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# Retrieving Historical Electrorefining Data

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## Abstract

Pyrochemical Operations began at Los Alamos National Laboratory (LANL) during 1962 (1). Electrorefining (ER) has been implemented as a routine process since the 1980's. The process data that went through the ER operation was recorded but had never been logged in an online database. Without a database new staff members are hindered in their work by the lack of information. To combat the issue a database in Access was created to collect the historical data. The years from 2000 onward were entered and queries were created to analyze trends. These trends will aid engineering and operations staff to reach optimal performance for the startup of the new lines.

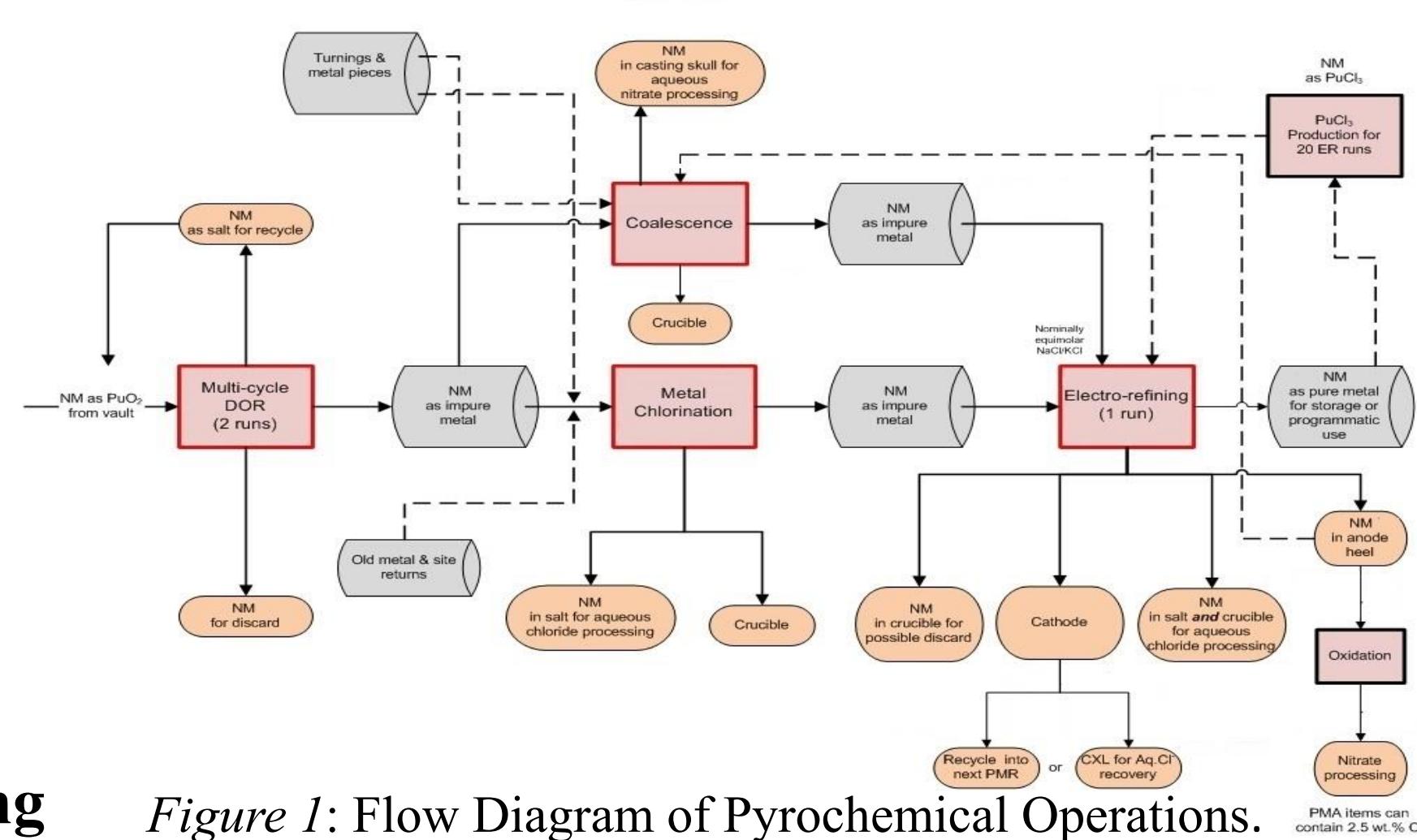
## Background Information

### Pyrochemical Operations

Pyrochemical operations from the 1950's-80's were composed of various operations that were evaluated for production of high purity plutonium metal. The flowsheet of the processes today is as shown below and contains the following operations:

