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Title: High Purity Germanium Detector Repair

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High Purity Germanium Detector Repair

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Abstract:

High purity germanium (HPGe) detectors are used to determine an objects output of gamma-ray radiation. This is done by counting charged particles released from the detectors inner materials when exposed to gamma-rays. As detectors age operational parameters change for the worse and detectors must be repaired. The damage that is received is due to both an accumulation of particulate matter and occasionally radiation damage (i.e. dislocation sites or trapping centers) to the germanium crystal. Liquid nitrogen operating temperatures expedites the collection of harmful particles. Crystal dislocations are created by the interaction between fast neutrons and the atoms of the crystal, these cause charge trapping, reduce amplitude of full-energy pulses and produce low-energy tails. All of which contribute to gamma-ray spectrum resolution degradation.

Under these conditions the spectrum that the instrument produces is of much lower quality resolution which may cause confusion during spectra analysis. For this project, known damaged detectors will be used to take a spectrum before undergoing the repair process, called detector annealing. After which a second spectra of the same source will be gathered in order to compare quality of resolution.

The procedure for detector annealing requires heating and a low pressure vacuum system. Application of high temperature dissociates particulate build up. The vacuum removes the particles and produces the necessary air free environment within the detector housing.

The annealing process is able to restore the detector to operate within tolerance. Results indicate that it will be beneficial to continue development and refinement of a detector repair station and procedure.

Introduction:

HPGe detectors are critically important to the nuclear industry. While this type of detector does have drawbacks, primarily related to the conditions needed to operate, they continue to be some of the most efficient and highest resolution detectors that are commercially available. Germanium detectors are capable of providing a great deal of information about the radiation source it is observing and are some of the few systems which can be used to reliably identify and quantify multiple isotopes within a single radiation source. This is very important for many health and safety reasons within the industry and critical to research and development so that tests are accurate and therefore accurate data can be collected. HPGe detectors ability to produce very high resolution spectra is also very important to the nuclear safeguards and nonproliferation fields. Allowing for very precise accounting of special nuclear materials, and nearly eliminates the possibility of using substitute materials in order to divert special nuclear materials.

Clearly, these devices have widespread and important use, and as such it is important that they are operating with the greatest possible accuracy. Therefore, a process for detector annealing is in development for the Los Alamos National Laboratory, Nuclear Process Infrastructure, such that technical equipment can be repaired on site. This will reduce cost for detector repair and eliminate shipping time as well as transport of equipment out of secure facilities, which may contain contamination and pose additional hazards. And ensure that equipment stays in operable conditions.

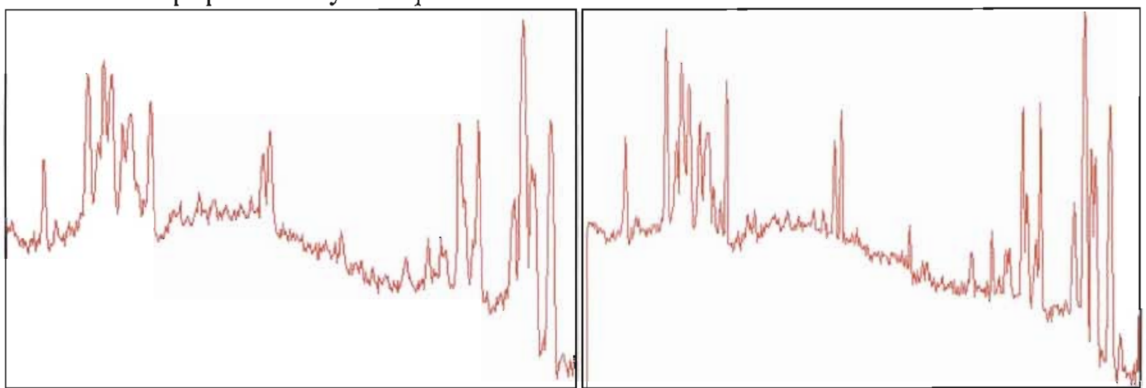


Figure 1.
X Axis: Energy level, 0 to 500 keV. Y Axis: Number of counts, 10 to 1100.
Poor and Good* resolution spectrum (histogram) of Plutonium, respectively, using HPGe detector. Wider peaks provide less information. Materials are recognized by what energy levels the primary peaks occur.
* The difference in resolution quality is not a result of the annealing process.

