalist RAPCAP:

Wire frame, single source, lead slug behind as backshield, all in holding fixture.



Backshield detail:



Target metal disc set up on holding fixture, the mini RAPCAP is in the operating position:



Showing the jig in the testing position, waiting for target insertion:



Beautiiful Cadmium identification after only 60 seconds:



Ph testing some of the prepared samples since we had them handy.



Our motto: Test Everything!!

Cover art © 2012 by George Dowell



"Macrophotograph of RAPCAP on an X-Ray Image Intensifier"

Kr 36 Krypton 9 1.64 Xe 54 Xenon 1 4.42 Radon 1.72 14.32 33.64 He 2 Helium Ar 18 Argon Neon Neon Lr 103 Lawrencium Br 35 Bromine Lu 71 Lutecium 7.65 8.1 Fluorine At 85 Astatine 1.42 13. CI 17 Clorine 53 Iodine 3.94 Yb 70 Ytterbium 7.41 8.40 Selenium Tellurium Polonium 13.4 No Nobelium 3 16 Sulphur Oxygen Energies Tm 69 ⁸8.10 Phosphorus Sb 51 Antimony As 33 Arsenic Mendelevit Nitrogen 13 19 83 7.18 Erbium 58 Erbium 35 7.81 Femium 6 Carbon Silicon 32 Pb 82 Lead 11 P 6.95 0.55 Holmium 7.53 AI 13 Aluminum TI 81 Thallium Gallium Gallium 49 Indium H0 67 Boron 83 86 6.72 Dysprosium 6.50 7.25 Periodic Table and XRF Cd 48 Cadmium Californium Hg 80 Mercury 30 Zinc 8 <mark>C</mark> 0³ Terbium 6.98 Berkelium Cu 29 Copper 47 Silver Bk 97 11 65 79 Gold 6.28 Gadolinium 06 6.71 Pd 46 Palladium Cm Curtum Curtum Ptatinum Nickel Gd 64 8 Americium 4.62 18.8 Europium 5.85 6.4 C0 27 Cobalt Rhodium Am 95 Eu 63 11 77 Iridium Samarium 34 6.21 Ruthenium Plutonium Osmium Iron 26 Pu 94 Sm 62 5.64 1.1.18 193 17.7 ethium 5.96 Tc 43 Technetium Neptunium Rhenium Manganese 72 Mu Pm 61 33 43 odymium 5.72 Molybdenum Cr 24 Chromium 42 42 74 Tungsten 02 Uranium PN 09 M Neo 23 Cd K L Beta 1 K Beta 1 Elemental Symbol 23 Vanadium 41 Niobium 73 Tantalum Atomic Number Legend Elemental Name Pa 91 Pr 59 Prase Fe K K Alpha 1 L Alpha 1 Zirconlum Thorium Titanium HI 72 Hafihium Cerium Cerium 1.84 Actinium 12.65 15.71 La 57 Lanthanum 4.85 5.04 Lanthanide Series 57 - 71 Sc 21 Scandium 39 Yttrium Radium 12.34 15.23 Mg 12 Magnesium Sr 38 Strontium Be 4 Beryllium Ca 20 Calcium Ba 56 Barium Lanthanide Series Francium 12.03 14.77 1 Hydrogen 19 Potassium Rb 37 Rubidium Na 11 Sodium 55 Cestum Lithium

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All PHOTOS by the author unless noted otherwise



THE END