- Multicycle Direct oxide reduction(MCDOR) : Converts  $\text{PuO}_2$  to impure Pu metal.
- Metal Chlorination: Removes  $\text{Am}^{241}$  from the impure metal produced by MCDOR.
- Coalescence: Recasts Pu metal at >98% purity to form a metal ingot that will be melted in the ER process.

The purpose of these operations is to produce a product that can be used as a feed item for ER to produce a Plutonium metal that is >99.9% purity. All of the processes above, including ER, have years of experimentation data along with handwritten data sheets. The data will be entered in an Access database for scientists, engineers, and operators to correlate input feed material characteristics to output quality. With this knowledge a high quality product can be made without having to repeat the experimentation that was already done. Since the flowsheet represents ER as the goal of the operations it was entered first.



Electrorefining *Figure 1: Flow Diagram of Pyrochemical Operations.*

The electrorefining process is mainly used in industry for the refinement of copper, nickel, lead, gold, and silver. After being evaluated through the 1950's it became the purification method for plutonium in the 1960's. Theoretical and experimental thermochemical calculations proved various impurities could be removed proving the ER process was the best for refinement. The metal is placed in a magnesium crucible and heated to above the impure metals melting point (3). To ensure that equilibrium conditions are met stirring of the molten anode and salt occurs. A small amount of  $\text{Pu}^{+3}$  ( $\text{PuCl}_3$ ) is initially added to the reaction so electrowinning of the salt will not occur. Once the  $\text{Pu}^{+3}$  is dissolved it travels through the salt, where it leaves most of the impurities, to the cathode where it is reduced to pure metal. The ring shape of the solidified pure plutonium ensures criticality safety.