HPGe detectors operate utilizing an intrinsic semi conducting material, germanium. Large germanium crystals of incredibly high purity (~99.9999999999999%) are grown for this application; ideally the crystals are formed as a single grain and are not only pure germanium but also have a continuous crystalline structure void of dislocations and all other inconsistencies. As the detector, and therefore the semiconductor, are encountered by gamma rays energy is transmitted from photons into some bonded electrons which are then freed and move into the conduction band. A high voltage is applied across the crystal enabling current flow and collection of the free electrons. The electrons are subsequently counted in a preamplifier as very small electrical charges, which cause a voltage pulse in the system. This small pulse is proportional to the energy introduced by the incident gamma-ray; the electrical system of a detector amplifies and digitizes this charge, calculates the probable energy level of the incident ray or photon and appropriately places the data on a histogram, in order of increasing energy. As data is collected the histogram forms the energy spectrum of the tested material. An energy calibrated histogram can be used to identify nuclear materials using gamma-ray spectrometry.

Germanium detectors must be operated at liquid nitrogen temperatures (77K), because of the material properties of intrinsic semiconductors. That is, as a result of the relatively small energy step required to move valence electrons into the conduction band. Without the implementation of cryogenics this band gap can be overcome by atomic vibrations induced by thermal energy, this causes a leakage current and introduces invalid data to the system causing noise in the spectra and compromises resolution of the histogram. Additionally the cryogenic conditions allow a more absolute vacuum decreasing interference and improving resolution of the final spectrum by preventing conduction heating.

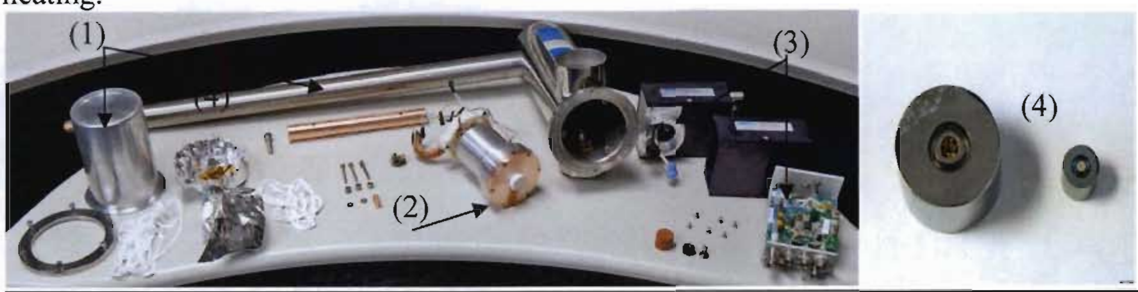


Figure 2.
(1) Cryostat components,
(2) Detector/ Crystal housing,
(3) Preamplifiers,
(4) Two Germanium crystals.

Vacuum System:

An essential part of this procedure is assembling an adequate vacuum system. A high (10^{-5} to 10^{-6} Torr) vacuum is required. To achieve this level of vacuum a great deal of specialized hardware and equipment is needed, including, multiple types of pumps, various fittings and valves and an accurate low pressure gauge. It is necessary to prevent additional contamination to the cryostat when connected to the vacuum system, this demands more specialized equipment, specifically mechanical pumping systems that do not utilize internal lubrication called dry pumps, and the introduction of pure nitrogen to ensure an inert environment. All materials were required to be nonporous, inert, clean and precisely manufactured and assembled.

The pumping system used is a Drytel 1025 by Adixen. The system is a single unit which houses two separate mechanical vacuum pumps, a roughing pump that removes the majority of present molecules via laminar flow, and a high vacuum pump which is able to remove remaining particles which move by less predictable molecular flow.
The roughing pump is a diaphragm pump, one type of dry pump. The roughing pump has a pump speed of approximately 1 cubic foot per minute, and once it has reached the lowest pressure it is capable of, the high vacuum pump is automatically activated. The high vacuum is achieved by a molecular drag pump with a pumping speed of 16 cubic feet per minute. At steady operating conditions the system has an effective pump speed of 9 cubic feet per minute. The Drytel 1025 system should be capable of reaching a 10^{-6} Torr vacuum if allowed sufficient time to pump down, estimated no more than 3 hours. This time increases greatly once a device is connected for repair and varies depending upon degree of damage.

