# Assessing automated image analysis and compound-specific stable isotope signatures for small arms propellant differentiation and potential brand identification.

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**Summary**

Smalls arms propellants (SAP) are readily accessible and cost-effective materials that firearms enthusiasts can acquire for the legitimate assembly of ammunition. Unfortunately, the ease of access and low cost of these materials is advantageous for their utilization in the construction of improvised explosive devices (IEDs). Typically, the SAP charge is loaded into a metal pipe (commonly steel) and sealed with screw-fit end caps. These devices are termed "pipe bombs" and are the most common IEDs in the United States. Two recent high profile domestic terrorist attacks using IEDs (Boston Marathon Bombing and NY/NJ attempted bombings) further demonstrate their continued usage. Thus, there is a need to develop robust metrics for the characterization of propellants that are used as explosives as well as for comparisons between known and recovered explosive residues. The goals of the research are to investigate the utility of high-throughput, low-cost quantitative automated image analysis, additive profiling and compound-specific stable isotope signatures of SAP for potential brand identification and sample discrimination. If found to be successful, the metrics will provide advanced methods for the examination of small arms propellants recovered from pre- and post-blast improvised explosive devices. The automated image analysis method is appealing since it is non-destructive and relies upon low-cost analytical equipment and instrumentation. A rigorous assessment of the existing GC/MS additive profiling methodology will be achieved as well as a better understanding of its role in SAP examination. The measurement of compound-specific stable isotope signatures is considered a final tier analysis, which may provide highly discriminatory information that could constrain geographic/ manufacturer-specific characteristics. The research is the first of its kind to combine uncorrelated data sets from complementary analytical methods for the characterization of SAP in forensic and intelligence casework.

**Project Design and Implementation**

The focus of the research is threefold:

1. Develop a high-throughput automated image analysis method for increasing the speed, accuracy, reproducibility, and amount of information for the characterization of small arms propellants.
2. Assess the strengths and limitations of additive profiling by GC/MS.
3. Measure the oxygen, carbon, and nitrogen stable isotope signatures of purified nitrocellulose.
4. Investigate the discriminatory information that can be gleaned from "data fusion," the combination of three uncorrelated data streams: micromorphometry, stable isotope signatures, and additive profiles to assess the potential for SAP discrimination and potential brand identification.

The workflow for the research consisted of four phases and are briefly summarized in the following subsections.

*Phase 1. Small arms propellant sample acquisition*

To obtain a robust assessment of the within and between sample variation of SAPs, the following sample collection plan was utilized. A review of Propellant Profiles 5$^{th}$ Edition, the authoritative reference for the "hand-loading" community shows that there are approximately 220 commonly available propellants available for purchase by the consumer. This number is high, it does however show that it is possible to acquire a significant number of samples that may provide a good representative sampling of the propellants *currently* available for bomb-makers. Consequently, for this project purchased approximately 204 one-pound (smallest unit of sale)

containers from different manufacturers and brands of SAP. The purchased samples were acquired in order to produce a data set that spans all major and minor product lines as well as from as many different countries as possible.

For five additional brands, we obtained 5 replicate containers from the same manufacturer's lot. These samples provided valuable information concerning within lot sample variation. Next for these five selected brands, 5 samples were collected from different manufacturer's lots. These samples provided a robust assessment of between lot sample variability. In total approximately 200 small arms propellant samples were purchased.

*Phase 2: Automated micromorphometry*

Manual methods for SAP micrometry have been previously described [1]. These methods while accurate and very useful for limiting the number of potential brands for a given unknown SAP, they are relatively time consuming and tedious. In addition, the manual methods do not efficiently take advantage of all information that can be obtained from SAP digital images. The research in this study investigated the utility of digital image analysis software to generate quantitative objective metrics for the characterization and differentiation of SAP samples. For this research we used ImageJ (FIJI), an open-source cross-platform software package available to download free of charge from the National Institutes of Health [2]. This package was utilized to minimize costs which may otherwise inhibit the adoption of the proposed technology in crime laboratories. In addition, the code is open source which is advantageous, allowing caseworkers the ability to customize input and output parameters to meet their needs.

*Phase 3. Additive Profiling*

Gas chromatography- mass spectrometry (GC/MS) is a routine instrumental method for the characterization of SAP. For this phase of the research, portions of each of the ~200 SAP samples were analyzed for their additive profiles. Samples were run in triplicate, thus approximately 600 samples will be characterized in terms of their additive profiles.

