# Rapid Dissolution for Destructive Assay of Nuclear Melt Glass

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**Abstract**

This study evaluates four methods for dissolving complex glassy debris resulting from nuclear detonations. The samples of interest simulate the glassy debris generated from a nuclear detonation's fireball coming in contact with solid masses. Each method attempts to achieve dissolution through different approaches involving either acid digestion, alkaline digestion, or molten salt fusion. Two of the four methods were modified to retain all elements of the debris or surrogate debris. This retention is critical to the proportional relationships used in identifying fuel types and designs of nuclear weapons. Analysis is conducted with an inductively coupled time of flight mass spectrometer (ICP-TOF-MS) to provide exact elemental composition and yield for each dissolution method. The samples analyzed were trinitite (trin), surrogate trinitite formulation (STF), urban surrogate melt glass (NYC), and MAPEP MaS 32 (MAPEP). All samples have well known elemental compositions except for trinitite, however there are published compositional norms that are predicted for the trinitite. The four methods used were a Lithium Fusion (Larivière Method), a Sodium Hydroxide Fusion (Maxwell Method), an Acidic Digestion (Eppich Method), and a Modified Rapid Acidic Digestion (Auxier Method). Outcomes for the Lithium and the Maxwell Method failed to produce meaningful results due to the mass difference in fusion material compared to the isotopes of interest in the sample material mass. At the maximum concentration limit of 25-35 parts per thousand the mass spectrometer could not meaningfully detect the barium or uranium in any of the samples. The acidic digestion, and the Auxier Method both showed success with detecting appropriate levels of uranium, barium, and other lighter elements. The Auxier Method shows the best results when compared to ideal 100% yield from each sample. For Auxier's Method, uranium averages a yield of 5%±.02% of ideal. This is 614% above the acidic digestion and over 1200% above the other methods. For barium, the Auxier Method averages a yield of 9% of ideal. This is 595% above the acidic digestion and 4300% above the other methods. The Auxier Method demonstrates repeatability across three runs of each sample and sample independence as percent yields were similar across sample types.

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# 1: Introduction

A nuclear weapon detonates in the downtown of a country's capital. Domestic insurgents take claim for the attack and a well-known international terrorist organizations declare support for insurgent's righteous use of the nation's weapons against them. News reports 'leaked intelligence' information pointing to support from less than friend regional neighbors. If this event were to happen, one of the first and most pressing questions asked would be, "Where'd the nuclear weapon come from?" This project represents a small slice of the effort to provide a government and the world a legally defensible and scientific rigorous answer to that question.

For the United States of America, The Nuclear Forensics and Attribution Act (NFAA) of 2010 provides the initiative develop and refine a variety of capabilities to combat and deal with a nuclear event [1][2]. The legislation embodies this directive and stresses the technological readiness of the country for violent nuclear events. The legislation directs interagency cooperation and academic collaboration [1]. While very young in terms of the nuclear program, the field of Technical Nuclear Forensics (TNF) was established to enhance the response capabilities, the technology capabilities, and analytical methods for analyzing interdicted pre-detonated weapons or detonated debris. Attribution is the key goal of field and must be resilient to complex environmental factors at the site of detonation and must see through any counter forensic employed such as doping or coping of a countries development process. This project seeks to develop the analytical component of this effort and provide a robust standardize method for quick transiting radioactive nuclear debris from a solid to a liquid phase while preserving and not contaminating the isotopic composition of the sample.

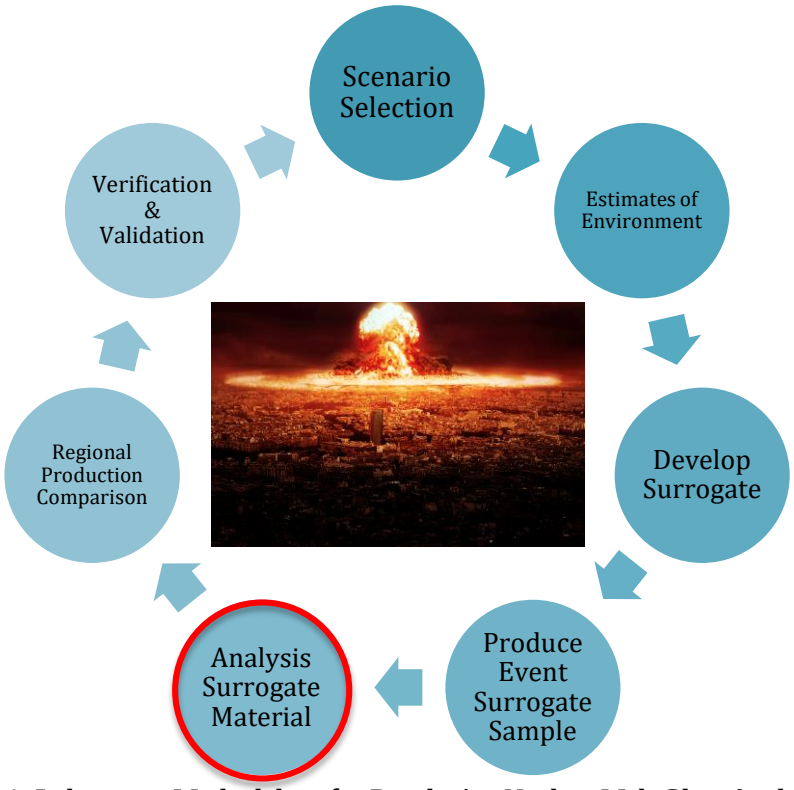



**Figure 1: Laboratory Methodology for Developing Nuclear Melt Glass Analysis Procedures.**

For this project Figure 1 shows the overarching laboratory model used and the specific area where this dissolution method applies. Figure 2 shows how the model will be applied in a real scenario. The processes must produce a result that is legally defensible and can provide a legitimate foundation for international response.

The subject of legal defensibility must be better understood prior to projects such as this to ensure that the final product is openly useful. The NFAA does not have language specifically referring to a defined legal standard, however it does recommend international cooperation and designates investigative agency who are bound by legal standards. The most relevant to nuclear forensics methods being the Daubert standard as it applies to Federal Rules of Evidence, Article

7, Rule 702 [3][4]. Any country wishing to attribute a nuclear incident to another sovereign nation or subnational entity will face intense scrutiny and as such must have a high standard

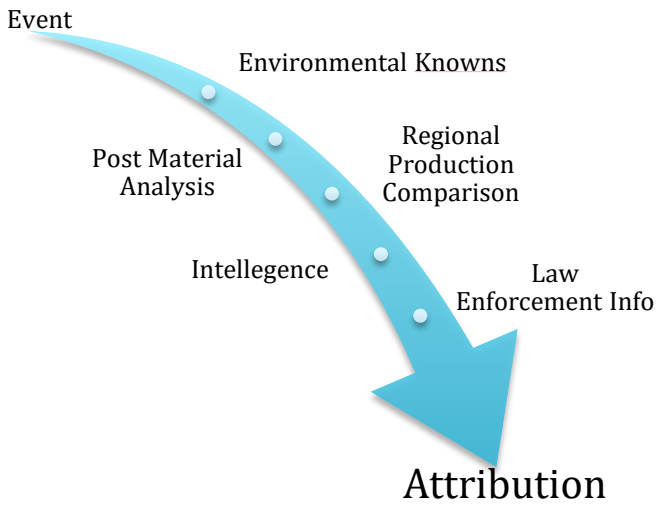



**Figure 2: Application of Methodology to Real World Events.**

of legally defensible forensic methodology. Based on the Daubert standard, judges are given means by which they can assess an expert's scientific testimony on the grounds of reasoning or methodology that is scientifically valid and can properly be applied to the facts. Under this standard, five factors are used to assess a methods validity: 1) whether the theory or technique in question can be and has been tested; 2) whether it has been subjected to peer review and publication; 3) its known or potential error rate; 4) the existence and maintenance of standards controlling its operation; and 5) whether it has attracted widespread acceptance within a relevant

scientific community [5]. For the United States, any research effort seeking board acceptance and government support must meet this standard.

Application of this standard to forensics has rightly received rigorous attention in the scientific community [3]. Understanding the law through precedence is one of the only reliable means of interpreting law, General Electric Co. v. Joiner points out that any gaps between reasoning and evidence and any abuse of discretion may invalid an expert's testimony [6]. In addition, efforts are being made to establish certified reference materials (CRM) and recognized databases of nuclear information that may act as known standard for other nuclear materials [7]. Both of these standards generally agree with the requirements for competence outlined in International Organization for Standardization (ISO) code 17025.

## 2: Methodology

The study covered four methods with four samples per method. Each of the four sample was completed in triplicate for each method to show repeatability, however three of the dissolutions were omitted due to various issues during the dissolution processes. Table 1 below shows the runs complete with the naming conventions that will be used for this thesis.

**Table 1: Terminology for Samples and Runs.**

| Methods / Samples | Auxier Method | Eppich Method | Lithium Method | Maxwell Method |
|---|---|---|---|---|
| **Trinitite** | Run 1<br>2<br>3 | Run 1<br>2<br>3* | Run 1<br>2<br>3 | Run 1<br>2<br>3 |
| **STF Gad Mod 1** | Run 1<br>2<br>3 | Run 1<br>2<br>3 | Run 1<br>2<br>3 | Run 1<br>2<br>3 |
| **NYC IND 1** | Run 1<br>2<br>3 | Run 1*<br>2<br>3 | Run 1<br>2<br>3 | Run 1<br>2<br>3 |
| **MAPEP MaS 32** | Run 1<br>2*<br>3 | Run 1<br>2<br>3 | Run 1<br>2<br>3 | Run 1<br>2<br>3 |

* Run Omitted

For all of these methods, lab procedures dictated the use for the following standard materials. High purity water at 18.2 MΩ was used for dilution or cleaning. Reagents for production of samples were obtained from Sigma Aldrich at above 99% purity. Lithium borates were in ultra-trace purities were obtained from the Corporation Scientifique Claisse. Acids and

bases used in the dissolution processes were obtained from Fisher Scientific and Sigma Aldrich and were not in ultra-trace purities. Weights were obtained using scales with an error of ±0.2 mg.

Each of the methods used were either modified or created to meet the needs of a rapid forensic process. This essential meant that modifications were made to reasonably accelerate the process and every effort was made to retain all of the elements in the process. Additionally, each process was done in triplicate on each sample for repeatability and reliability. The samples were always separated into three aliquots of equal mass.

It is also noteworthy that all surrogate samples simulating nuclear weapons have not experienced a neutron flux of any kind. While the High Flux Isotope Reactor (HFIR) at Oakridge National Laboratory (ORNL) was used in the past to irradiate samples to simulate a detonations activation of materials, this would not be appropriate for an experimental project still testing chemistry methods. This is the result of isotopic invariance to chemical processes.

### 2.1: The Eppich Procedure

The Eppich Method utilized a multiple-stage acidic dissolution process [8]. Stage 1 utilizes a 2.5:1 ratio of concentrated $HNO_3$ and concentrated HF to react for 24 hours until a white fluoride precipitate is formed. Assumptions made for stage 1 were definitions of concentrated $HNO_3$ is 15.8 M, concentrated HF is 22.6 M, the sample to acid concentration was made to 15 mg of sample to 17.5 mL of acidic solution, and an evaporation temperature of 80 ºC. Stage 2 adds 1 mL of concentrated $HClO_4$ to the fluoride precipitate and evaporates to remove fluoride. Assumptions for stage 2 were concentrated $HClO_4$ of 11.6 M and an evaporation temperature of 150 ºC. Finally, dissolution is performed using concentrated HCl then drying and dissolving again into 3 M HCl. Assumptions for stage 3 were concentrated HCl is 12.1 M, 6 mL

of 3 M HCl was used on the sample, and evaporation for this stage were at 150 ºC. The sample is then transferred to a watch glass of a known weight and evaporated so the sample dry weight can be obtained.

### 2.2: The Auxier Procedure

The Auxier Method is an unpublished modified version of the above Eppich Method. It utilizes a similar three stage process with different ratios and reaction environments. The changes were made specifically to improve the method's timeliness and sample independence. Stage 1 utilizes a 5:2 ratio of 14 mL of 15.8 M HF and 22.6 M $HNO_3$ to react with 10 mg of sample in a 32 mL Parr microwave bomb. The bomb is heated at 700 watts for 35 seconds and then air cooled for approximately 20-30 minutes before opening. The sample is then transferred to a Teflon container for evaporation at 225 ºC. For stage 2 the sample is transferred to a large watch glass adding a 5:2 ratio of 7 mL of 22.6 M $HNO_3$ and 11.6 M $HClO_4$ then evaporates at 225 ºC. Stage 3 adds 2 mL of 12.1 M HCl and evaporates at 225 ºC. The chlorine salt precipitate is then transferred to a watch glass of a known weight and evaporated so the sample dry weight can be obtained.