Figure 3: Feed Metal before ER

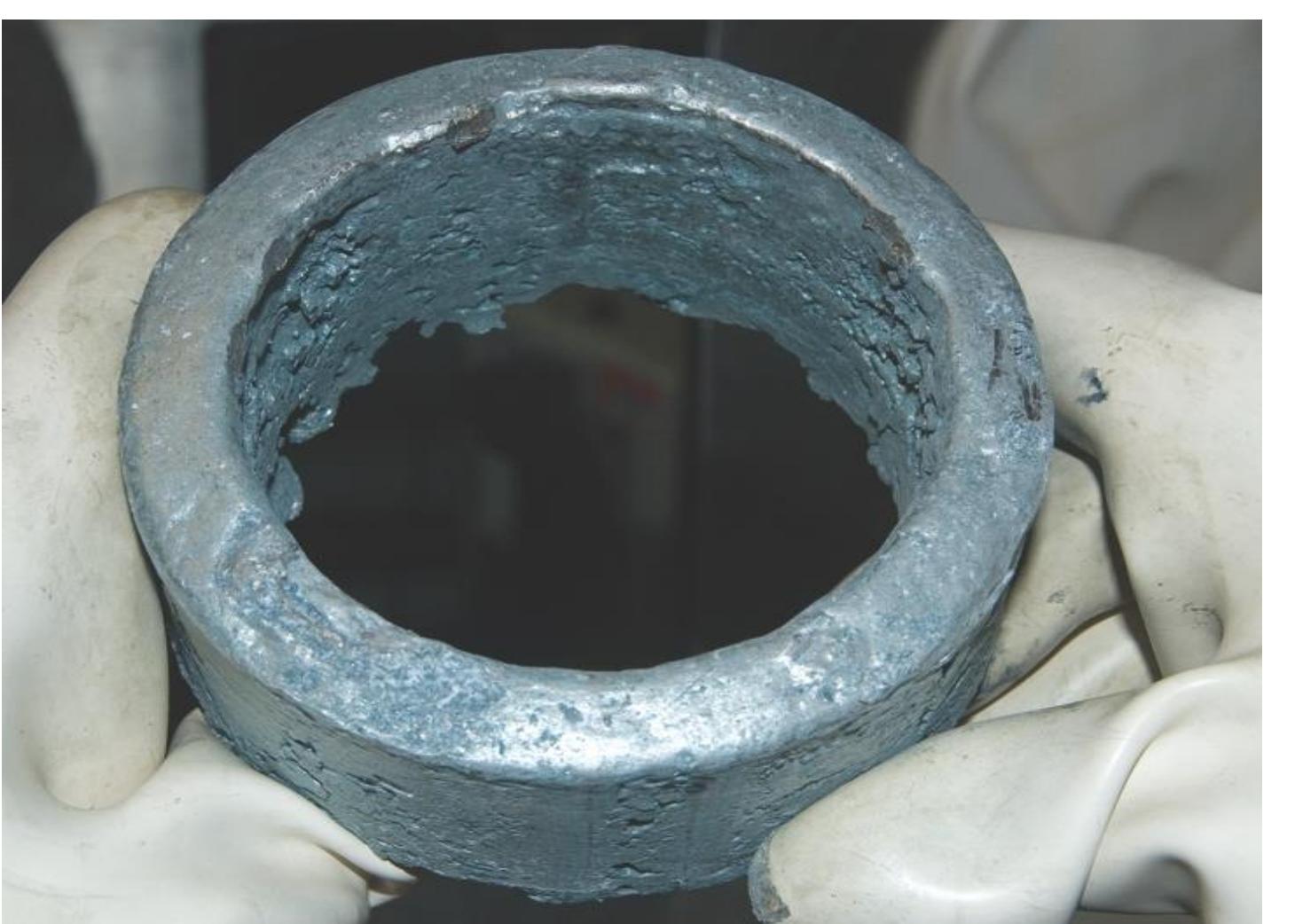


Figure 4: ER Product Metal, after ER.