Phase 4: Compound –Specific Isotope Ratio Mass Spectrometry (IRMS)

This phase of the research characterized carbon, nitrogen, and oxygen stable isotopic compositions measured for nitrocellulose, the primary component of most small arms propellant. The analysis of nitrocellulose is particularly appealing since it is the major component of most propellants and it is currently not being analyzed in a forensic setting. The stable isotopes of nitrocellulose are also very interesting since they *may* retain their primary isotopic signatures, which may constrain geographic origin.

**Expected applicability of the research**

The research meets several criteria under the category of development and interpretation for the forensic analysis of SAP. The first is that it has the potential to provide caseworkers with a high-throughput, cost effective, quantitative, non-destructive automated method for the characterization of small arms propellants. The second outcome for the research is a detailed analysis of the strengths and limitations of additive profiling for the characterization of SAP. This method of analysis widely utilized in the forensic science community. The third is that this research will provide a robust assessment of the utility of nitrocellulose stable isotope signatures for the discrimination of SAP. Though not many state and local crime laboratories have this

capability, larger federal and small commercial laboratories could be contracted if a case is of high enough importance to warrant such measurements. This technology may provide higher levels of discrimination as well as increased confidence in obtaining brand identification, a primary goal for these types of investigations. Lastly, this research will produce a unique data set in which three complementary techniques are used on the same analytical samples. This will provide caseworkers with a thorough foundation on which they can shape their laboratory's analytical workflow.

In addition, the techniques and capabilities have the potential to be transitioned to shooting scene reconstruction, where unburned and partially burned propellant can be deposited onto primary and intermediate targets (i.e., clothing). These particles from the target could potentially be compared to particles that are inside the barrel of a firearm or in the ejected cartridge case from a fired round of ammunition.

**Outcomes**

*Phase 1 sample collection*

Two hundred and four (204) samples of smokeless propellant were acquired from local firearms dealers, gun shows, and an online shooting supply retailer (see attached spreadsheet with sample details). Of these, there are 154 unique brands from nine different distributors (Table 1). The morphology for each of the propellant samples was placed into one of the following four descriptive classes: spherical, flattened spherical, cylindrical, and flake (Figure 1). These categories follow the definitions from the Sporting Arms and Ammunition Manufacturers' Institute [3]. These four classes are easily recognizable which minimizes potential variability in classification by different analysts.