### 2.3: The Lithium Procedure

The Lithium Method was provided by Dr. Dominic Larivière from Laval University, Canada. This method is one presented at the MARC X conference and incorporates the M4 Fluxer procedure from Claisse's technical manuals [9][10]. Dr. Larivière also provided at guide (Appendix D) to his procedure which was modified slightly for the available furnace capacity [11]. The procedure executed in the lab use a 1:6.8 ratio of sample to flux where 0.5 grams of sample was dissolved. Flux is a combination of equal parts 98.5:1.5 ultrapure lithium metaborate ($LiMBO_2$) and lithium bromide, and 99:1 ultrapure lithium tetraborate ($LiTBO_2$) and lithium

bromide. The lithium bromide is used to prevent adhesion to the crucible during cooling. The sample is placed in a 95:5 platinum and gold crucible of 25 mL capacity. The sample is loaded into the crucible and heated for 60 seconds at 80-100 ºC to dry the sample. The flux is then added to the crucible and placed in the oven for 60 seconds at 1000 ºC. The crucible is removed and allowed to cool for 60 seconds and is then placed back in the oven at 1000 ºC for 180 seconds. The sample is removed from the oven and cools until it begins to crack during freezing. Given the above sample and flux masses, this occurs between 45-60 seconds. The sample is placed in an acid bath of 75 mL of 6 M $HNO_3$ and 3 M HF. The acid is evaporated for 24 hours at 80 ºC and then transferred to a watch glass of a known weight and evaporated so the sample dry weight can be obtained. Due to a limited number of crucibles, each was heavily reused in this process, between samples each crucible was cleaned using a 6 M HCl solution and high purity water.

### 2.4: The Maxwell Procedure

The Maxwell Method uses a NaOH flux to isolate specific actinides [12]. This procedure was heavily modified to attempt complete dissolution and retention of all elements. Additionally, a large amount of the sample preparation in Dr. Maxwell's method is removed for this application because the samples selected for comparison do not contain organic materials like the asphalt analyzed in the paper. A 0.5 gram sample is loaded into a 35 mL high purity graphite crucible with 2 grams of NaOH in air. A high purity graphite lid is used to prevent leakage from the crucible. The crucible is heated to 600 ºC for 20 minutes and then cooled for 10-20 minutes. Water is used to remove the supernate and sample from the crucible into 10 mL of 12.1 M HCl. 22.6 M $HNO_3$ is used to assist in removing any sample from the crucible. The sample is then

evaporate at 200 ºC. The sample was then transferred to a beaker of known weight and evaporated so the sample dry weight can be obtained.

### 2.5: Trinitite Samples

Four samples were selected for analysis. Each is defined with either a known isotopic distribution or accepted estimates. The trinitite sample was comprised of approximately 15 grams of crushed trinitite aggregated from two separate trinitite rocks. The samples were crushed by hand using a mortar and pestle. Dr. Eby shows a commonly accepted isotopic distribution for Trinitite that does not include the actinides found in some trinitite from the unburned weapon fuel and tamper [13]. The accepted mass fractions are listed in Table 2 and are used as an approximation for the ideal case of trinitite.

### 2.6: Surrogate Trinitite Formulation Melt Glass Samples

Surrogate trinitite was developed by Josh Molgaard in 2014 [14]. This melt glass is manufactured with a predicted weapon type that can be changed to meet the needs of an experiment. For this project Gadget Modification 1 was used where the tamper of device is estimated and the appropriate mass fraction is added in the form of UNH. Table 3 shows the actual mass fractions of the oxides that were mixed together. This sample consisted of 2 melts of approximately 1 gram masses, each melted in a high purity graphite crucible for 30 minutes at 1500 ºC. They were crushed by hand using a mortar and pestle and aggregated together. This aggregate was then divided up into aliquots for each run.

### 2.7: Surrogate New York City Melt Glass Samples

This urban matrix is a surrogate developed with three components of an urban environment. Each component is expected to be taken up into a surface detonation fireball of a

nuclear weapon. For the sample used in this analysis, Andy Giminaro *et al.* calculated and showed that the isotopes listed in Table 4 would like result from a nuclear detonation from

**Table 2: Commonly Accepted Isotopic Fractions for Trinitite.**

| Trinitite Composition | | | | | |
|---|---|---|---|---|---|
| *Element* | *Fraction of Total Mass* | *Isotope* | *Natural Abundance* | *AMU* | *Nuclide Isotope % of Total Mass* |
| Si | 21.80% | Si-28 | 92.22% | 27.97692653 | 20.1046% |
| | | Si-29 | 4.69% | 28.9764947 | 1.0213% |
| | | Si-30 | 3.09% | 29.97377017 | 0.6741% |
| Al | 4.63% | Al-27 | 100.00% | 26.9815386 | 4.6300% |
| Ca | 3.70% | Ca-40 | 96.94% | 39.962591 | 3.5868% |
| | | Ca-42 | 0.65% | 41.958618 | 0.0239% |
| | | Ca-43 | 0.14% | 42.9587666 | 0.0050% |
| | | Ca-44 | 2.09% | 43.9554818 | 0.0772% |
| | | Ca-46 | 0.00% | 45.953693 | 0.0001% |
| | | Ca-48 | 0.19% | 47.952534 | 0.0069% |
| K | 1.46% | K-39 | 93.26% | 38.9637067 | 1.3616% |
| | | K-41 | 6.73% | 40.9618258 | 0.0983% |
| O | 68.50% | O-16 | 99.76% | 15.99491462 | 68.3335% |
| | | O-17 | 0.04% | 16.9991317 | 0.0260% |
| | | O-18 | 0.21% | 17.999161 | 0.1404% |

downtown New York City (NYC) [15]**.** In addition, a weapon configuration of Improvised Nuclear Device (IND) 1 was selected for the actinide elements added to the sample. IND 1 is a device that uses natural uranyl nitrate to represent fuel for a tamper and fuel pit. The sample was mixed with the elements listed in Table 4 and melted in a high purity graphite crucible at 1550

ºC for 30 minutes. Four 1 gram samples were created and crushed by hand using a mortar and pestle. These were aggregated together and then divided up into aliquots for each run.

## 2.8: MAPEP Samples

MAPEP MaS 32 is a complex soil analyte produced by the Radiological and Environmental Sciences Laboratory [16]. The analyte has a well-known composition and is verified twice a year by a number of laboratories and universities. This sample contains a

**Table 3: Known Isotopic Fractions for STF MOD 1.**

| STF MOD 1 | | | | | | |
|---|---|---|---|---|---|---|
| *Element* | *Fraction of Total Mass* | *Isotope* | *Natural Abundance* | *AMU* | *Normalized Fraction* | *Nuclide Isotope % of Total Mass* |
| Si | 26.90% | Si-28 | 92.22% | 27.97692653 | 26.8463% | 24.7585% |
| | | Si-29 | 4.69% | 28.9764947 | | 1.2577% |
| | | Si-30 | 3.09% | 29.97377017 | | 0.8301% |
| Al | 8.50% | Al-27 | 100.00% | 26.9815386 | 8.4830% | 8.4830% |
| Ca | 7.49% | Ca-40 | 96.94% | 39.962591 | 7.4750% | 7.2464% |
| | | Ca-42 | 0.65% | 41.958618 | | 0.0484% |
| | | Ca-43 | 0.14% | 42.9587666 | | 0.0101% |
| | | Ca-44 | 2.09% | 43.9554818 | | 0.1559% |
| | | Ca-46 | 0.00% | 45.953693 | | 0.0003% |
| | | Ca-48 | 0.19% | 47.952534 | | 0.0140% |
| Fe | 1.64% | Fe-54 | 5.85% | 53.939611 | 1.6367% | 0.0957% |
| | | Fe-56 | 91.75% | 55.934937 | | 1.5018% |
| | | Fe-57 | 2.12% | 56.935394 | | 0.0347% |
| | | Fe-58 | 0.28% | 57.933276 | | 0.0046% |
| Mg | 0.62% | Mg-24 | 78.99% | 23.9850417 | 0.6188% | 0.4888% |
| | | Mg-25 | 10.00% | 24.98583692 | | 0.0619% |
| | | Mg-26 | 11.01% | 25.98259293 | | 0.0681% |
| | | Na-23 | 100.00% | 22.98976928 | | 0.6188% |
| K | 3.94% | K-39 | 93.26% | 38.9637067 | 3.9321% | 3.6670% |
| | | K-41 | 6.73% | 40.9618258 | | 0.2646% |
| Ti | 0.31% | Ti-46 | 8.25% | 45.952632 | 0.3094% | 0.0255% |

**Table 3 Continued.**

| STF MOD 1 | | | | | | |
|---|---|---|---|---|---|---|
| *Element* | *Fraction of Total Mass* | *Isotope* | *Natural Abundance* | *AMU* | *Normalized Fraction* | *Nuclide Isotope % of Total Mass* |
| | | Ti-47 | 7.44% | 46.951763 | | 0.0230% |
| | | Ti-48 | 73.72% | 47.947946 | | 0.2281% |
| | | Ti-49 | 5.41% | 48.94787 | | 0.0167% |
| O | 49.60% | Ti-50 | 5.18% | 49.944791 | 49.5010% | 2.5642% |
| | | O-16 | 99.76% | 15.99491462 | | 49.3807% |
| | | O-17 | 0.04% | 16.9991317 | | 0.0188% |
| | | O-18 | 0.21% | 17.999161 | | 0.1015% |
| Na | 1.00% | Na-23 | 100.00% | 22.98976928 | 0.9980% | 0.9980% |
| U | 0.20% | U-234 | 0.01% | 234.040952 | 0.1996% | 0.0000% |
| | | U-235 | 0.72% | 235.04393 | | 0.0014% |
| | | U-238 | 99.27% | 238.050788 | | 0.1982% |

**Table 4: Known Isotopic Fractions for Urban Melt Glass NYC IND 1.**

| NYC IND 1 | | | | | | |
|---|---|---|---|---|---|---|
| *Element* | *Fraction of Total Mass* | *Isotope* | *Natural Abundance* | *AMU* | *Normalized Fraction* | *Nuclide Isotope % of Total Mass* |
| Si | 58.08% | Si-28 | 92.22% | 27.97692653 | 52.22% | 48.1618% |
| | | Si-29 | 4.69% | 28.9764947 | | 2.4467% |
| | | Si-30 | 3.09% | 29.97377017 | | 1.6147% |
| Al | 14.55% | Al-27 | 100.00% | 26.9815386 | 13.08% | 13.0828% |
| Ca | 6.64% | Ca-40 | 96.94% | 39.962591 | 5.97% | 5.7878% |
| | | Ca-42 | 0.65% | 41.958618 | | 0.0386% |
| | | Ca-43 | 0.14% | 42.9587666 | | 0.0081% |
| | | Ca-44 | 2.09% | 43.9554818 | | 0.1245% |
| | | Ca-46 | 0.00% | 45.953693 | | 0.0002% |
| | | Ca-48 | 0.19% | 47.952534 | | 0.0112% |
| Fe | 9.88% | Fe-54 | 5.85% | 53.939611 | 8.88% | 0.5193% |
| | | Fe-56 | 91.75% | 55.934937 | | 8.1511% |
| | | Fe-57 | 2.12% | 56.935394 | | 0.1882% |
| | | Fe-58 | 0.28% | 57.933276 | | 0.0251% |
| Mg | 2.68% | Mg-24 | 78.99% | 23.9850417 | 2.41% | 1.9035% |
| | | Mg-25 | 10.00% | 24.98583692 | | 0.2410% |