Pressure measurements at very low pressures are difficult to accurately attain even with highly specialized equipment for two primary reasons. One, a single gauge has a limited range therefore multiple gauges are often required. Second, vacuum pressure gauges have a very high measurement uncertainty, commonly up to +/- 50%; however, this is primarily attributed to the characteristics of molecular flow than any component of the gauge itself.
The pressure gauge selected for this application is a Bayard- Alpert type gauge which measures pressure indirectly. A Bayard- Alpert gauge uses a cathode and anode to ionize, collect and count gaseous particles. Using statistics and the relations represented in the natural gas law pressure is approximated. This particular gauge is entirely self contained, with a digital pressure readout and no need to interface with any other systems, allowing it to stand alone for easy operation, the range of measurement is 10^{-3} to 10^{-9} Torr, which is sufficient for this application, although it cannot display pressure during the roughing pump process.

The final challenge for this particular system was incorporating a source for dry nitrogen. This element is required by the detector manufacturer and helps to ensure an inert environment within the vacuum system and detector. The system is to be purged with nitrogen as the vacuum system and detector are opened and joined. The heat source used for this system is electrical heating tape, operated by an Ortec temperature controller. The tape is applied around the exterior of the cryostat and a simple dial is used to adjust the temperature.

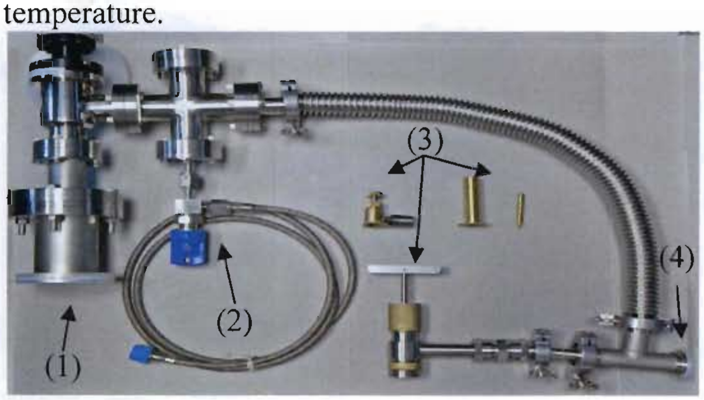


Figure 3.
Complete vacuum system.
(1) Inlet to vacuum pump,
(2) Nitrogen valve and hose,
(3) Three Ortec valve operators,
(4) Vacuum gauge connection.

Repair:

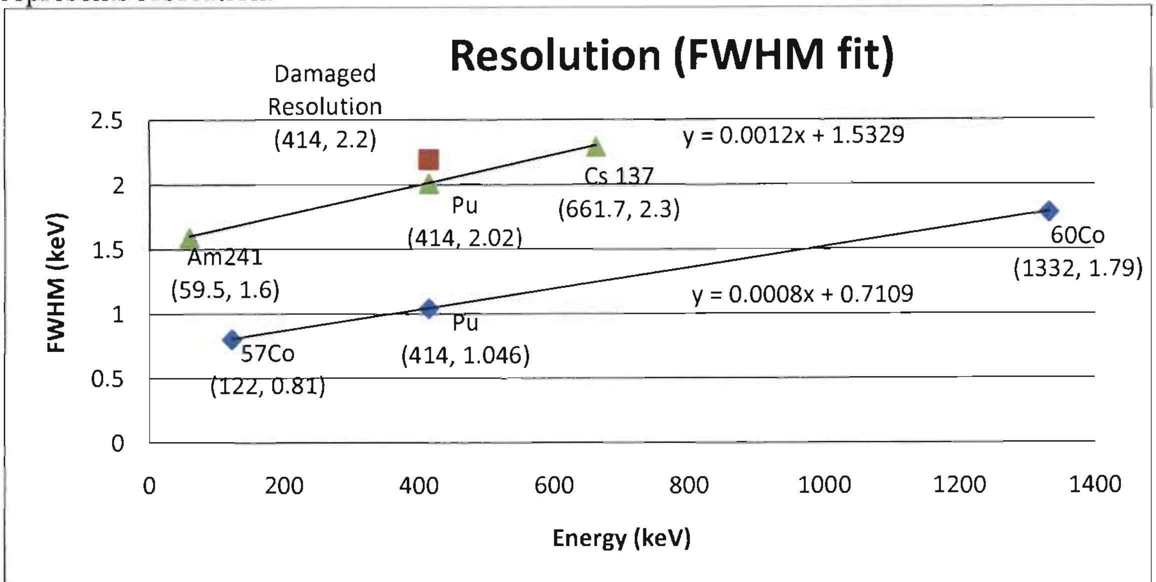
The repair process consists of three primary operations, the most fundamental and critical of which is known as detector annealing. This procedure requires the application of high heat and a low pressure vacuum system this will remove contamination from the detector and can also repair damage done to the crystalline structure of the germanium. Typical detector design incorporates a molecular sieve within the cryostat, the sieve coupled with liquid nitrogen temperatures of normal operation can effectively collect and limit various particles inside the cryostat which would otherwise contribute to thermal conductivity and spectral noise. With age and operation this sieve may become saturated and lose its capturing capabilities, potentially allowing the crystal to rise in temperature. Heating the cryostat will dissociate all particles from the sieve and other surfaces while the vacuum will simultaneously remove them. If necessary or desired, additional heating also repairs dislocation sites, minor faults in the crystalline lattice. Given the high purity of the crystals used and knowing that detectors are common in various radiation areas, most dislocation sites result from neutron damage. The greatest effect of a dislocation site in a detectors crystal is charge trapping. This occurs when an electron is released by gamma ray energy but the electron is not properly conducted across the crystal impurity and is counted as a lower energy gamma ray. This abnormality lowers the accuracy and detail of the detector measurements, similar to that seen in Figure 1. This relatively simple "pump and bake" procedure is able to restore a functional detector to warranty specifications.