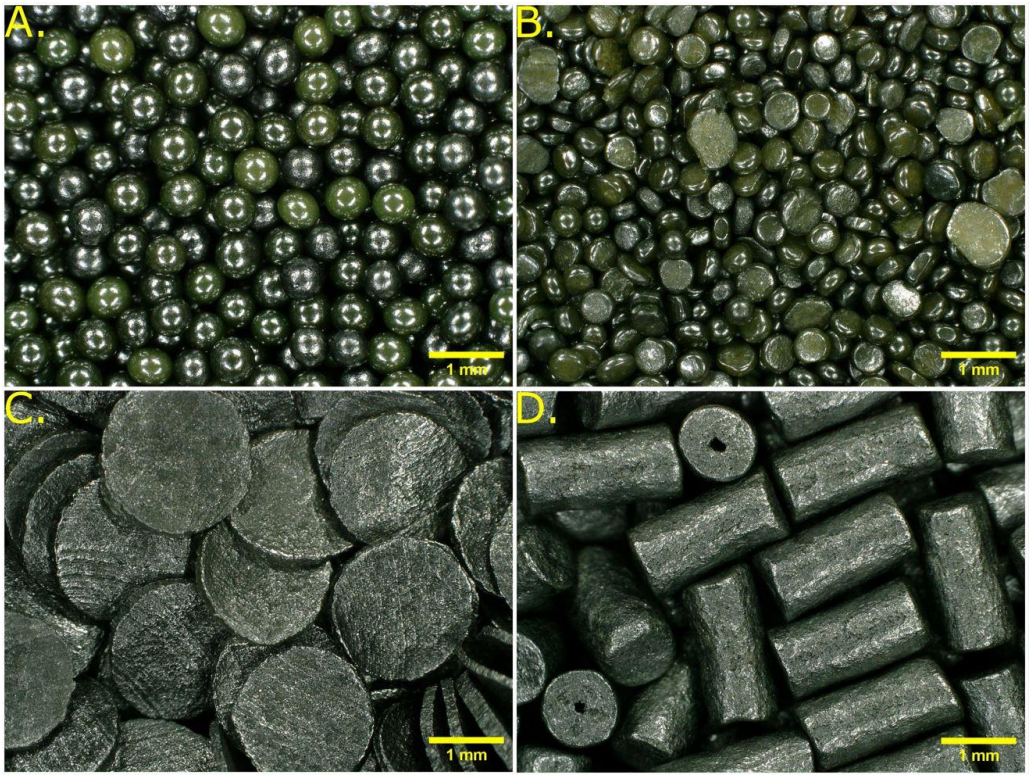


Figure 1. Photomicrographs showing the smokeless powder granule morphology descriptions used in this study: (a.) spherical (Hodgdon H380), (b.) flattened spherical (Western Accurate No. 9), (c.) flake (Hodgdon HI-Skor 800-X), and (d.) cylindrical (IMR 4831).

In addition, nine-to-ten replicate samples from five brands (H335, BL-C(2), Bullseye, Red Dot, and Reloader 23) were purchased to assess intra- and inter-lot variability. Note that duplicate samples from five brands (4064, LT-32, Reloader 19, Reloader 22, and Reloader 7) were inadvertently purchased. These samples were included to provide additional information about potential inter-lot variation.

**Table 1. Summary table showing the distribution of smokeless powder samples used for this study.**

| Distributor | Total Samples | Flake | Spherical | Flattened Spherical | Cylindrical |
|---|---|---|---|---|---|
| Alliant | 65 | 36 | 0 | 4 | 25 |
| Hodgdon | 54 | 6 | 1 | 34 | 13 |
| IMR | 22 | 4 | 0 | 0 | 18 |
| Lovex | 2 | 0 | 1 | 1 | 0 |
| Norma | 2 | 0 | 0 | 0 | 2 |
| Ramshot | 1 | 0 | 1 | 0 | 0 |
| VihtaVouri | 14 | 0 | 0 | 0 | 14 |
| Western | 33 | 0 | 7 | 15 | 11 |
| Winchester | 11 | 0 | 0 | 11 | 0 |
| **Total** | 204 | 46 | 10 | 65 | 83 |

*Phase 2 micromorphometry*

Each of the sample containers was shaken to mix the propellants. An aliquot was then taken from each bottle by gently pouring a portion of the propellant into clean disposable Petri dishes. The samples were then examined using stereomicroscopy at ~5-50x magnification. Photomicrographs of each sample were captured at 20x and 50x magnification. Individual propellant granules were placed in a grid pattern onto new featureless and colorless 2.54 x 2.54 cm squares of restickable mounting tabs (3M, St. Paul, MN, USA). This substrate was chosen because it secures the granules in place and provides a transparent background which was needed for image capture. The granules were placed onto the mount so that they did not touch each other. Some manufacturers include colored granules in their products to help with identification (e.g., Alliant Red Dot, Blue Dot, Green Dot).

The sample preparations were illuminated with transmitted light by placing them on a light table. This method of illumination produced images with high contrast between the granules and the colorless and featureless background. Because the images were captured in this manner, no post-image capture processing (e.g., watershed, edge sharpening, brightness/ contrast adjustment), with the exception of cropping, was performed prior to image analysis. The preparations were imaged using a consumer-grade DSLR camera (Canon EOS 70D) held secure using a copy stand. The images were captured at the largest image size (5472 x 3648) and ISO was set to 100 (lowest value). The height of the camera was adjusted so the preparations filled the field-of-view. The camera parameters which included aperture, shutter speed, and focus were manually adjusted. Exposures were captured using a time-delayed shutter release minimize camera vibration. One image for each preparation was collected; however, in some instances, multiple preparations were needed to capture a sufficient number of granules for a given sample.

The images of the preparations were analyzed using customized code written in FIJI, an open-source image processing software package [2]. Briefly the captured images were converted to 8-bit greyscale, binarized, and the particles were detected and measured using the base functions in FIJI. For each granule eight parameters were determined (see reference ImageJ website for descriptions of the parameters [4]). These parameters can be divided into either size- (major ellipse diameter, minor ellipse diameter, area, and perimeter) or shape-dependent (aspect ratio, circularity, roundness, and solidity) metrics. During a pilot study for this research, it was noted that orientation of granules (e.g., cylindrical) could change the meaning of the length and width measurements. These could be interchanged depending on the orientation of the granule. To circumvent this problem, we measured the length of the major and minor axes of the best fit

ellipse (for each particle). This measurement is independent of orientation, and thus chosen for this research.