**Table 4 Continued.**

| NYC IND 1 | | | | | | |
|---|---|---|---|---|---|---|
| *Element* | *Fraction of Total Mass* | *Isotope* | *Natural Abundance* | *AMU* | *Normalized Fraction* | *Nuclide Isotope % of Total Mass* |
| | | Mg-26 | 11.01% | 25.98259293 | | 0.2653% |
| Na | 3.08% | Na-23 | 100.00% | 22.98976928 | 2.77% | 2.7694% |
| K | 3.39% | K-39 | 93.26% | 38.9637067 | 3.05% | 2.8426% |
| | | K-41 | 6.73% | 40.9618258 | | 0.2051% |
| Mn | 11.00% | Mn-55 | 100.00% | 54.938045 | 9.89% | 9.8908% |
| Ti | 0.58% | Ti-46 | 8.25% | 45.952632 | 0.52% | 0.0430% |
| | | Ti-47 | 7.44% | 46.951763 | | 0.0388% |
| | | Ti-48 | 73.72% | 47.947946 | | 0.3845% |
| | | Ti-49 | 5.41% | 48.94787 | | 0.0282% |
| | | Ti-50 | 5.18% | 49.944791 | | 0.0270% |
| O | 0.55% | O-16 | 99.76% | 15.99491462 | 0.49% | 0.4933% |
| | | O-17 | 0.04% | 16.9991317 | | 0.0002% |
| | | O-18 | 0.21% | 17.999161 | | 0.0010% |
| S | 0.06% | S-32 | 94.99% | 31.972071 | 0.05% | 0.0512% |
| | | S-33 | 0.75% | 32.9714588 | | 0.0004% |
| | | S-34 | 4.25% | 33.9678669 | | 0.0023% |
| | | S-36 | 0.01% | 35.9670808 | | 0.0000% |
| Ba | 0.05% | Ba-130 | 0.11% | 129.906321 | 0.04% | 0.0000% |
| | | Ba-132 | 0.10% | 131.905061 | | 0.0000% |
| | | Ba-134 | 2.42% | 133.9045084 | | 0.0011% |
| | | Ba-135 | 6.59% | 134.9056886 | | 0.0030% |
| | | Ba-136 | 7.85% | 135.9045759 | | 0.0035% |
| | | Ba-137 | 11.23% | 136.9058274 | | 0.0050% |
| | | Ba-138 | 71.70% | 137.9052472 | | 0.0322% |
| P | 0.09% | P-31 | 100.00% | 30.9737616 | 0.08% | 0.0809% |
| N | 0.01% | N-14 | 99.64% | 14.00307401 | 0.01% | 0.0090% |
| | | N-15 | 0.36% | 15.0001089 | | 0.0000% |
| C | 0.01% | C-12 | 98.93% | 12 | 0.01% | 0.0089% |
| | | C-13 | 1.07% | 13.00335484 | | 0.0001% |
| U | 0.56% | U-234 | 0.01% | 234.040952 | 0.51% | 0.0000% |
| | | U-235 | 0.72% | 235.04393 | | 0.0037% |
| | | U-238 | 99.27% | 238.050788 | | 0.5043% |

number of trace radioactive elements and some that are expected to be similar to yield outcomes from a simple nuclear weapon. To prepare the MAPEP five 1 gram samples were melted in high purity graphite crucible at 1500 ºC for 30 minutes. Four of the samples were then selected and crushed by hand using a mortar and pestle. These were aggregated together and then divided up into aliquots for each run. Based on the bi-annual analysis, the MAPEP used contains the isotopes of interest listed in Table 5.

## 2.8: ICP-TOF-MS Analysis Method

The inductive coupled time of flight mass spectrometer (ICP-TOF-MS) used for this analysis was an Optimass-9500 produced by GBC. The method used an internal standard

**Table 5: Known Isotopic Fractions in MAPEP MaS 32.**

| MAPEP | | | | | |
|---|---|---|---|---|---|
| *Element* | *Fraction of Total Mass* | *Isotope* | *Natural Abundance* | *AMU* | *Nuclide Isotope % of Total Mass* |
| Be | 0.00003930% | Be-9 | 100.00% | 9.0121830650 | 0.0000393000% |
| V | 0.00009800% | V-50 | 0.25% | 49.94715601 | 0.0000002450% |
| | 0.00009800% | V-51 | 99.75% | 50.9439570400 | 0.0000977550% |
| Cr | 0.00009800% | Cr-50 | 4.35% | 49.94604183 | 0.0000042581% |
| | 0.00009800% | Cr-52 | 83.79% | 51.9405062300 | 0.0000821132% |
| | 0.00009800% | Cr-53 | 9.50% | 52.94064815 | 0.0000093110% |
| | 0.00009800% | Cr-54 | 2.37% | 53.9388791600 | 0.0000023177% |
| Ni | 0.00000135% | Ni-58 | 68.08% | 57.93534241 | 0.0000009190% |
| | 0.00000135% | Ni-60 | 26.22% | 59.9307858800 | 0.0000003540% |
| | 0.00000135% | N-61 | 1.14% | 60.9310555700 | 0.0000000154% |
| | 0.00000135% | N-62 | 3.63% | 61.9283453700 | 0.0000000491% |
| | 0.00000135% | N-64 | 0.93% | 63.92796682 | 0.0000000125% |
| Zn | 0.00016100% | Zn-64 | 48.60% | 63.9291420100 | 0.0000782460% |
| | 0.00016100% | Zn-66 | 27.90% | 65.9260338100 | 0.0000449190% |
| | 0.00016100% | Zn-67 | 4.10% | 66.92712775 | 0.0000066010% |
| | 0.00016100% | Zn-68 | 18.80% | 67.9248445500 | 0.0000302680% |
| | 0.00016100% | Zn-70 | 0.60% | 69.9253192000 | 0.0000009660% |

**Table 5 Continued.**

| MAPEP | | | | | |
|---|---|---|---|---|---|
| *Element* | *Fraction of Total Mass* | *Isotope* | *Natural Abundance* | *AMU* | *Nuclide Isotope % of Total Mass* |
| Co | 0.00010900% | Co-59 | 100.00% | 58.9331942900 | 0.0001090000% |
| Cu | 0.00018300% | Cu-63 | 69.15% | 62.92959772 | 0.0001265445% |
| | 0.00018300% | Cu-65 | 30.85% | 64.9277897000 | 0.0000564555% |
| Se | 0.0000001230% | Se-74 | 0.87% | 73.922475934 | 0.0000000011% |
| | 0.0000001230% | Se-76 | 9.36% | 75.919213704 | 0.0000000115% |
| | 0.0000001230% | Se-77 | 7.63% | 76.9199141540 | 0.0000000094% |
| | 0.0000001230% | Se-78 | 23.78% | 77.9173092800 | 0.0000000292% |
| | 0.0000001230% | Se-80 | 49.61% | 79.9165218 | 0.0000000610% |
| | 0.0000001230% | Se-82 | 8.73% | 81.9166995000 | 0.0000000107% |
| As | 0.00005560% | As-75 | 100.00% | 74.9215945700 | 0.0000556000% |
| Tc | 0.0000000000% | Tc-99 | 100.00% | 98.9062508000 | 0.0000000000% |
| Cd | 0.00001890% | Cd-106 | 1.25% | 105.9064599000 | 0.0000002363% |
| | 0.00001890% | Cd-108 | 0.89% | 107.9041834000 | 0.0000001682% |
| | 0.00001890% | Cd-110 | 12.49% | 109.9030066100 | 0.0000023606% |
| | 0.00001890% | Cd-111 | 12.80% | 110.9041828700 | 0.0000024192% |
| | 0.00001890% | Cd-112 | 24.13% | 111.9027628700 | 0.0000045606% |
| | 0.00001890% | Cd-113 | 12.22% | 112.9044081300 | 0.0000023096% |
| | 0.00001890% | Cd-114 | 28.73% | 113.9033650900 | 0.0000054300% |
| | 0.00001890% | Cd-116 | 7.49% | 115.9047631500 | 0.0000014156% |
| Ag | 0.0000009970% | Ag-107 | 51.84% | 106.9050916000 | 0.0000005168% |
| | 0.0000009970% | Ag-109 | 48.16% | 108.9047553000 | 0.0000004802% |
| Sb | 0.00012000% | Sb-121 | 57.36% | 120.9038120000 | 0.0000688320% |
| | 0.00012000% | Sb-123 | 42.64% | 122.9042132000 | 0.0000511680% |
| Ba | 0.00048500% | Ba-130 | 0.11% | 129.9063207000 | 0.0000005141% |
| | 0.00048500% | Ba-132 | 0.10% | 131.9050611000 | 0.0000004899% |
| | 0.00048500% | Ba-134 | 2.42% | 133.9045081800 | 0.0000117225% |
| | 0.00048500% | Ba-135 | 6.59% | 134.9056883800 | 0.0000319712% |
| | 0.00048500% | Ba-136 | 7.85% | 135.9045757300 | 0.0000380919% |
| | 0.00048500% | Ba-137 | 11.23% | 136.9058271400 | 0.0000544655% |
| | 0.00048500% | Ba-138 | 71.70% | 137.9052470000 | 0.0003477450% |
| Hg | 0.00000042% | Hg-196 | 0.15% | 195.9658326000 | 0.0000000006% |
| | 0.00000042% | Hg-198 | 9.97% | 197.9667686000 | 0.0000000415% |
| | 0.00000042% | Hg-199 | 16.87% | 198.9682806400 | 0.0000000702% |
| | 0.00000042% | Hg-200 | 23.10% | 199.9683265900 | 0.0000000961% |
| | 0.00000042% | Hg-201 | 13.18% | 200.9703028400 | 0.0000000548% |
| | 0.00000042% | Hg-202 | 29.86% | 201.9706434000 | 0.0000001242% |
| | 0.00000042% | Hg-204 | 6.87% | 203.9734939800 | 0.0000000286% |

**Table 5 Continued.**

| MAPEP | | | | | |
|---|---|---|---|---|---|
| *Element* | *Fraction of Total Mass* | *Isotope* | *Natural Abundance* | *AMU* | *Nuclide Isotope % of Total Mass* |
| Tl | 0.00020200% | Tl-203 | 29.52% | 202.9723446000 | 0.0000596385% |
| | 0.00020200% | Tl-205 | 70.48% | 204.9744278000 | 0.0001423615% |
| Pb | 0.00007100% | Pb-204 | 1.40% | 203.9730440000 | 0.0000009940% |
| | 0.00007100% | Pb-206 | 24.10% | 205.9744657000 | 0.0000171110% |
| | 0.00007100% | Pb-207 | 22.10% | 206.9758973000 | 0.0000156910% |
| | 0.00007100% | Pb-208 | 52.40% | 207.9766525000 | 0.0000372040% |
| U | 0.00000005% | U-235 | 0.72% | 235.0439300000 | 0.0000000003% |
| | 0.00001620% | U-238 | 99.27% | 238.0507880000 | 0.0000160824% |
| Am | 0.00000000% | Am-241 | 100.00% | 241.0568293000 | 0.0000000000% |
| Cs | 0.00000000% | Cs-134 | 100.00% | 133.9067184750 | 0.0000000000% |
| Co | 0.00000000% | Co-57 | 100.00% | 56.9362914000 | 0.0000000000% |
| Fe | 0.00000000% | Fe-55 | 100.00% | 54.9382934000 | 0.0000000000% |
| Mn | 0.00000000% | Mn-54 | 100.00% | 53.9403589000 | 0.0000000000% |
| Ni | 0.00000000% | Ni-63 | 100.00% | 62.9296694000 | 0.0000000000% |
| Pu | 0.00000000% | Pu-238 | 100.00% | 238.0495601000 | 0.0000000000% |
| Pu239/240 | 0.00000000% | Pu-239/240 | 100.00% | 239.0521636000 | 0.0000000000% |
| K | 0.00000000% | K-40 | 100.00% | 39.9639981660 | 0.0000000000% |
| Sr | 0.00000000% | Sr-90 | 100.00% | 89.9077380000 | 0.0000000000% |
| U234/233 | 0.00000000% | U-234/233 | 100.00% | 233.0396355000 | 0.0000000000% |
| Zn | 0.00000000% | Zn-65 | 100.00% | 64.9292410000 | 0.0000000000% |

comprised of Inorganic Ventures IV-Stock-21 a multi-element mass spec standard, CCS-1 a rare earth mass spec standard, and a silicon standard. Using these three standards the following elements were bench marked in both runs of the mass spectrometer: Ag, Al, As, Ba, Be, Bi, Ca, Ce, Cd, Co, Cr3, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ho, In, K, La, Li, Lu, Mg, Mn, Na, Nd, Ni, Pb, Pr, Rb, Sc, Se, Si, Sm, Sr, Tb, Th, Tl, Tm, U, V, Y, Yb, and Zn. The calibration standards used had the following concentrations in parts per billion (ppb): 1, 5, 50, 100, 250, 500, and 1000.

Prior to introduction every sample is doped with 2% $HNO_3$ to assist in preventing contamination from successive sample inductions. Additionally, to prevent contamination 45 seconds of high purity water with 2% $HNO_3$ is run between each sample. The samples are inducted for 40 seconds into the sample intake system seen in Figure 3. 10 seconds is used to pump in the sample and prime the detector, then 3 replications are taken of the sample. Each replication is integrated signal strength over 10 seconds. Unfortunately the software provided with the Optimass system does not allow for easy comparison of data, therefore python code was used to manipulate the raw data files from each replicate to generate results.