Further repair is accomplished through electrical component replacement and complete reconditioning of the molecular sieve materials. Electrical repairs are severely limited due to the manufactures design of the electrical components, such as the preamp, which has inaccessible electrical components. However, the field-effect transistor (FET), which is a delicate electrical component on the detector used to shape and control electron (or hole) flow, it is this component which senses and outputs detector crystal signals and associated energies. With a malfunctioning FET the spectra produced will give no information, either as a straight horizontal line if the FET was shorted, or pure noise if the FET has become open. The preamp hybrid is the only other replaceable circuit component. The function of this component is to ensure the device is inoperable before reaching operating conditions, particularly crystal temperature. If this part fails it disables any use of the detector. All other electronic elements are sealed and must be serviced by the manufacturer or replaced.

As stated previously, the cryostat and detector contain molecular sieve materials which maintain the vacuum conditions by collecting contamination. Typically there are three separate elements to do this, a general gas absorbent, an oxygen absorbent and a hydrogen absorbent. Standard annealing partially restores the general sieve and will restore the detector, however the repair is slightly less effective and results may be short lived compared to a complete repair. Complete reconditioning is accomplished only by opening the cryostat and removing the sieves. The sieve material can be reconditioned or replaced. To recondition the general sieve it must be heated above 350 degrees Celsius in a nitrogen rich environment for a minimum of 24 hours and reinstalled. The oxygen getter is restored by a similar but more advanced process, requiring more precise control over the reconditioning atmosphere e.g. narrow temperature range, multiple nitrogen exchanges, a 4% hydrogen flow for 6 hours and cannot come in contact with atmosphere. Typically the hydrogen getter can be disregarded as there is little free hydrogen in the atmosphere, the standard annealing process is sufficient to remove contamination.

Results:

The detector to undergo the repair process is a Canberra model GR1518, the active volume of the crystal is approximately 68 cubic centimeters. The manufacturer calibrated this device using Cobalt isotopes ^{57}Co and ^{60}Co and warranties a resolution of 1.8 keV at those energy levels. This represents a resolution of less than 1.1 keV at the 414 keV peak of Plutonium. As shown below (Figure 4) by the linear interpolation using manufacturer specifications, and also the polynomial fit, that predicts a resolution near 0.95 keV at the same plutonium energy levels. This data was produced by a similar detector which was operating correctly. When the Canberra detector was operated it produced a peak with full width at half maximum (FWHM) of approximately 2.2 keV. FWHM is the value that represents resolution.