The large multi-dimensional dataset, which consists of approximately 680,000 measurements, was processed using customized code written in R (version 4.2.1), an open-source statistical analysis software package [5,6]. The data were analyzed by linear discriminant analysis (LDA) using the MASS package [7]. Prior to statistical analysis, the data were scaled (mean = 0, standard derivation = 1) and each class (brand) was given equal prior probability. LDA was performed separately for each of the four granule classes. This statistical method was first "trained" by randomly selecting a portion (80% for each sample) of the data in which the classes (brands) are known. The algorithm calculates discriminant functions that attempt to minimize within group variation while at the same time, trying to maximize between group variation. Next the remaining 20% of the data (for each sample) is treated as if the source was unknown and is associated to the class (brand) which had the closest distance in 8-dimensional space. The distance was calculated by summing the posterior probabilities for the test samples relative to each of the brands in the training set.

To better understand how the samples were being separated by LDA, the discrimination potential for each variable was calculated. This was calculated by summing the absolute value of the product of the LD proportions and the loadings (coefficients) for each variable across all eight LD functions and then normalizing to 100%. The results of this analysis are shown in Table 3. The variables that are responsible for the largest overall contribution to sample discrimination are area, perimeter, and the major and minor axes of the best fit ellipsoid. These results demonstrate that across all granule morphologic classes, the size-dependent variables have a greater impact on sample discrimination than the shape-dependent variables.

*Classification of the "test" dataset*

On the basis of the shortest distance in 8-dimensional space, the algorithm associated 171 of the 204 propellant samples to the correct classification (brand). This resulted in an overall success rate of 84%. However, the success rate is dependent on the granule morphologic class (Table 4). The accuracy for both cylindrical and spherical propellants was 90%. The success rate for flake propellants was slightly lower at 85% and that for flattened spherical propellants was 74%. For the 33 misclassified samples (Table 5), 21 of them were associated to brands from the same distributor as themselves.

For the 33 misclassified samples, the position (rank order) for the correct class (brand) was evaluated. The correct rank for 20 of the samples was in the second position, 9 samples in the third position, and 2 samples in each of the fifth and sixth positions. In total 99% of the samples in the study were correctly classified within the top 5 rank positions.

*Intra- and inter-lot variability study*

To better understand the utility of micromorphometry to identify the brand of SAP, we investigated the potential variation that may be present both within and between manufacturing lots of five different SAP brands. The selected brands include: Hodgdon H335 and BL-C(2) which are both flattened spherical, Alliant Bullseye and Red Dot which are flake, and Alliant Reloader 23 is a cylindrical-type propellant. For each brand nine or ten replicate bottles were purchased that were from either the same or different lots (see Supplemental information for details). The individual replicates were treated as separate classes for LDA. The results from this analysis are shown in Table 6. Twenty-two of the of the 49 samples were associated with their specific source (i.e., sample SP-007.B was classified as SP-007.B), while 23 were

associated with the same brand. For these 23 samples that were correctly associated at the brand-level, 11 of them were associated with different replicate samples within the same lot (i.e., sample SP-007.G was classified as SP-007.H). Four samples were misclassified as being from different brands. All four of these samples were flattened spherical-type propellants.

As previously mentioned, duplicate samples from five brands (4064, LT-32, Reloader 19, Reloader 22, and Reloader 7) were inadvertently purchased. These pairs of samples were manufactured in different lots. Nine of these samples were successfully associated with their specific source. One of the Alliant Reloader 19 replicates (SP-157) was misclassified as Reloader 22 (SP-158).

The results from this study show that micromorphometry can provide valuable information to help determine the brand of small arms propellant. In this study, the overall success rate for accurately classifying SAPs was 84%; however, this value is dependent on the granule morphology class. The accuracy for both cylindrical and spherical propellants is 90%. The success rate for flake propellants was slightly lower at 85% and that for flattened spherical propellants was 74%. The accuracy of the method increases to 99% when the top five "hits" are evaluated. When used alone (see discussion below), this small list of five possible brands may provide valuable intelligence information during the initial phases of criminal investigations.