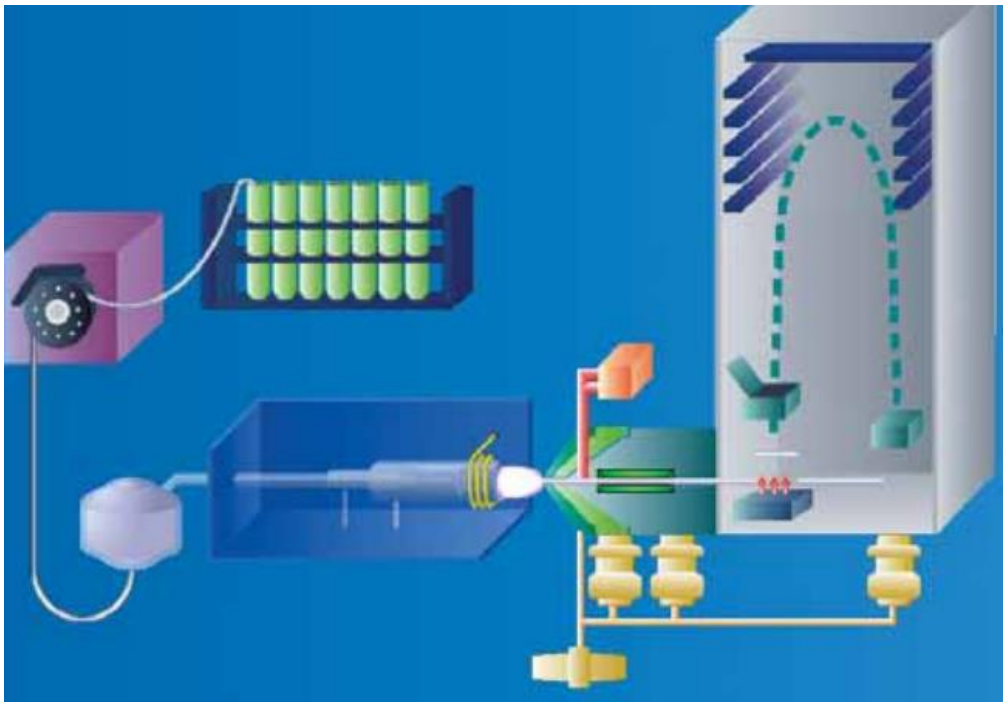


**Figure 3: Optimass-9500 Operational Schematic.**

# 3: Results

Results were derived from a code generated in Python for analyzing internal calibration standards with a first order polynomial fit. This curve is then applied to the average of the replicates per atomic mass unit (amu) bin. A peak is input by the user and given a 0.1 amu width and the peak area is integrated to find the total signal intensity for the given element. This is then multiplied by the calibration curve resulting in a concentration value. This elemental concentration is divide the total concentration of the sample introduced to the mass spec. This ratio is then compared to the ratio of ideal elemental mass per total mass of the sample prior to dissolution. The value output is the effective yield of the dissolution procedure. Additionally, on these graphs the Modified Eppich Method is the Auxier Method

## 3.1: Uranium Comparison

The uranium content was measured about the 238 peak. Figure 4 shows the fraction of ideal for each method. The yields for MAPEP are misleading because of extremely low expected quantities of uranium relative to the background signal. Error is represented using the standard deviation of the concentrations and assumes no error in the ideal mass. No uranium is expected in the trinitite. Of the four procedures Auxier Method is one order of magnitude above it next closest competitor, Eppich.

## 3.2: Barium Comparison

Data is represented the same way as it was with uranium. The error is also calculated the same way. The isotope of barium is measured about the 138 peak. Figure 5 shows the fraction of ideal for each method. The results are extremely similar with MAPEP suffering from misleading results because of extremely low expected quantities. In this case, barium is not expected in

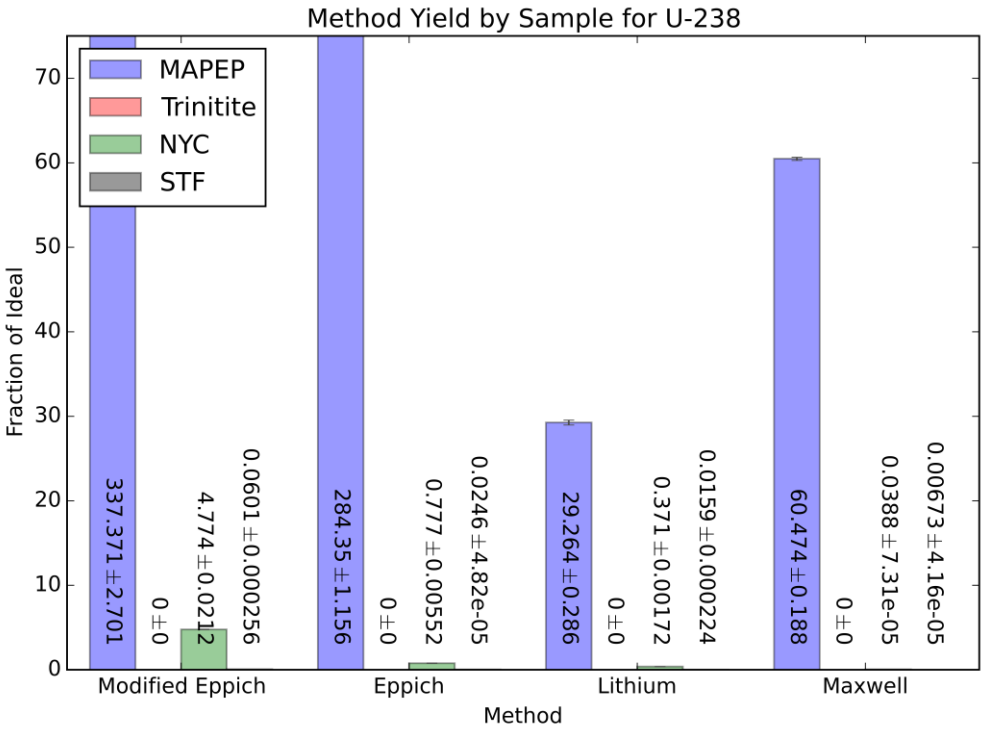



**Figure 4: Method Yield by Sample for U-238.**

trinitite and STF. Of the four procedures Auxier Method is almost one order of magnitude above it next closest competitor, Eppich. Both Lithium and Maxwell are another order of magnitude behind Eppich.

### 3.3: Comparison of Lighter Elements

The lighter elements deviate from the previous trend with barium and uranium. The Auxier procedure is not necessarily the best for each element however does show the most consistency with Al-27. Again, MAPEP does not include Al-27 or Fe-56 and Trinitite does not include Ni-58 or Fe-56. The MAPEP continues to be misleading with results well above 100% compared to ideal and nickel shows a significant peaks in Figure 7.