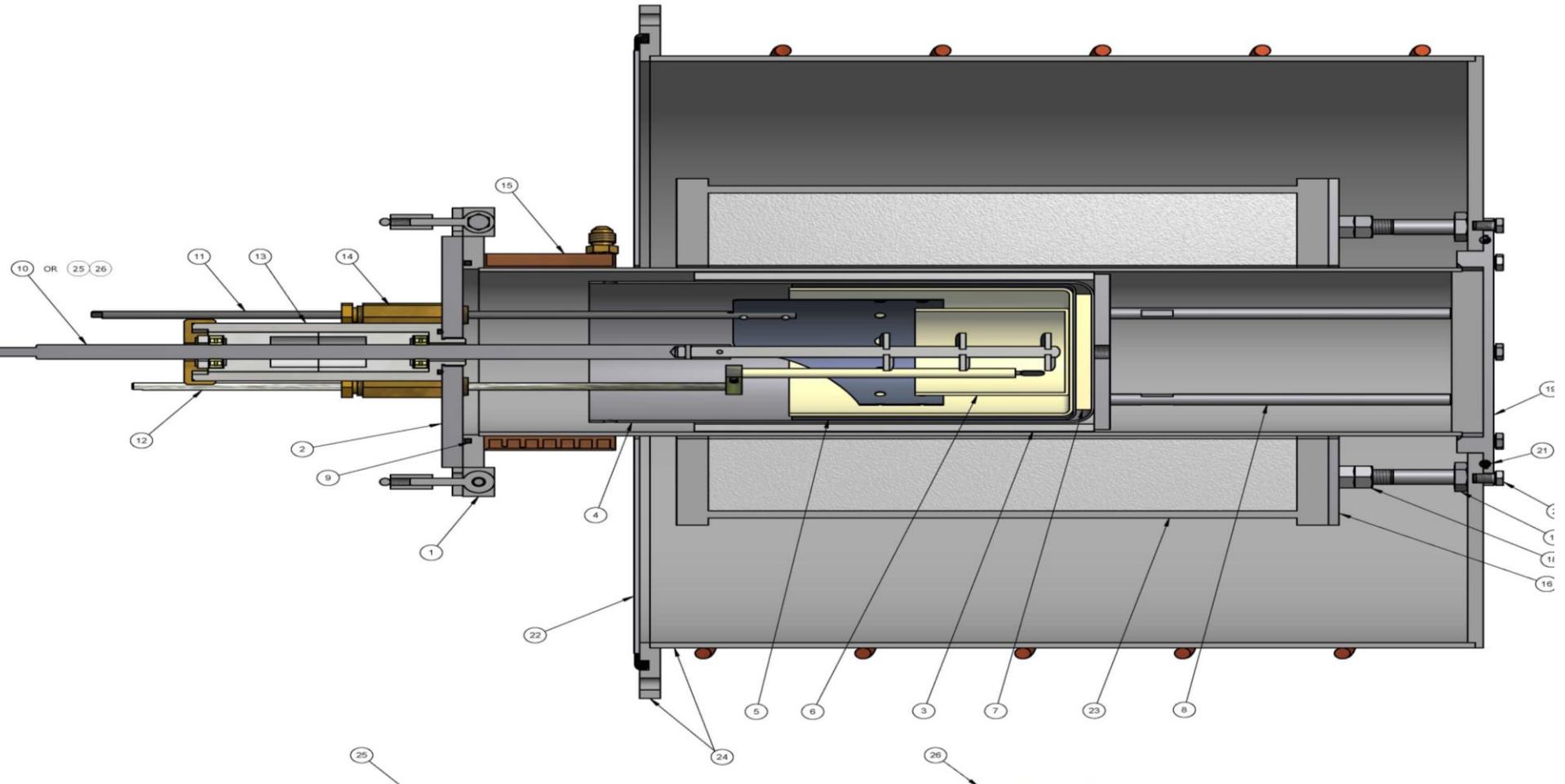


Figure 5: ER Furnace



Figure 6: ER Crucible, shows the inner section



Figure 7: ER (Tungsten) Cathode. Suspended above outer portion of crucible to allow



Figure 8: ER Anode is lowered into the inner portion making electrical contact with the impure metal.

## Purpose

With so many employee's retiring from LANL and other national laboratories across the country it is vital to capture their knowledge prior to their departure. Working with the ER data many of the people that worked to fine tune the process have left LANL so the comments they left are vital to the success of the continuation of the ER process. The adjustments made by the retirees are being discovered and will be put to use to minimize waste and make production easier to adjust.

## Methods

Microsoft Access 2013 was the database used to create the queries and store data for the ER runs performed. The queries were used to analyze various trends that were noticed throughout the entered data sheets. There were approximately 3267 data sheets in the Access database. Out of those 3267 sheets there were 342 sheets for the years 2000-2008. For the 342 sheets there were 10 that did not have PDF's associated with them. A run sheet without a PDF means that a run was done but there is no run sheet found for it yet.

## Results

The beginning phase of the ER data capture process gave over 300 manual entries of run sheets spanning from 2000-2012. Three queries were created from the entries. The queries are as follows:

1. Amount of feed into ER versus the amount of Pu recovered. This allowed for a calculation of product yield for specific dates.
2. Amount of feed into ER versus the amount of Pu recovered. With comments to identify the changes in product yield.
3. The number of times an anode rod was used.