The results from the study also demonstrate limitations that must be taken into account. The first limitation is that the accuracy of the method is predicated on the necessity for the unknown propellant to be in the database. Without a database which includes a significant portion of the available SAPs, false associations would occur. Another limitation is the occurrence of the same SAP being sold under different brand names by different distributors. This is a consequence of the industry in which product lines and entire distributors are bought,

sold, or contracted to make particular product lines. This impacted the success rate for two samples in the presented research project. For example, Winchester 231 (SP-001) was "falsely" associated with Hodgdon HP-38 (SP-004). However, these two brands are known to be the same product, sold under different brand names (for additional examples see [8]). The other example is the "false" association between Western Accurate 2230 (SP-059) and Western Ramshot X-Terminator (SP-098), which again are known to be the same SAP. Thus, care must be taken into account when including/ excluding particular brands.

It is important to note that the described method provides investigative information to help reduce the number of potential brands to a smaller subset of morphologically similar to be compared to an unknown sample. Once samples of known distributor and brand are obtained, additional descriptive and quantitative information can be used to further differentiate between them. Such information may include the degree of graphite coating, gross color, thickness ([9,10]), and morphological subcategories (e.g., cracked ball, lamel, perforated/ non-perforated granules, among many others). These additional metrics of comparison should be performed prior to destructive instrumental methods.

Additional future research will investigate the impact that the number of granules analyzed for a given sample has on the accuracy of the classification algorithm. The results from this analysis will help determine the minimum and optimal number of granules to be analyzed to maximize classification accuracy and efficiency. In addition, work is currently underway to develop a standalone application in which criminalists can utilize the described database and methodology for case work [11].

*Phase 3 GC/MS additive profiling*

Each of the 204 samples of SAP were analyzed in triplicate (three separate aliquots from each powder) by GC/MS. The samples were run on two different instruments:

- PSU: HP 6890 coupled to an Agilent 5973
- Microtrace: Thermo Trace 1310 gas chromatograph coupled to a Thermo ISQ LT single quadrupole mass spectrometer.

Both instruments used 30-meter RTX-1 columns and were operated using the following conditions:

- Inlet temperature: 175°C
- MS temperature: 280°C at PSU and 250°C at MT
- Flow rate: 1.0 mL/min He
- Split ratio: 10:1
- Oven temperature:
    - Initial temperature: 100°C (hold for 3 minutes)
    - Ramp at 10°C/min up to 250°C
    - Final temperature: 250°C (hold for 5 minutes)
- Solvent delay: 5 minutes
- Total run time: 23 minutes

The SAP samples were extracted using a method adapted from [12]. The method is briefly outlined below:

1. Place approx. 30 mg of powder is placed into a 5 mL disposable centrifuge tube.
2. Filled to 5 mL with 1:3 butanol:methanol mixture. Plug the cap and allow the powder to extract for 60 minutes. Note this extraction time was later reduced to 15 minutes when sonication was utilized.
3. The extracts were centrifuged at 3.0 relative centrifugal force (rcf) for 30 seconds. The supernatant liquid was poured into 10 mL syringes fitted with 0.45 µm filters.
4. The filtrates were collected in glass beakers or vials.
5. 200 µL of the extract was added to new GC vials containing 800 µL of 1:3 butanol:methanol mixture.

6. Samples were stored at 4°C until GC/MS analysis

The peaks detected in the GC chromatograms were evaluated based on their retention times, mass fragmentation patterns, counts, and comparison to known standards. Fourteen different additives were detected in the 204 samples of smokeless powder (Table 2).

**Table 2. Summary of additives detected in the samples of SAP.**

| | |
|---|---|
| Nitroglycerin | Methyl Centralite |
| 2,6-Dinitrotoluene | Ethyl Centralite |
| 2,4-Dinitrotoluene | Dibutyl Phthalate |
| Diphenylamine | 2-nitrodiphenylamine |
| Trinitrotoluene | N-Methyl-N,N'-diphenylurea |
| Triethyl Acetyl Citrate | Diisopentyl Phthalate |
| Dibutyl Adipate | Tributyl Citrate |

The area under the curve (AUC) values for the peaks identified in each sample were reduced to the following categorical values: not detected (n.d.), trace (<1%), minor (1-10%), and major (>10%). A limitation of this approach is that the level at which an additive may be present could be at the limit for being placed into one of the "bins" (e.g., major, minor, trace, n.d.). This process of reducing the data to categorical values allowed better comparison of the data collected on the two instruments. The data were analyzed using customized code written in R. By combining general propellant morphology and GC/MS additive profiles, 75 unique combinations were recognized. Each combination consists of 1-14 different brands. The unique combinations can be found in the attached as a spreadsheet. The data were also examined to look for additive

profile trends based on the SAP distributor. The data are shown in the attached spreadsheet. These tabulated results may provide caseworkers with a framework in which to interpret the strength of a comparison between questioned and known samples using additive profiles.