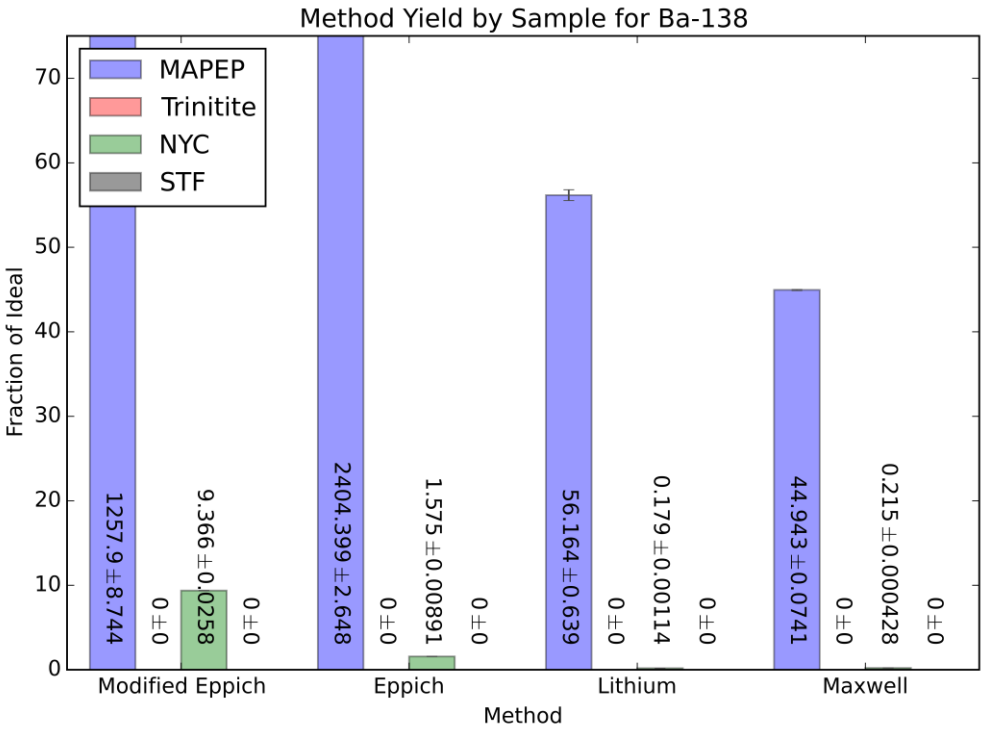



**Figure 5: Method Yield by Sample for Ba-138.**

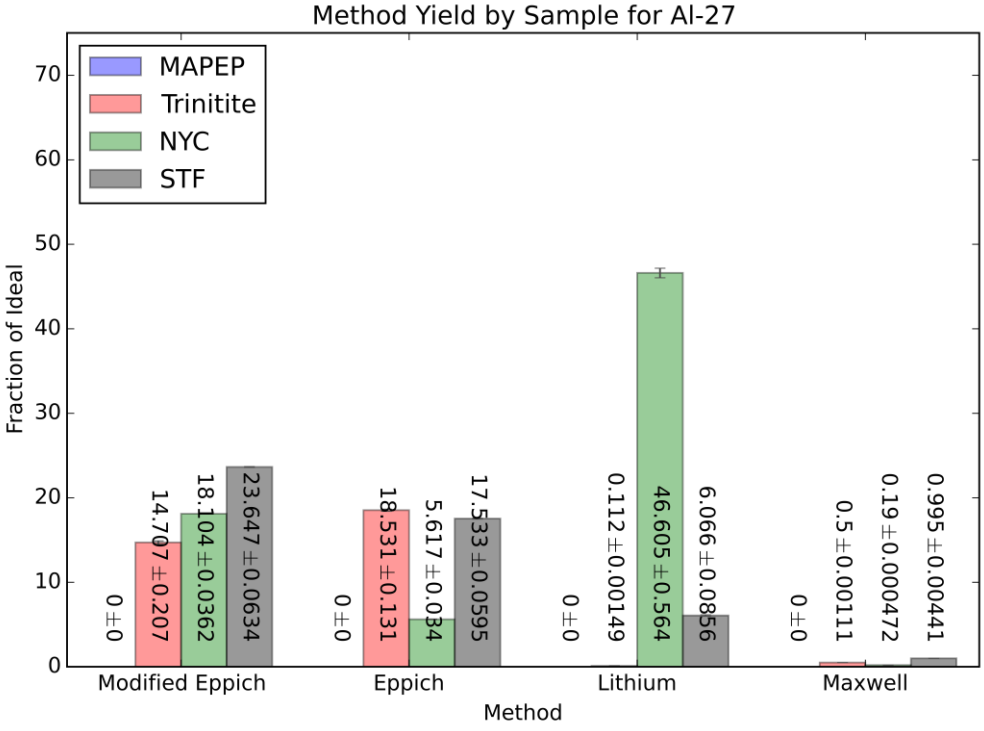



**Figure 6: Method Yield by Sample for Al-27.**

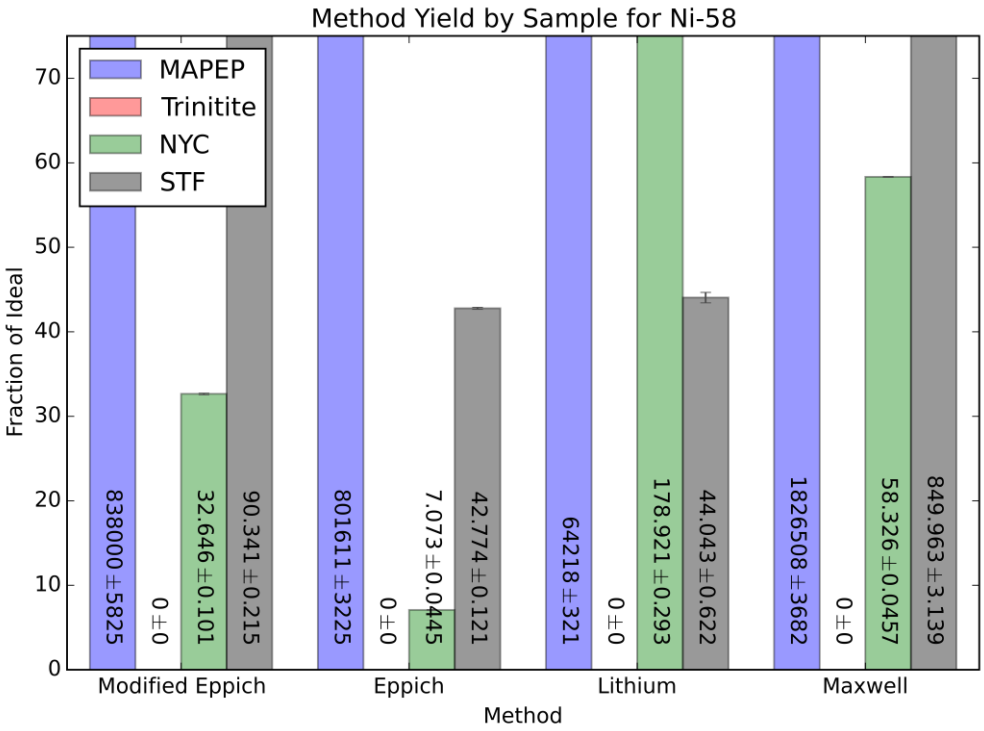



**Figure 7: Method Yield by Sample for Ni-58.**

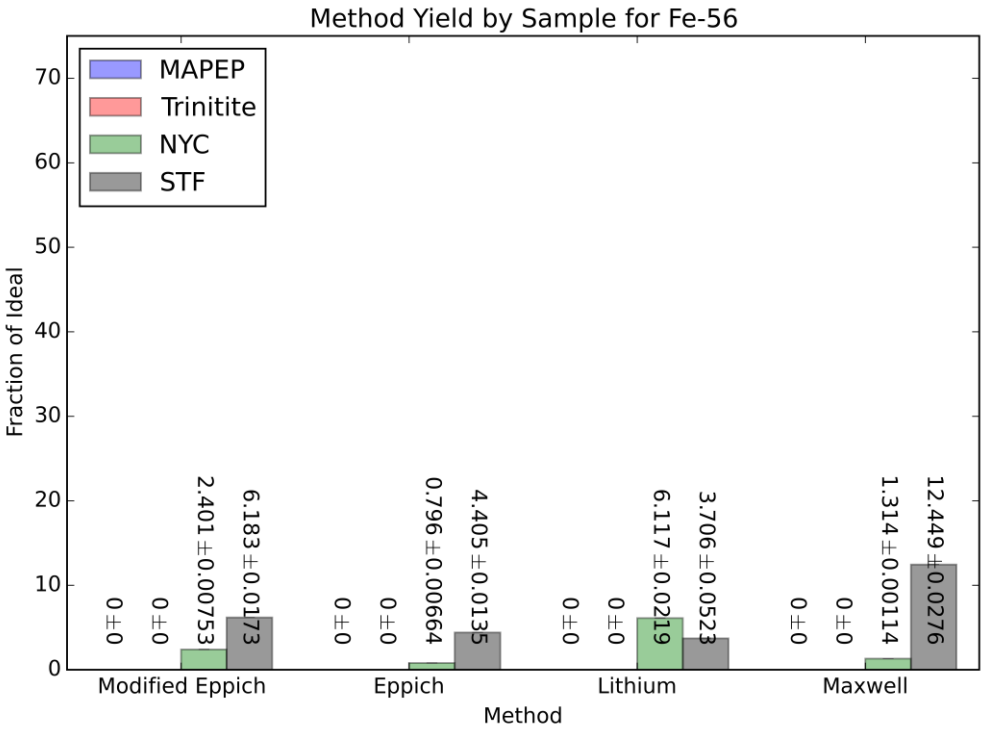



**Figure 8: Method Yield by Sample for Fe-56.**

## 4: Conclusion

In conclusion the Auxier Method shows the most reliability and some sample independence with the isotopic yields. The ICP-TOF-MS performances appears varied across the data. For rapid analysis, extracting isotopic information may be better obtained from a laser-ablation mulit-collector mass spectrometer (LA-MC-MS). The current method will however serve gas phase separation techniques well as it needed for phase conversion.

The Lithium and Maxwell techniques that were tested may be useful for other purposes but for the requirements demanded of this process they do not serve a useful purpose. With the elements of interest occurring at near back ground levels while approaching the limits of sample intake concentration on the ICP-TOF-MS due to other elements introduced, it is likely that the modifications to the methodologies will need to be changed if attempted again. Additionally, adding lithium to the samples will cause particular challenges if the debris of interest is generated from a boosted or thermonuclear device.

## 5: Future Work

Continued work into research on dissolutions is evident as none of the methods presented here, or any methods identified in the literature review provide 100% dissolution. While the current methods would likely meet the legal standard for international courts, they likely do not provide a definitive answer that is robust enough to apply to real world events, particularly those involving the complexities of a modern urban environment.

In order to one day answer this question future work should focus on high pressure and temperature applications to further traditional acidic methods. High pressure systems with acids similar to those found in the Eppich procedure present particular hazards that must receive special attention if attempted in an academic environment.

Additionally, fusion methods may be continued but the complex chemistry and radioactive nature may present particular challenges in real world applications. More importantly though, there is a concern about removal of particular isotopes in the sedimentation processes which would skew the critical proportionality ratios.

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# Appendixes

# Appendix A – Lithium Method

**Alkaline Fusion**

Soil solutions were prepared using fluxer digestion (0.5 g of soil was dissolved in 3.4 g of flux) for soil and sediments; solutions with high amounts of iron (such as red sludges) used a mix of $LiMBO_2$/$LiTBO_2$ (1.7 g of $LiMBO_2$ and 1.7 g $LiTBO_2$).The fluxes were purchased from Corporation Scientifique Claisse and are composed of ultrapure lithium metaborate : lithium bromide (98.5:1.5) and lithium tetraborate : lithium bromide (99:1).(Bouchard, M.; Rivard, S. Ness, S. *ISO 9516-1 Simplified Borate Fusion & WDXRF Analytical Method for Iron Ores Analysis Including Exploration Samples*; Technical Report from Corporation Scientifique Claisse: Quebec, QC, 2013) Lithium bromide was added to ensure that the flux would not stick to the crucibles.

The protocol used for the fusion was a revised method from the one suggested by the manufacturer for the M4 Fluxer. (Table S3) but the same fusion parameters were used regardless of the flux mix used. Pouring of the fused sample into 100 mL 3 M $HNO_3$ solution was preferred; this was found to be the optimal concentration to enhance the solubility of lithium metaborate and tetraborate and enhance the solubility of most metals in solution. After dissolution, the samples were clear solutions and were stable for weeks so did not require further filtration.

Solutions were treated with PEG-6000 to eliminate the silica (Dai, X.; Kramer-Tremblay S. *Health Phys.* **2011**, *101,* 144-147.) in solution, which could impact the analysis by clogging the ICP-MS nebuliser or the cartridge if the analyte were separated via solid phase extraction.

**Fusion Protocol**

**Table S2** : Fusion Protocol Used on the M4 fluxer for the Dissolution of Environmental Samples.

| Function | Steps | Time (min) |
|---|---|---|
| | | Proposed Protocol |
| 0 | Pre-heating | 00:05 |
| 1 | Oxidation | 00:30 |
| 2 | First dissolution | 00:30 |
| 3 | Cooling | – |
| 4 | Heating | 01:00 |
| 5 | Final dissolution | 03:00 |
| 6 | Cooling | - |
| 7 | Pouring | 00:10 |
| 8 | Stirring | 10:00 |
| Total | | 15:15 |

# Appendix B – Python Analysis Scripts

**File Average Tool**

```
import numpy as np
import csv
import shutil as sh

numFiles = 1

method = "LithSTF"
run = "2"
replicants = [3,0,0]

file1 = method+run+str(replicants[0])+".scn"
file2 = method+run+str(replicants[1])+".scn"
file3 = method+run+str(replicants[2])+".scn"
outFile = method+run+".csv"


#file1 = "EppichSTF11.scn"
#file2 = "EppichSTF12.scn"
#file3 = "EppichSTF13.scn"
#outFile = "EppichSTF1.csv"

if numFiles == 1:
num_lines = sum(1 for line in open(file1))
inCalib = np.genfromtxt(file1, skip_footer = (num_lines-4), skip_header = 1,delimiter =
"\t",usecols = 1)
inFile1 = np.genfromtxt(file1, skip_header = 4, skip_footer = 3)
elif numFiles == 2:
num_lines = sum(1 for line in open(file1))
inCalib = np.genfromtxt(file1, skip_footer = (num_lines-4), skip_header = 1,delimiter =
"\t",usecols = 1)
inFile1 = np.genfromtxt(file1, skip_header = 4, skip_footer = 3)
inFile2 = np.genfromtxt(file2, skip_header = 4, skip_footer = 3)
elif numFiles == 3:
num_lines = sum(1 for line in open(file1))
inCalib = np.genfromtxt(file1, skip_footer = (num_lines-4), skip_header = 1,delimiter =
"\t",usecols = 1)
inFile1 = np.genfromtxt(file1, skip_header = 4, skip_footer = 3)
inFile2 = np.genfromtxt(file2, skip_header = 4, skip_footer = 3)
inFile3 = np.genfromtxt(file3, skip_header = 4, skip_footer = 3)

# Write the data to a csv file
if numFiles == 1:
outArray = np.zeros((num_lines,7))
for i in range(0,2):
```

```
outArray[i,0] = inCalib[i]
for i in range(0,len(inFile1)):
outArray[i,1] = ((i-inCalib[0])/inCalib[1])**2
outArray[i,2] = inFile1[i]
elif numFiles == 2:
outArray = np.zeros((num_lines,7))
for i in range(0,2):
outArray[i,0] = inCalib[i]
for i in range(0,len(inFile1)):
outArray[i,1] = ((i-inCalib[0])/inCalib[1])**2
outArray[i,2] = inFile1[i]
outArray[i,3] = inFile2[i]
for i in range(0,len(inFile1)):
outArray[i,5] = (inFile1[i]+inFile2[i])/2
outArray[i,6] = np.std((inFile1[i],inFile2[i]))

elif numFiles == 3:
outArray = np.zeros((num_lines,7))
for i in range(0,2):
outArray[i,0] = inCalib[i]
for i in range(0,len(inFile1)):
outArray[i,1] = ((i-inCalib[0])/inCalib[1])**2
outArray[i,2] = inFile1[i]
outArray[i,3] = inFile2[i]
outArray[i,4] = inFile3[i]
for i in range(0,len(inFile1)):
outArray[i,5] = (inFile1[i]+inFile2[i]+inFile3[i])/3
outArray[i,6] = np.std((inFile1[i],inFile2[i],inFile3[i]))

with open(outFile,"w") as f:
writer = csv.writer(f)
writer.writerows(outArray)
f.close()

if numFiles == 1:
sh.move(file1,"ProcessedSCN/")
elif numFiles == 2:
sh.move(file1,"ProcessedSCN/")
sh.move(file2,"ProcessedSCN/")
elif numFiles == 3:
sh.move(file1,"ProcessedSCN/")
sh.move(file2,"ProcessedSCN/")
sh.move(file3,"ProcessedSCN/")

sh.move(outFile,"ProcessedCSV/")
```

**Data Analysis Tool 1**

```
import glob
import os
import numpy as np
import csv
import matplotlib.pyplot as plt
import shutil as sh

concName = np.genfromtxt("ConcLibrary.txt", delimiter = ",", usecols = 0, dtype = "str")
concVal = np.genfromtxt("ConcLibrary.txt", delimiter = ",", usecols = 1)

corrVal = 1
stanConc = 850

plt.close("all")

lbound = 230
ubound = 245

stanData = np.genfromtxt("CS1.txt",delimiter = ",")

for i in range(0,len(stanData)-1):
stanData[i,5] = stanData[i,5]*stanConc

for file in glob.glob("*.csv"):
fileName = file
fileBase = str(os.path.splitext(fileName)[0])
outFile = fileBase+"_ConcCorrection.csv"
dataIn = np.genfromtxt(fileName, delimiter = ",")
concData = np.zeros((len(dataIn),3))
for i in range(0,len(concName)-1):
if concName[i] == fileBase:
index = i
for i in range(0,len(concData)-1):
concData[i,0] = dataIn[i,1]
concData[i,1] = concVal[index]*dataIn[i,5]*corrVal
concData[i,2] = concVal[index]*dataIn[i,6]
with open(outFile,"w") as f:
writer = csv.writer(f)
writer.writerows(concData)
f.close()
plt.figure()
plt.plot(concData[:,1])
plt.plot(stanData[:,5])
plt.xlim((lbound,ubound))
```

```
plt.xlabel("Mass (AMU)")
plt.ylabel("Concentration ($^{\mu g}/{mL}$)")
plt.title(fileName)
plt.grid("on")
plt.show()
figurePDF = fileBase + ".pdf"
figurePNG = fileBase + ".png"
plt.savefig(figurePDF, dpi=1000, format='pdf', orientation='landscape',
bbox_inches='tight')
plt.savefig(figurePNG, dpi=1000, format='png', orientation='landscape',
bbox_inches='tight')
sh.move(outFile,"CompletedCSV/")
sh.move(figurePDF,"CompletedFigures/")
sh.move(figurePNG,"CompletedFigures/")
```