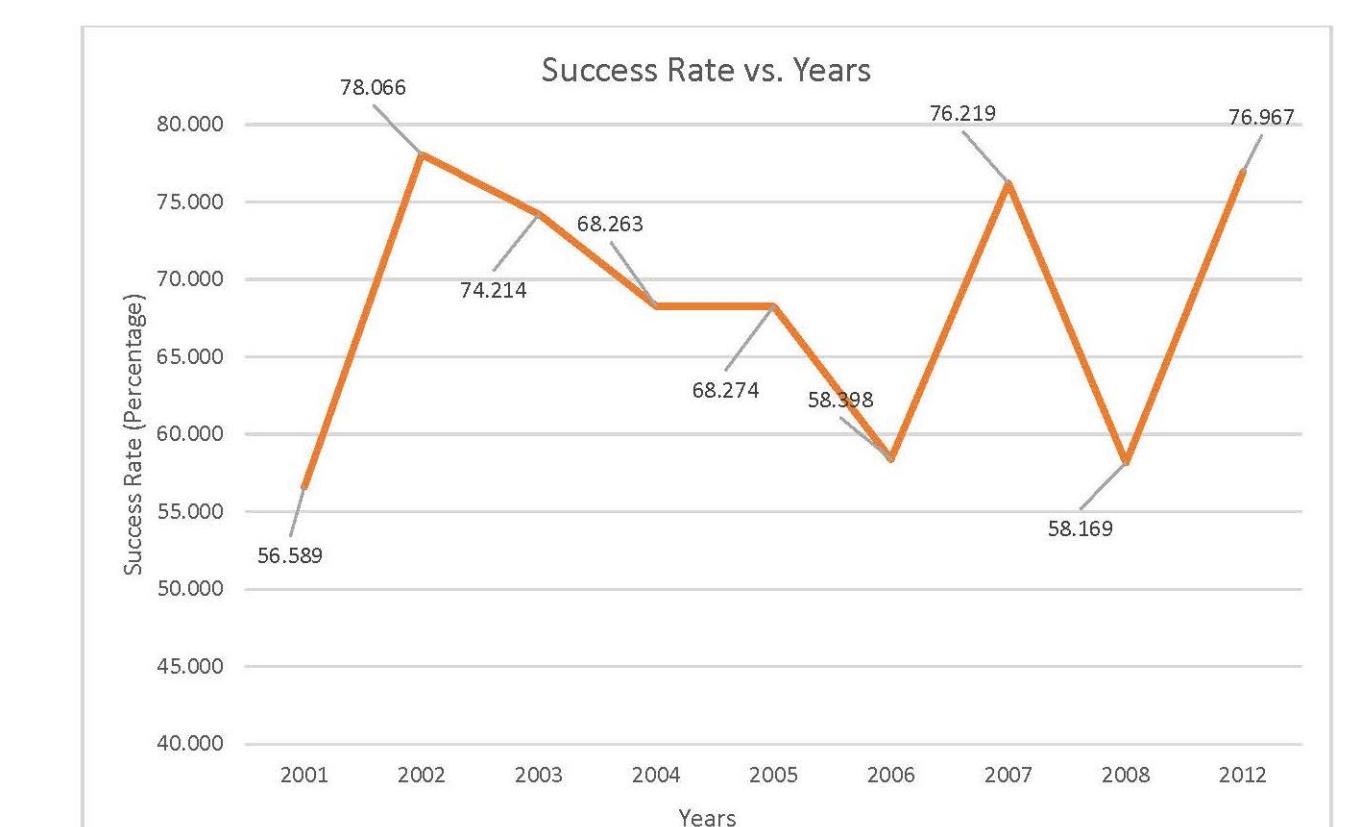


Figure 9: Graph of Success Rate vs. Years.