From the previous micromorphometry phase of the research, 32 samples were misclassified. These misclassified pairs were compared using GC/MS additive profiles. The results of this combined analysis are shown in Table 3. As can be seen, 18 of the 32 could be differentiated using additive profiling. Thus 14 samples could not be differentiated by the two combined methods.

**Table 3. Summary table showing the misclassified pairs and the results from their comparison using GC/MS additive profiles.**

| Distributor | Brand | Distributor | Brand | Differentiated by Additive Profiling? |
|---|---|---|---|---|
| Winchester | 231 | Hodgdon | HP-38 | No |
| Winchester | 296 | Western | Accurate No. 7 | Yes |
| Hodgdon | H110 | Western | Accurate No. 9 | Yes |
| Hodgdon | H335 | Hodgdon | CFE 223 | Yes |
| Hodgdon | H335 | Hodgdon | BL-C(2) | No |
| Hodgdon | H335 | Hodgdon | BL-C(2) | No |
| Hodgdon | Titewad | Western | Ramshot Zip | Yes |
| Hodgdon | Longshot | Winchester | 572 | Yes |
| Hodgdon | International | Hodgdon | HI-Skor 700-X | No |
| Winchester | AutoComp | Hodgdon | Lil' Gun | Yes |
| Hodgdon | BL-C(2) | Hodgdon | H335 | No |
| Hodgdon | Leverevolution | Hodgdon | H335 | Yes |
| Hodgdon | HI-Skor 700-X | Hodgdon | Clays | No |
| IMR | 8208 XBR | Hodgdon | Benchmark | No |
| IMR | 4955 | IMR | 8133 | Yes |
| Western | Accurate 2230 | Western | Ramshot X-Terminator | Yes |
| Western | Ramshot Silhouette | Western | Accurate No. 5 | No |
| Alliant | E3 | Alliant | Red Dot | Yes |
| Alliant | Herco | Alliant | Blue Dot | No |
| Alliant | Reloader 25 | Hodgdon | H4831SC | Yes |
| VihtaVuori | N540 | VihtaVuori | N550 | No |
| Hodgdon | CFE BLK | Western | Accurate 1680 | No |
| Western | Ramshot TAC | Western | Ramshot X-Terminator | Yes |
| Alliant | Unique | Alliant | Blue Dot | No |
| VihtaVuori | N160 | VihtaVuori | N150 | Yes |
| Ramshot | Ramshot True Blue | Western | Accurate 4100 | Yes |
| Alliant | Power Pistol | Alliant | BE-86 | No |
| VihtaVuori | N165 | VihtaVuori | N150 | Yes |
| Alliant | 0.41 | Alliant | 20/28 | Yes |
| Alliant | Power Pro 1200-R | Hodgdon | BL-C(2) | Yes |
| Western | Accurate Nitro 100 NF | Hodgdon | Longshot | Yes |
| Alliant | Reloader 19 | Alliant | Reloader 22 | No |

*Phase 4 Isotope ratio mass spectrometry*

The nitrocellulose (NC) fraction of each of the 204 samples of SAP was analyzed in triplicate (three separate aliquots from each powder) by isotope ratio mass spectrometry (IRMS). The following isotope systems were measured for all 204 SAP samples: $\delta^{13}C$, $\delta^{15}N$, $\delta^{18}O$; which resulted in approximately 1800 isotopic measurements. The samples were run at the stable isotope facilities at PSU and the University of California-Davis.

The purified NC was obtained by performing 2-4 additional methanol:butanol extractions (after initial GC/MS extraction). These multiple extractions ensured that the additives were sufficiently removed from the NC matrix. Following the final methanol:butanol extraction, the NC was dissolved in reagent-grade acetone and syringe-filtered into clean 10 mL glass beakers. Filtration was necessary to remove particles of graphite coating. The acetone was evaporated overnight in a fume hood at room temperature. Portions of the NC were weighed and placed into silver capsules (for oxygen isotope analysis) or tin (for carbon and nitrogen isotope analysis). The samples were run with appropriate standards and reported relative to atmosphere (nitrogen), Vienna Pee Dee Belemnite (carbon) and Vienna Standard Mean Ocean Water (oxygen).