**Data Analysis Tool 2**

```
import glob
import os
import numpy as np

concName = np.genfromtxt("ConcLibrary.csv", delimiter = ",", usecols = 0, dtype = "str")
concVal = np.genfromtxt("ConcLibrary.csv", delimiter = ",", usecols = 1)

corrVar = 1

for file in glob.glob("*.csv"):
fileName = file
fileBase = str(os.path.splitext(fileName)[0])
dataIn = np.genfromtxt(fileName, delimiter = ",")
concData = np.zeros((len(dataIn),3))
for i in range(0,len(concName)-1):
if concName[i] == fileName:
index = i
for i in range(0,len(concData)-1):
concData[i,0] = dataIn[i,1]
concData[i,1] = concVal[index]*dataIn[i,5]*corrVar
concData[i,2] = concVal[index]*dataIn[i,6]
```

**Library of Concentrations**

```
a = ["a","b","c"]

Name_Conc_lib = [CS1;
CS2;
CS3;
```

CS4;
CS5;
CS6;
CS7;
METrin1;
METrin2;
METrin3;
MESTF1;
MESTF2;
MESTF3;
MENYC1;
MENYC2;
MENYC3;
MEMAPEP1;
MEMAPEP2;
MEMAPEP3;
EppichTrin1;
EppichTrin2;
EppichTrin3;
EppichSTF1;
EppichSTF2;
EppichSTF3;
EppichNYC1;
EppichNYC2;
EppichNYC3;
EppichMAPEP1;
EppichMAPEP2;
EppichMAPEP3;
LithTrin1;
LithTrin2;
LithTrin3;
LithSTF1;
LithSTF2;
LithSTF3;
LithNYC1;
LithNYC2;
LithNYC3;
LithMAPEP1;
LithMAPEP2;
LithMAPEP3;
MaxTrin1;
MaxTrin2;
MaxTrin3;
MaxSTF1;
MaxSTF2;
MaxSTF3;

```
MaxNYC1;
MaxNYC2;
MaxNYC3;
MaxMAPEP1;
MaxMAPEP2;
MaxMAPEP3]

Conc_lib = [1;
5;
50;
100;
250;
500;
1000;
36.9;
620;
570;
615;
1085;
1363;
1000;
785;
3.45;
2325;
620;
2925;
1615;
1350;
0;
1675;
810;
1555;
23060;
756;
968;
845;
680;
565;
46922;
45332;
94060;
24467;
57.925;
59.475;
3.323;
3.896;
```

5.886;
460.8;
545.4;
625;
19.778;
15.164;
17.075;
7.32;
7.2;
6.73;
9.898;
9.608;
9.72;
7.564;
9.328;
7.526]

**Multi File Plotter Tool Folder**

```
#import glob
import os
import numpy as np
import csv
import matplotlib.pyplot as plt
import shutil as sh

concName = np.genfromtxt("ConcLibrary.txt", delimiter = ",", usecols = 0, dtype = "str")
concVal = np.genfromtxt("ConcLibrary.txt", delimiter = ",", usecols = 1)

corrVal = 1
stanConc = 850

plt.close("all")

lbound = 230
ubound = 245

stanData = np.genfromtxt("CS1.txt",delimiter = ",")

for i in range(0,len(stanData)-1):
stanData[i,5] = stanData[i,5]*stanConc

fileList = ["EppichMAPEP1.csv", "EppichMAPEP2.csv","EppichMAPEP3.csv"]

# Read in the data
```

```
for j in range(0,len(fileList)):

dataIn = np.genfromtxt(fileList[i], delimiter = ",")

fileBase = str(os.path.splitext(fileList[i])[0])
for i in range(0,len(concName)-1):
if concName[i] == fileBase:
index = i
if j == 0:
concData = np.zeros((len(dataIn),3))
concData[i,0] = dataIn[i,1]
concData[i,1] = concVal[index]*dataIn[i,5]*corrVal
concData[i,2] = concVal[index]*dataIn[i,6]
else:
tempData = np.zeros((len(dataIn),3))
for k in range(0,len(concData)-1):
tempData[k,0] = dataIn[k,1]
tempData[k,1] = concVal[index]*dataIn[k,5]*corrVal
tempData[k,2] = concVal[index]*dataIn[k,6]
concData = np.append(concData,tempData,axis=1)
plt.figure()
plt.plot(concData[:,],concData[:,1])
plt.xlim((lbound,ubound))
plt.xlabel("Mass (AMU)")
plt.ylabel("Concentration ($^{\mu g}/{mL}$)")
plt.grid("on")
plt.show()

figurePDF = fileBase + ".pdf"
figurePNG = fileBase + ".png"
plt.savefig(figurePDF, dpi=1000, format='pdf', orientation='landscape',
bbox_inches='tight')
plt.savefig(figurePNG, dpi=1000, format='png', orientation='landscape',
bbox_inches='tight')
```

**Combine Data Plotter by Sample**

```
import numpy as np
import matplotlib.pyplot as plt
plt.close("all")
n_groups = 4
height = 1.5E-1

## U-238
## MAPEP, STF, Trinitite, NYC
#name = "U-238"
```

#means_eppich = (284.3508147, 0.024680525, 0, 0.777072393)
#std_eppich = (1.156464607, 4.82904E-05, 0, 5.52E-03)
#
#means_modeppich = (337.3711118, 0.060162795, 0, 4.774538965)
#std_modeppich = (2.701613354, 0.000256848, 0, 0.021296332)
#
#means_lith = (29.26476219, 0.015915333, 0, 0.371645805)
#std_lith = (0.286110244, 0.000224963, 0, 0.001728572)
#
#means_maxwell = (60.47475945, 0.006734025, 0, 0.038885538)
#std_maxwell = (0.188712835, 4.16894E-05, 0, 7.31E-05)

## Ba
## MAPEP, STF, Trinitite, NYC
#name = "Ba"
#means_eppich = (2404.399933, 0, 0, 1.575550674)
#std_eppich = (2.648975387, 0, 0, 8.91E-03)
#
#means_modeppich = (1257.900747, 0, 0, 9.366860868)
#std_modeppich = (8.74467847, 0, 0, 0.02581968)
#
#means_lith = (56.16431259, 0, 0, 0.179530095)
#std_lith = (0.639329281, 0, 0, 0.001141014)
#
#means_maxwell = (44.94385896, 0, 0, 0.215388005)
#std_maxwell = (0.074106759, 0, 0, 4.28E-04)

## Ni
## MAPEP, STF, Trinitite, NYC
#name = "Ni"
#means_eppich = (801611.6917, 42.77451106, 0, 7.073928219)
#std_eppich = (3225.32926, 0.12146243, 0, 4.45E-02)
#
#means_modeppich = (8.38E+05, 90.3413856, 0, 32.64603628)
#std_modeppich = (5825.562756, 0.215972616, 0, 0.101327054)
#
#means_lith = (64218.05563, 44.04346732, 0, 178.9210009)
#std_lith = (321.7888108, 0.622727363, 0, 0.293930283)
#
#means_maxwell = (1826508.364, 849.9638471, 0, 58.326623)
#std_maxwell = (3682.310669, 3.139001796, 0, 4.57E-02)

## Fe
## MAPEP, STF, Trinitite, NYC
#name = "Fe"
#means_eppich = (0, 4.405913187, 0, 0.796350085)

```
#std_eppich = (0, 0.013557853, 0, 6.64E-03)
#
#means_modeppich = (0, 6.183203151, 0, 2.40117125)
#std_modeppich = (0, 0.017369401, 0, 0.007536074)
#
#means_lith = (0, 3.706784269, 0, 6.117670666)
#std_lith = (0, 0.052306003, 0, 0.02191475)
#
#means_maxwell = (0, 12.44990821, 0, 1.314150418)
#std_maxwell = (0, 0.02760913, 0, 1.14E-03)

# Al
# MAPEP, STF, Trinitite, NYC
name = "Al"
means_eppich = (0, 17.53354612, 18.53171346, 5.617168405)
std_eppich = (0, 0.059510733, 0.131918864, 3.40E-02)

means_modeppich = (0, 2.36E+01, 14.70724655, 18.10422393)
std_modeppich = (0, 0.063415259, 0.207274147, 0.036201525)

means_lith = (0, 6.066922839, 0.112371096, 46.60529142)
std_lith = (0, 0.085697644, 0.001497006, 0.564649832)

means_maxwell = (0, 0.995013506, 0.500043197, 0.190074398)
std_maxwell = (0, 0.004408886, 0.001110124, 4.72E-04)


fig, ax = plt.subplots()

index = np.arange(n_groups)
bar_width = 0.2

opacity = 0.4
error_config = {'ecolor': '0.3'}

rects1 = plt.bar(index, means_eppich, bar_width,
alpha=opacity,
color='b',
yerr=std_eppich,
error_kw=error_config,
label='Eppich')

rects2 = plt.bar(index + bar_width, means_modeppich, bar_width,
alpha=opacity,
color='r',
yerr=std_modeppich,
```

```
error_kw=error_config,
label='Modified Eppich')
rects3 = plt.bar(index + 2*bar_width, means_lith, bar_width,
alpha=opacity,
color='g',
yerr=std_lith,
error_kw=error_config,
label='Lithium')
rects4 = plt.bar(index + 3*bar_width, means_maxwell, bar_width,
alpha=opacity,
color='k',
yerr=std_maxwell,
error_kw=error_config,
label='Maxwell')



def autolabel1(rects):
# attach some text labels
for ii,rect in enumerate(rects1):
#height = 3 #rect.get_height()
plt.text(rect.get_x()+rect.get_width()/2., 1.02*height, '%s'%
(str(means_eppich[ii])+"$\pm$"+str(std_eppich[ii])),
ha='center', va='bottom',rotation='270')
autolabel1(rects1)

def autolabel2(rects):
# attach some text labels
for ii,rect in enumerate(rects2):
#height = 3 #rect.get_height()
plt.text(rect.get_x()+rect.get_width()/2., 1.02*height, '%s'%
(str(means_modeppich[ii])+"$\pm$"+str(std_modeppich[ii])),
ha='center', va='bottom',rotation='270')
autolabel2(rects2)

def autolabel3(rects):
# attach some text labels
for ii,rect in enumerate(rects3):
#height = 3 #rect.get_height()
plt.text(rect.get_x()+rect.get_width()/2., 1.02*height, '%s'%
(str(means_lith[ii])+"$\pm$"+str(std_lith[ii])),
ha='center', va='bottom',rotation='270')
autolabel3(rects3)

def autolabel4(rects):
# attach some text labels
```

```
for ii,rect in enumerate(rects4):
#height = 3 #rect.get_height()
plt.text(rect.get_x()+rect.get_width()/2., 1.02*height, '%s'%
(str(means_maxwell[ii])+"$\pm$"+str(std_maxwell[ii])),
ha='center', va='bottom',rotation='270')
autolabel4(rects4)

title = 'Method Yield by Sample for '+ name

ax.set_yscale("log", nonposx="clip")
plt.xlabel('Sample')
plt.ylabel('Fraction of Ideal')
plt.title(title)
plt.xticks(index + 2*bar_width, ('MAPEP', 'STF', 'Trinitite', 'NYC'))
#plt.ylim((0,100))
plt.xlim((-0.1,3.9))
plt.legend(loc = 1,prop={'size':12})

plt.tight_layout()
plt.show()

# Generate the figure names
figurePDF = "SampleByMethod_"+name+".pdf"
figurePNG = "SampleByMethod_"+name+".png"
# Save the files as PDF and PNG
plt.savefig(figurePDF, dpi=1000, format='pdf', orientation='landscape',
bbox_inches='tight')
plt.savefig(figurePNG, dpi=1000, format='png', orientation='landscape',
bbox_inches='tight')
```

**Combine Data Plotter by Method**

```
import numpy as np
import matplotlib.pyplot as plt

plt.close("all")

n_groups = 4

height = 1.5E-3

## Modified Eppich, Eppich, Lithium, Maxwell
#name = "Al"
#means_trin = (14.70724655,18.5317135, 0.112371096, 0.500043197)
#std_trin = (0.207274147, 0.131918864, 0.001497006, 0.001110124)
#
```

#means_stf = (23.64772654, 17.53354612, 6.066922839, 0.995013506)
#std_stf = (0.063415259, 0.059510733, 0.085697644, 0.004408886)
#
#means_nyc = (18.10422393, 5.617168405, 46.60529142, 0.190074398)
#std_nyc = (0.036201525, 3.40E-02, 0.564649832, 4.72E-04)
#
#means_mapep = (0, 0, 0, 0)
#std_mapep = (0, 0, 0, 0)