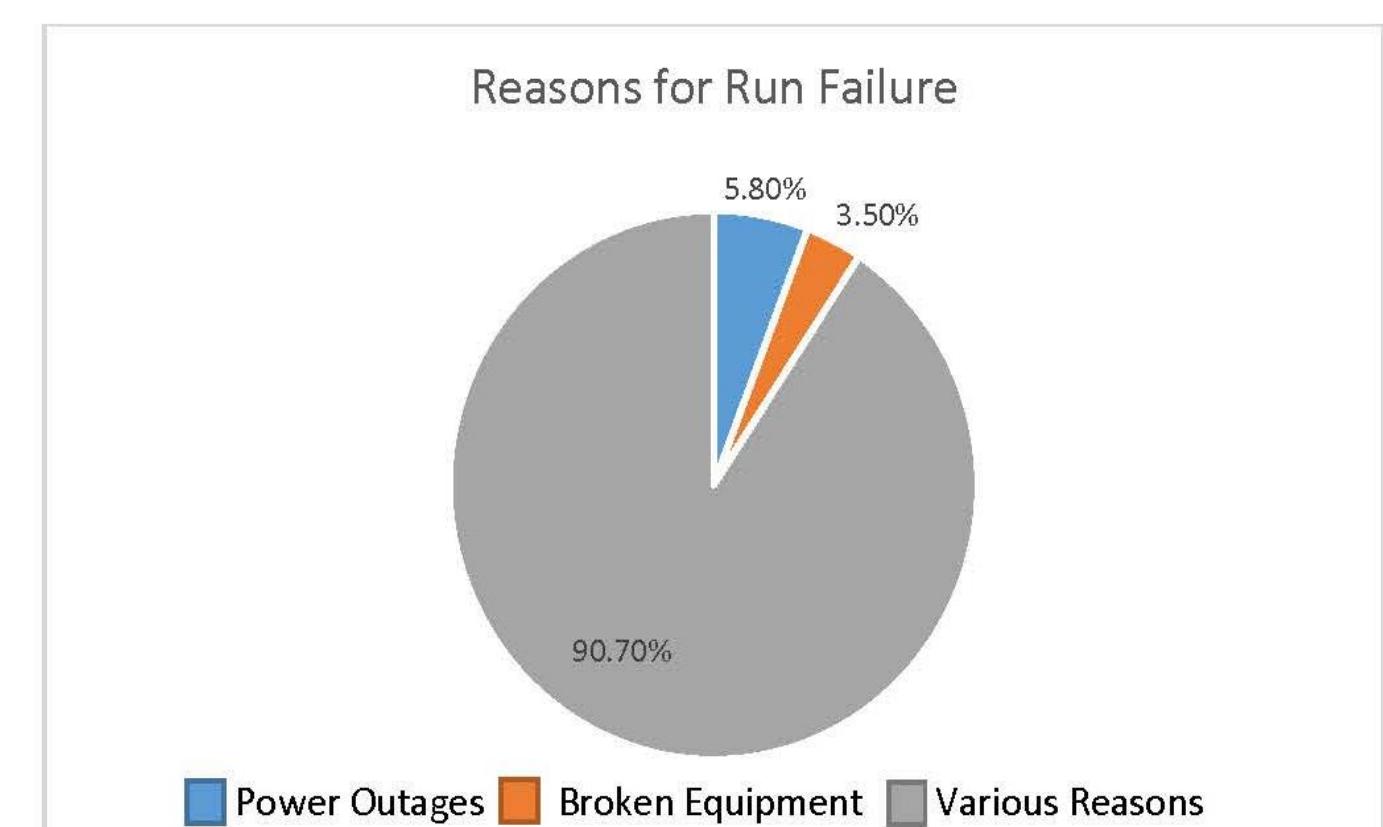


Figure 9: Cause of Failure Pie Chart.

Another query created was the number of times an anode was used vs. the date. This was done to get a rough average on how many times an anode was used and if that would factor into it's breaking during operation. After analyzing the data it was determined through the comments and graph below that anode usage did not factor into success rate.