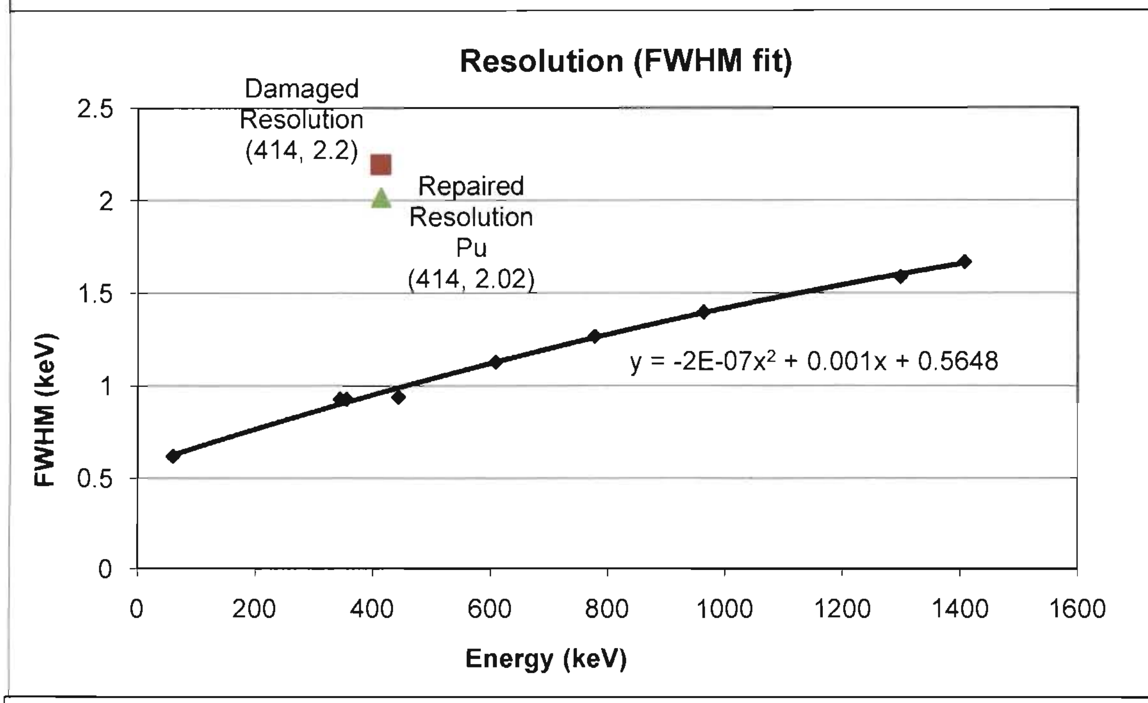


Figure 4.
Both plots show the original resolution data point, Damaged Resolution.
Top: Linear approximation of resolution for energy peak of 414 keV, manufacturers' data for specific detector (lower trend line).
Linear fit to data points acquired after repair (upper trend line).
Bottom: Polynomial fit to experimental data produced by fully functional detector, used to give second approximation of resolution for a peak occurring at 414 keV.

The device was selected for repair. Because the detector did produce the appropriate peaks but only at a lower than expected resolution it was assumed that all electrical components were operating correctly. The short time of use and operation also supported the decision to not perform a complete sieve reconditioning, as it would compromise the vacuum quality with little to no benefit. Neutron repair was also an unwarranted procedure. The detector required, and underwent, the pump and bake procedure. It is recommended the system be allowed to operate for several days to reach temperatures high enough to outgas all contaminants and allow the vacuum to stabilize. Due to the vacuum conditions the most time consuming portion of the procedure is assuring the internal parts of the system are adequately heated and cooled while connected to the pumping station. The heated vacuum system stabilized at pressures in the low 10^{-5} Torr range, after returning to room temperature the vacuum improved to 7.6×10^{-7} Torr, this is expected to improve by more than one order of magnitude when cryogenic conditions are established. After the pump and bake was performed the detector was used to collect data, the points produced are FWHM of 1.6 at 59.5 keV and 2.3 keV at 661.7keV, ^{241}Am and ^{137}Cs respectively, these points are also plotted above. Again these points were used to predict FWHM at 414 keV, FWHM was estimated to be 2.02 keV FWHM. This is still very poor resolution, the detector did show a noticeable improvement, 8.18%, but additional diagnosis and repair will be required, beginning with a second annealing.

Future work:

These results are unfortunate however there is still justification for continuation of the project. Future works will include the assembly of electronics testing equipment. Equipment will include a simple circuit with an ammeter to test the FET and all the components to run the detector, in sync with an oscilloscope. The most basic electronics station requires an FET tester, a high voltage supply, a signal amplifier and an oscilloscope. With these devices the type and severity of damage can be assessed and the proper repairs can be made. The final step for an HPGe repair station is the ability to perform complete sieve reconditioning. This requires the ability to reach very high temperatures (350 C), a glove box, and nitrogen which can be introduced to both the glove box and heated system in significant quantities. Commercial restoration of these detectors ranges from two thousand to nine thousand dollars and has a turnaround of weeks if not months if returned to original equipment manufacturer facilities and even independent repair centers. Development of an in-house repair station will allow a turnaround of approximately one week and requires a capitol investment comparable to 1 to 3 individual repairs, depending on the level of repair needed. Development of a precise procedure will allow duplication of a repair station within secure facilities at LANL. This should simplify the detector shipping process and shorten the turnaround. The completion and operation of the repair station described here will increase the efficiency of performing critical maintenance to HPGe detectors, as well as increasing detector accuracy and efficiency.

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