## Modified Eppich, Eppich, Lithium, Maxwell
#name = "Fe"
#means_trin = (0, 0, 0, 0)
#std_trin = (0, 0, 0, 0)
#
#means_stf = (6.183203151, 4.405913187, 3.706784269, 12.44990821)
#std_stf = (0.017369401, 0.013557853, 0.052306003, 0.02760913)
#
#means_nyc = (2.40117125, 0.796350085, 6.117670666, 1.314150418)
#std_nyc = (0.007536074, 6.64E-03, 0.02191475, 1.14E-03)
#
#means_mapep = (0, 0, 0, 0)
#std_mapep = (0, 0, 0, 0)

## Modified Eppich, Eppich, Lithium, Maxwell
#name = "Ni"
#means_trin = (0, 0, 0, 0)
#std_trin = (0, 0, 0, 0)
#
#means_stf = (90.3413856, 42.77451106, 44.04346732, 849.9638471)
#std_stf = (0.215972616, 0.12146243, 0.622727363, 3.139001796)
#
#means_nyc = (32.64603628, 7.073928219, 178.9210009, 58.326623)
#std_nyc = (0.101327054, 4.45E-02, 0.293930283, 4.57E-02)
#
#means_mapep = (8.38E+05, 801611.6917, 64218.05563, 1826508.364)
#std_mapep = (5825.562756, 3225.32926, 321.7888108, 3682.310669)

## Modified Eppich, Eppich, Lithium, Maxwell
#name = "Ba"
#means_trin = (0, 0, 0, 0)
#std_trin = (0, 0, 0, 0)
#
#means_stf = (0, 0, 0, 0)
#std_stf = (0, 0, 0, 0)
#
#means_nyc = (9.366860868, 1.575550674, 0.179530095, 0.215388005)

```
#std_nyc = (0.02581968, 8.91E-03, 0.001141014, 4.28E-04)
#
#means_mapep = (1257.900747, 2404.399933, 56.16431259, 44.94385896)
#std_mapep = (8.74467847, 2.648975387, 0.639329281, 0.074106759)

# Modified Eppich, Eppich, Lithium, Maxwell
name = "U-238"
means_trin = (0, 0, 0, 0)
std_trin = (0, 0, 0, 0)

means_stf = (0.0601628, 0.0246805, 0.0159153, 6.73402E-03)
std_stf = (0.000256848, 4.82904E-05, 0.000224963, 4.16894E-05)

means_nyc = (4.774539, 0.7770724, 0.3716458, 0.0388855)
std_nyc = (0.021296332, 5.52E-03, 0.001728572, 7.31E-05)

means_mapep = (337.3711118, 284.3508147, 29.2647622, 60.4747594)
std_mapep = (2.701613354, 1.156464607, 0.286110244, 0.188712835)


fig, ax = plt.subplots()

index = np.arange(n_groups)
bar_width = 0.2

opacity = 0.4
error_config = {'ecolor': '0.3'}

rects1 = plt.bar(index, means_mapep, bar_width,
alpha=opacity,
color='b',
yerr=std_mapep,
error_kw=error_config,
label='MAPEP')

rects2 = plt.bar(index + bar_width, means_trin, bar_width,
alpha=opacity,
color='r',
yerr=std_trin,
error_kw=error_config,
label='Trinitite')
rects3 = plt.bar(index + 2*bar_width, means_nyc, bar_width,
alpha=opacity,
color='g',
yerr=std_nyc,
error_kw=error_config,
```

```
label='NYC')
rects4 = plt.bar(index + 3*bar_width, means_stf, bar_width,
alpha=opacity,
color='k',
yerr=std_stf,
error_kw=error_config,
label='STF')

def autolabel1(rects):
# attach some text labels
for ii,rect in enumerate(rects1):
#height = 3 #rect.get_height()
plt.text(rect.get_x()+rect.get_width()/2., 1.02*height, '%s'%
(str(means_mapep[ii])+"$\pm$"+str(std_mapep[ii])),
ha='center', va='bottom',rotation='270')
autolabel1(rects1)

def autolabel2(rects):
# attach some text labels
for ii,rect in enumerate(rects2):
#height = 3 #rect.get_height()
plt.text(rect.get_x()+rect.get_width()/2., 1.02*height, '%s'%
(str(means_trin[ii])+"$\pm$"+str(std_trin[ii])),
ha='center', va='bottom',rotation='270')
autolabel2(rects2)

def autolabel3(rects):
# attach some text labels
for ii,rect in enumerate(rects3):
#height = 3 #rect.get_height()
plt.text(rect.get_x()+rect.get_width()/2., 1.02*height, '%s'%
(str(means_nyc[ii])+"$\pm$"+str(std_nyc[ii])),
ha='center', va='bottom',rotation='270')
autolabel3(rects3)

def autolabel4(rects):
# attach some text labels
for ii,rect in enumerate(rects4):
#height = 3 #rect.get_height()
plt.text(rect.get_x()+rect.get_width()/2., 1.02*height, '%s'%
(str(means_stf[ii])+"$\pm$"+str(std_stf[ii])),
ha='center', va='bottom',rotation='270')
autolabel4(rects4)

title = 'Method Yield by Sample for '+ name
```

```
ax.set_yscale("log", nonposx="clip")
plt.xlabel('Method')
plt.ylabel('Fraction of Ideal')
plt.title(title)
plt.xticks(index + 2*bar_width, ('Modified Eppich', 'Eppich', 'Lithium', 'Maxwell'))
#plt.ylim((0,100))
plt.xlim((-0.1,3.9))
plt.legend(loc = 1,prop={'size':12})

plt.tight_layout()
plt.show()

## Generate the figure names
figurePDF = "MethodBySample_"+name+".pdf"
figurePNG = "MethodBySample_"+name+".png"
## Save the files as PDF and PNG
plt.savefig(figurePDF, dpi=1000, format='pdf', orientation='landscape',
bbox_inches='tight')
plt.savefig(figurePNG, dpi=1000, format='png', orientation='landscape',
bbox_inches='tight')
```

# Appendix C - Presentation

# Dissolution of Melt Glass for Destructive Assay

Jonathan A. Gill
Masters Thesis Presentation
14 October 2015

THE UNIVERSITY OF TENNESSEE KNOXVILLE

## Overview

- Impetus
- BLUF
- Approach
  - Methods
  - Samples
- Fallout
- Deductions
- Yet to come
- Acknowledgements






# Impetus

- Nuclear Forensics and Attribution Act (NFAA) in 2010
- National Nuclear Security Agency (NNSA) est. 2000
- Need for liquid phase melt glass
- Enable separations of lanthanides, actinides, and transition metals
- Rapid and Resilient to Sample Variation





# How the Real World looks in the Lab



Application

Event

Environmental Knowns

Post Material Analysis

Regional Production Comparison



Attribution

Laboratory









# BLUF

- The Modified Eppich Method or the Auxier Method is the best for separating heavier elements:
  - Partially Sample Independent
  - Best yield on Barium and Uranium
- The Lithium Procedure and Maxwell Procedure may preform well if supernates can be extracted though this will likely compromise elemental ratios.



# Approach - Methods and Samples

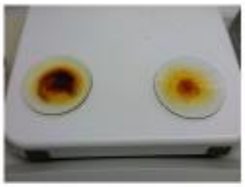
| Methods / Samples | Eppich Method | Auxier Method | Lithium Method | Maxwell Method |
|---|---|---|---|---|
| Trinitite | Run 1<br>2<br>3 | Run 1<br>2<br>3* | Run 1<br>2<br>3 | Run 1<br>2<br>3 |
| STF Gad Mod 1 | Run 1<br>2<br>3 | Run 1<br>2<br>3 | Run 1<br>2<br>3 | Run 1<br>2<br>3 |
| NYC IND 1 | Run 1<br>2<br>3 | Run 1*<br>2<br>3 | Run 1<br>2<br>3 | Run 1<br>2<br>3 |
| MAPEP MaS 32 | Run 1<br>2*<br>3 | Run 1<br>2<br>3 | Run 1<br>2<br>3 | Run 1<br>2<br>3 |



* Run was significantly inconsistent or failed

# Approach - Eppich Method









$X + 2.5HNO_3 + 1HF \rightarrow XF_a + X(NO_3)_b$

- Evaporated in Teflon for 24 hours @ 80 ºC

$XF_a + X(NO_3)_b + 5HNO_3 + 2HClO_4 \rightarrow X(ClO_4)_c + X(NO_3)_d$

- Evaporated in Teflon @ 225 ºC

$X(ClO_4)_c + X(NO_3)_d + HCl \rightarrow XCl_e$

- Evaporated in Teflon @ 225 ºC

Process Time: ~28 hrs

$XCl_e + HCl\ (3\ M) \rightarrow XCl_f$

- Evaporated in Teflon @ 225 ºC

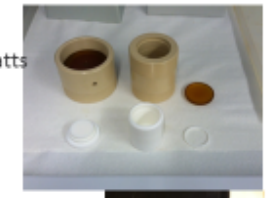
* Reactive agents in concentrate form unless otherwise listed
** a, b, c, d, e, f represent stoichiometric coefficient

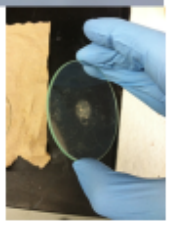
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# Approach – Auxier Method

$X + 2HNO_3 + 5HF \rightarrow XF_a + X(NO_3)_b$

- Heated 32 mL Parr Microwave Bomb, 35 sec @ 700 watts
- Cools for 20-30 mins
- Evaporated in Teflon @ 225 ºC





$XF_a + X(NO_3)_b + 5HNO_3 + 2HClO_4 \rightarrow X(ClO_4)_c + X(NO_3)_d$

- Evaporated in Teflon @ 225 ºC

Process Time: ~4-6 hrs

$X(ClO_4)_c + X(NO_3)_d + HCl \rightarrow XCl_e$

- Evaporated in Teflon @ 225 ºC

* Reactive agents in concentrate form unless otherwise listed
** a, b, c, d, e represent stoichiometric coefficient

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# Approach - Lithium Method



Fusion X w/ 1:1 $Li_2B_4O_7$ & $LiBO_2$ + LiBr → $Li_aB_bO_c + XB_d$

- Loaded into a 95:5 platinum and gold crucible of 25 mL crucible
- Dry at @ 100 ºC for 60 sec
- Heat @ 1000 ºC for 60 sec, cool for 60 sec
- Heat for 180 sec, cool for 45-60 sec

Process Time: ~25 hrs

$Li_aB_bO_c + XB_d + HNO_3$ (6 M) + HF (3 M) → $XF_e + X(NO_3)_f$

- Dry at @ 80 ºC for 24 hrs



* Reactive agents in concentrate form unless otherwise listed
** a, b, c, d, e, f represent stoichiometric coefficient



# Approach - Maxwell Method



Fusion X w/ NaOH → $X(OH)_a$ + Na

- Load a 35 mL high purity graphite crucible w/ lid
- Heat @ 600 ºC for 20 min, cooled for 10-20 min





$X(OH)_a + HNO_3$ → $X(NO_3)_b$

- High purity water and Nitric Acid is used to transfer out of the crucible



Process Time: ~20 hrs

$X(NO_3)_b$ + HCl → XCl

- Evaporate at 200 ºC



* Reactive agents in concentrate form unless otherwise listed
** a, b represent stoichiometric coefficient

# Approach - Trinitite



- Approximately 15 grams of trinitite was selected and aggregated into a sample body
- Predictions for elemental fractions come from



| Element | Fraction |
|---|---|
| Si | 18.03% |
| Al | 45.37% |
| Ca | 4.13% |
| Fe | 0.92% |
| Mg | 0.41% |
| Na | 0.55% |
| K | 2.78% |
| Ti | 0.16% |
| O | 27.64% |



# Approach - STF Gadget Mod 1



- Mix was prepared by melting for 30 minutes at 1500 ºC
- Mod 1 Gadget uses a realistic estimate of the uranium tamper on gadget but omits the Pu-239/240 primary fuel.
- 2 samples of 1 gram mass were aggragated

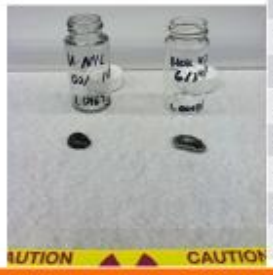


| Element | Fraction |
|---|---|
| Si | 26.09% |
| Al | 8.5% |
| Ca | 7.49% |
| Fe | 1.64% |
| Mg | 0.62% |
| Na | 1% |
| K | 3.94% |
| Ti | 0.31% |
| O | 49.6% |
| U | 0.2% |

# Approach - NYC IND 1

- Utilizes Giminaro's *et. al.* method for estimating downtown Manhattan's composition from a 1 kt weapon.
- Mix was prepared by melting for 30 minutes at 1550 ºC
- Aggregated 4 1-grams samples