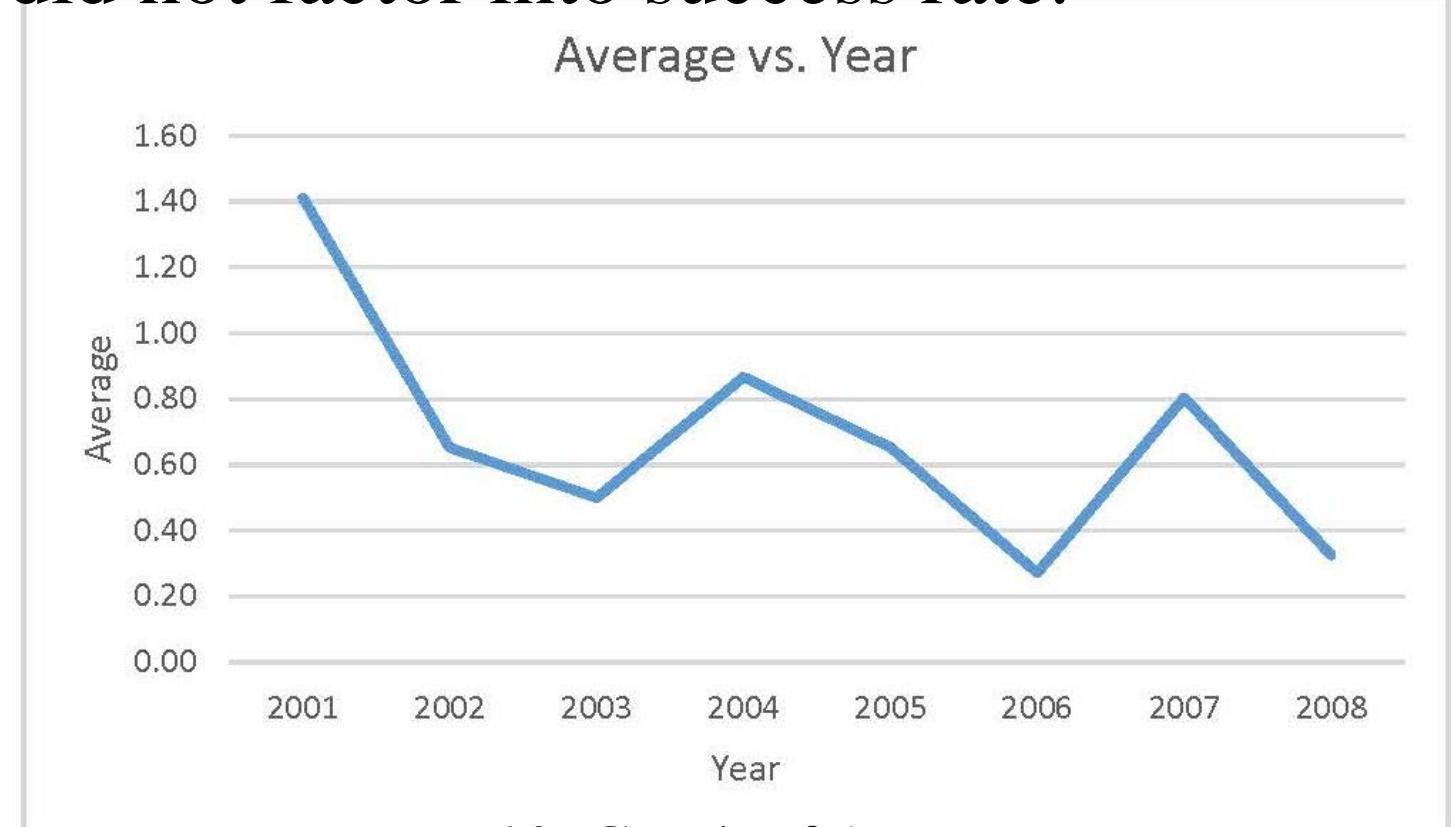


Figure 10: Graph of Average vs. Years.

## Conclusion

There were interesting aspects to this project some of which are analyzing legacy data to see what trends can be recognized, learning about the ER process, and experimenting with Access to analyze trends. Creating queries allowed for a better understanding of the successful and failed runs. This allows for engineers, chemists and technicians to use the traits of the good runs while avoiding failed run characteristics.

## Future Work

Due to the many processes that fall under pyrochemical operations there remains a large amount of run sheets waiting to be recorded. Currently run sheets from Molten Salt Extraction (MSE) are being scanned to be logged in Access in the future. Gaining a database collection of data from various processes will aid in a better understanding of the process to prevent past mistakes and eventually create an easier way to store and collect data.

## References

1. Christensen D.C., Mullins L.J. "Present Status of Plutonium Metal Production and Purification at Los Alamos-1982". U.S. Dept. of Commerce. 1981.
2. Morgan A.N., Mullins L.J. "A Review of Operating Experience at the Los Alamos Plutonium Electrorefining Facility 1963, 1977". U.S. Dept. of Commerce. 1981.
3. Seaborg Institute. "Actinide Research Quarterly 3rd quarter 2008". Los Alamos National Laboratory. 2008.