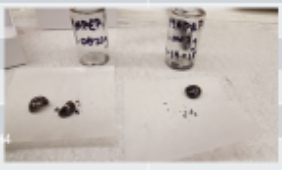
| Element | Fraction |
|---|---|
| Si | 52.22% |
| Al | 13.08% |
| Ca | 5.97% |
| Fe | 8.88% |
| Mg | 2.41% |
| Na | 2.77% |
| K | 3.39% |
| Ti | 0.58% |
| O | 0.55% |
| Mn | 11% |
| S | 0.06% |
| P | 0.09% |
| N | 0.01% |
| C | 0.01% |
| Ba | 0.05% |
| U | 0.56% |



# Approach – MAPEP MaS32

- Soil Analyte Standard
- Produced by Radiological and Environmental Sciences Laboratory
- Melted at 1500 ºC for 30 minutes in 1 gram aliquots
- Aggregated 4 1-grams samples
- Incomplete List of Elements

| Analyte | Ref Value (mg/kg) | Analyte | Ref Value (Bq/kg) |
|---|---|---|---|
| Antimony | 93.4 | Americium-241 | 85.5 |
| Arsenic | 69.4 | Cesium-134 | 622 |
| Barium | 1078 | Cobalt-57 | 1116 |
| Beryllium | 54.3 | Cobalt-60 | 779 |
| Cadmium | 7.84 | Iron-55 | 680 |
| Chromium | 147 | Manganese-54 | 1009 |
| Cobalt | 293 | Nickel-63 | 980 |
| Copper | 192 | Plutonium-238 | 0.48 |
| Lead | 57.4 | Plutonium-239/240 | 58.6 |
| Mercury | 0.155 | Potassium-40 | 824 |
| Nickel | 218 | Strontium-90 | 858 |
| Selenium | 20 | Technetium-99 | 589 |
| Silver | 69.6 | Uranium-234/233 | 89 |
| Technetium-99 | 1.9E-5 | Uranium-238 | 259 |
| Thallium | 157 | Zinc-65 | 541 |
| Uranium-235 | 0.0670 | | |
| Uranium-238 | 20.8 | | |
| Uranium-Total | 20.9 | | |
| Vanadium | 187 | | |
| Zinc | 628 | | |



# Instrument and Method



- Optimass 9500 ICP-TOF-MS (Inductively Coupled Plasma time-of-flight mass spectrometer)
- Mass Range: 0.163 amu to 264.313 amu
- Resolution: ~0.032 amu
  - Each aliquot was measured in triplicate
- Samples
  - Every sample introduced is dissolved/suspended in 2% $HNO_3$





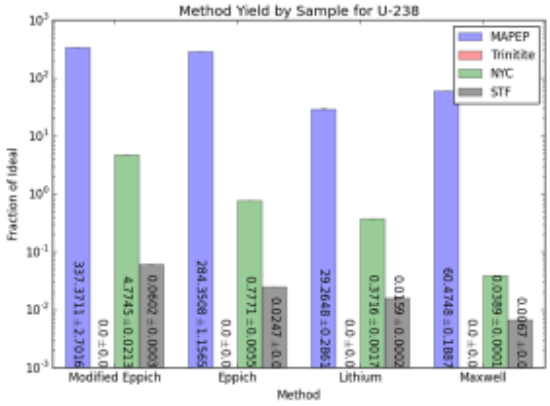


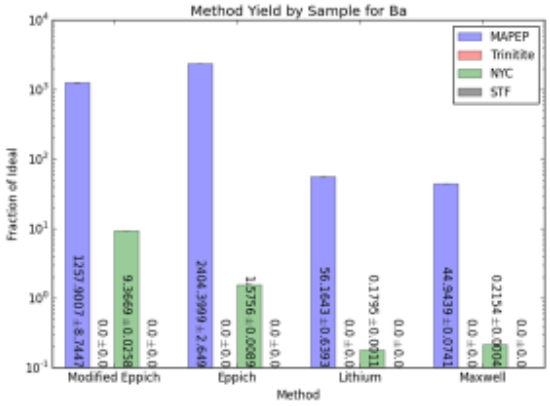
# Approach – Coding: The Matt Cook Solution

- Python Code used for comparison as GBC code is not adequate.
- Analyzed internal calibration standards with a first order polynomial fit.
- A peak is input by the user and given a 0.1 amu width, then peak area is integrated to find the total signal intensity for the given isotope.
- Curve is applied to peaks in 3 replicates per atomic mass unit (amu) bin and averaged with error.
- The average is now a concentration and divided by the total concentration of the diluted sample.
- Ratio is compared to the ratio of ideal elemental mass per total mass of the sample prior to dissolution.



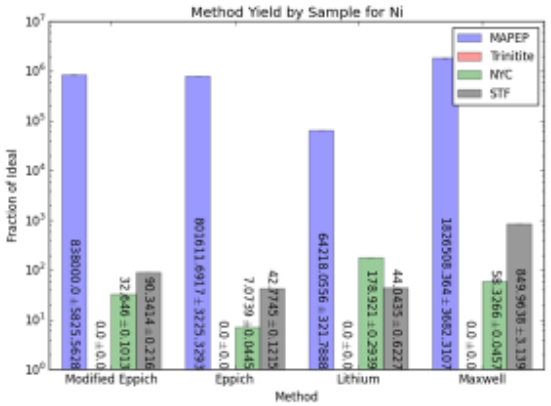
# Fallout - By Sample Uranium and Barium

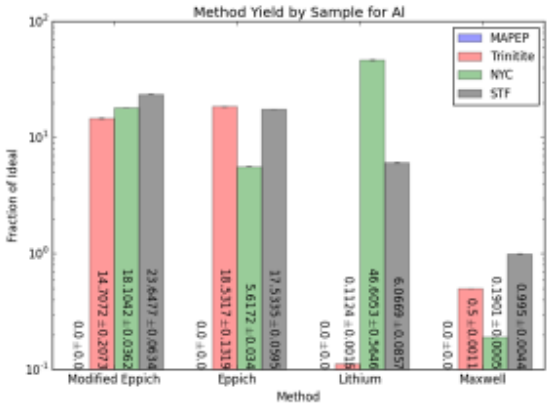


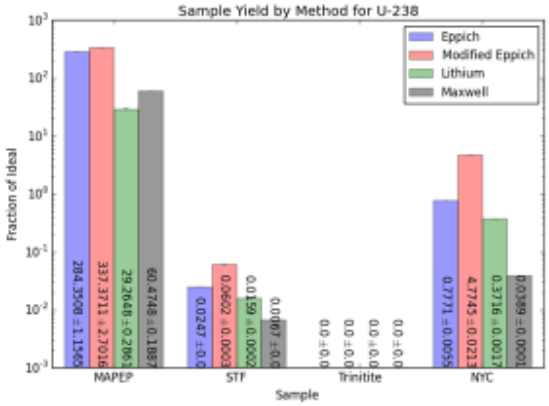


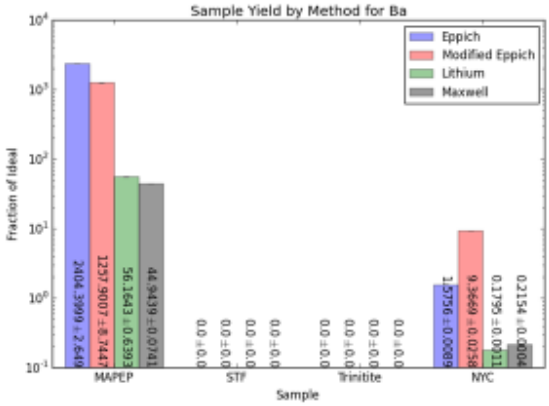

# Fallout - By Sample Lighter Elements



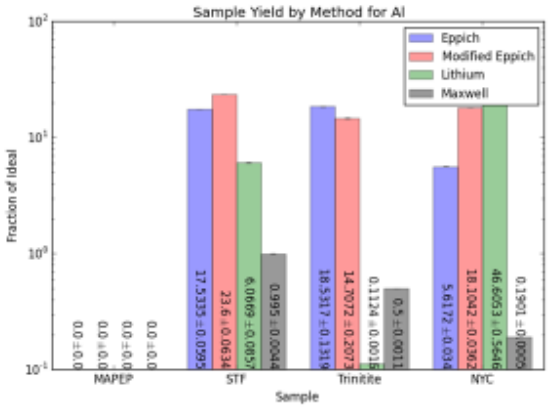
# Fallout - By Method Uranium and Barium

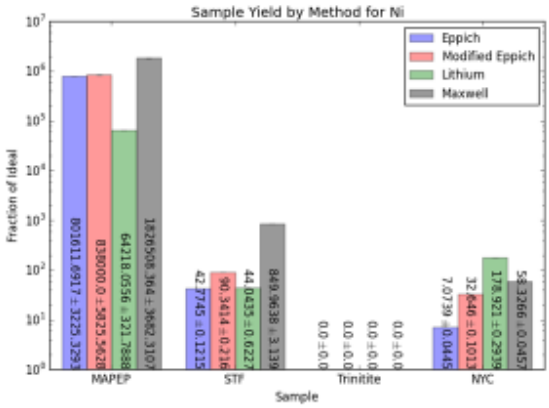





# Fallout - By Method Lighter Elements



## Deductions

- Auxier Method is the fastest with the best yields for fissile materials and elements around daughter product masses
- Still not suitable for broad acceptance
- ICP-TOF-MS not suitable for these modifications to fusion methods
- No method works across all elements
- MAPEP was not a suitable standard for comparison in this experiment



## Yet to come

- Test other fusion methods like bisulphite techniques
- Continue to use Auxier Method on new urban melts
- Test Lithium and Maxwell procedures with centrifuging
- Dope STF and Urban with other elements consistent with uranium or plutonium fission daughters to check dissolution, i.e. Nd, Cs, Pd, Ru, and Zr.

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Andy Giminaro (UT Graduate Student)
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Dr. John Auxier (UT Research Professor)





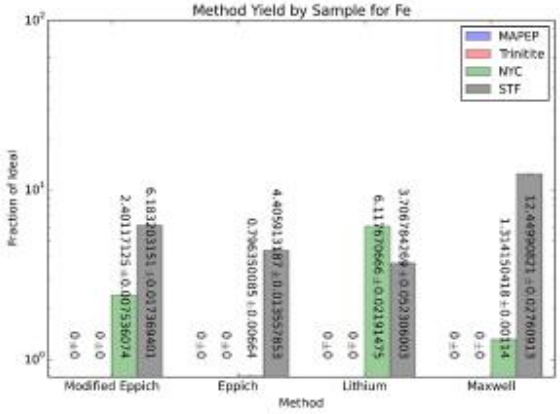

# Backup Slides

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## Approach - Methods and Samples

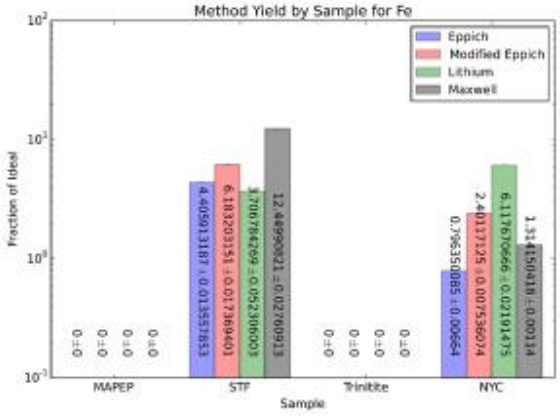
- Methods
  - Eppich Method
  - Modified Eppich Method or Auxier Method
  - Lithium Method or Laval Method
  - Maxwell Method
- Samples
  - Trinitite
  - STF Gadget Mod 1
  - NYC IND 1
  - MAPEP MaS32

# Fallout – By Method Lighter Elements






# Fallout – By Sample Lighter Elements



# Vita

Jonathan Gill graduated from the United States Military Academy in 2005 with a Bachelor of Science in Nuclear Engineering. Following graduation, Jonathan served honorably for 8 years as an Army Engineer Officer. During this time he earned a Masters of Science in Engineering Management from the Missouri University of Science and Technology. In 2014, he enrolled at the University of Tennessee to pursue his Doctorate and a concurrent Masters of Science in Nuclear Engineering. Upon completion of his Master's degree under Dr. Howard L. Hall, Jon will pursue his Doctorate instructed by Dr. Howard Hall and under the direct supervision of Dr. Kevin Y. Hour at the Lynchburg Technology Center, which is owned and operated by Babcox and Wilcox. His interests in the nuclear field focus on nuclear security applications for special nuclear materials and post-detonation analysis. Jon's professional goals are to work for the United States on technical solutions to nuclear security problems.