The results for the three isotope systems were visualized based on the distributor and country of manufacture (Figures 2-7).

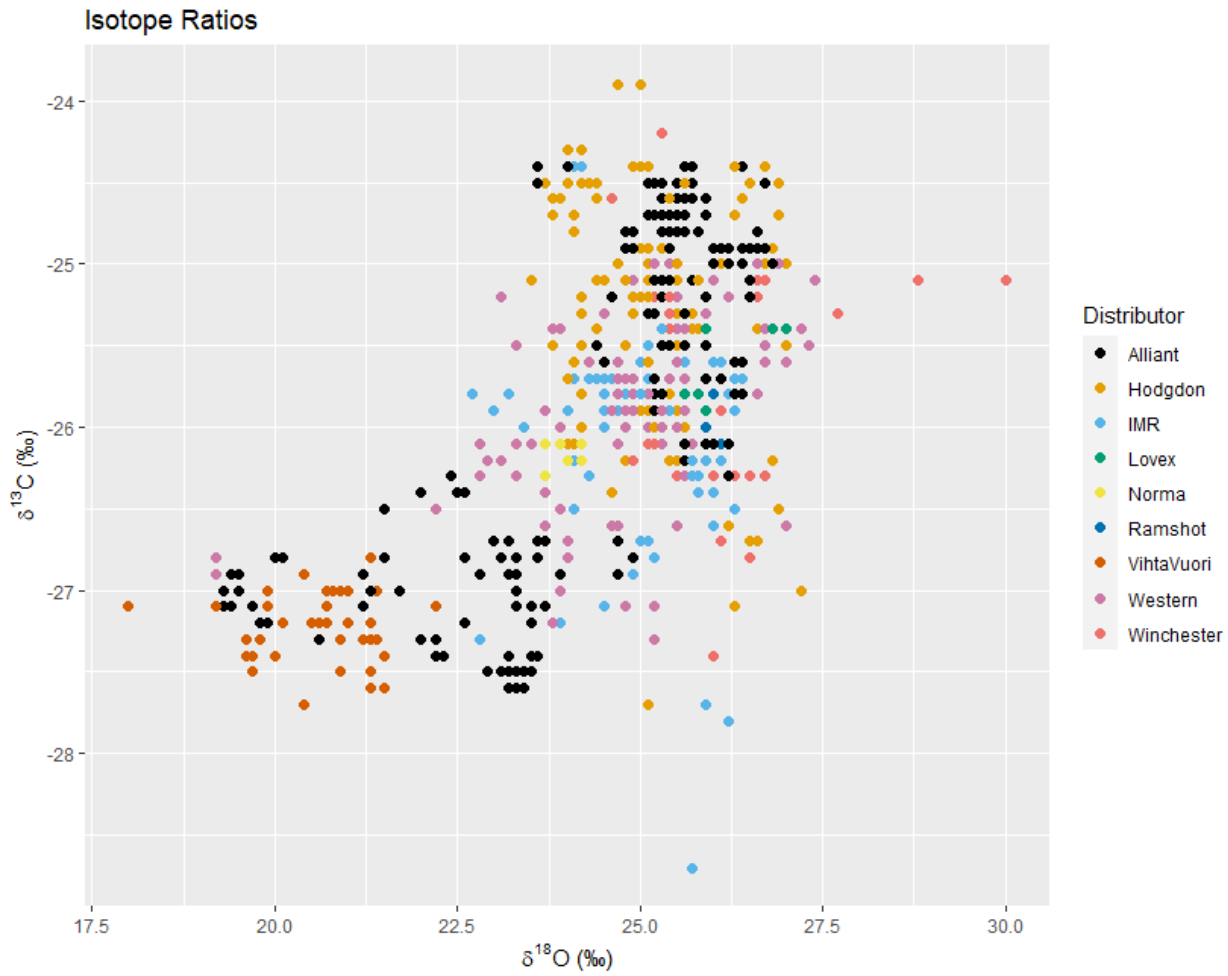


Figure 2. Oxygen and carbon isotopic ratios for the SAP samples colored by distributor.

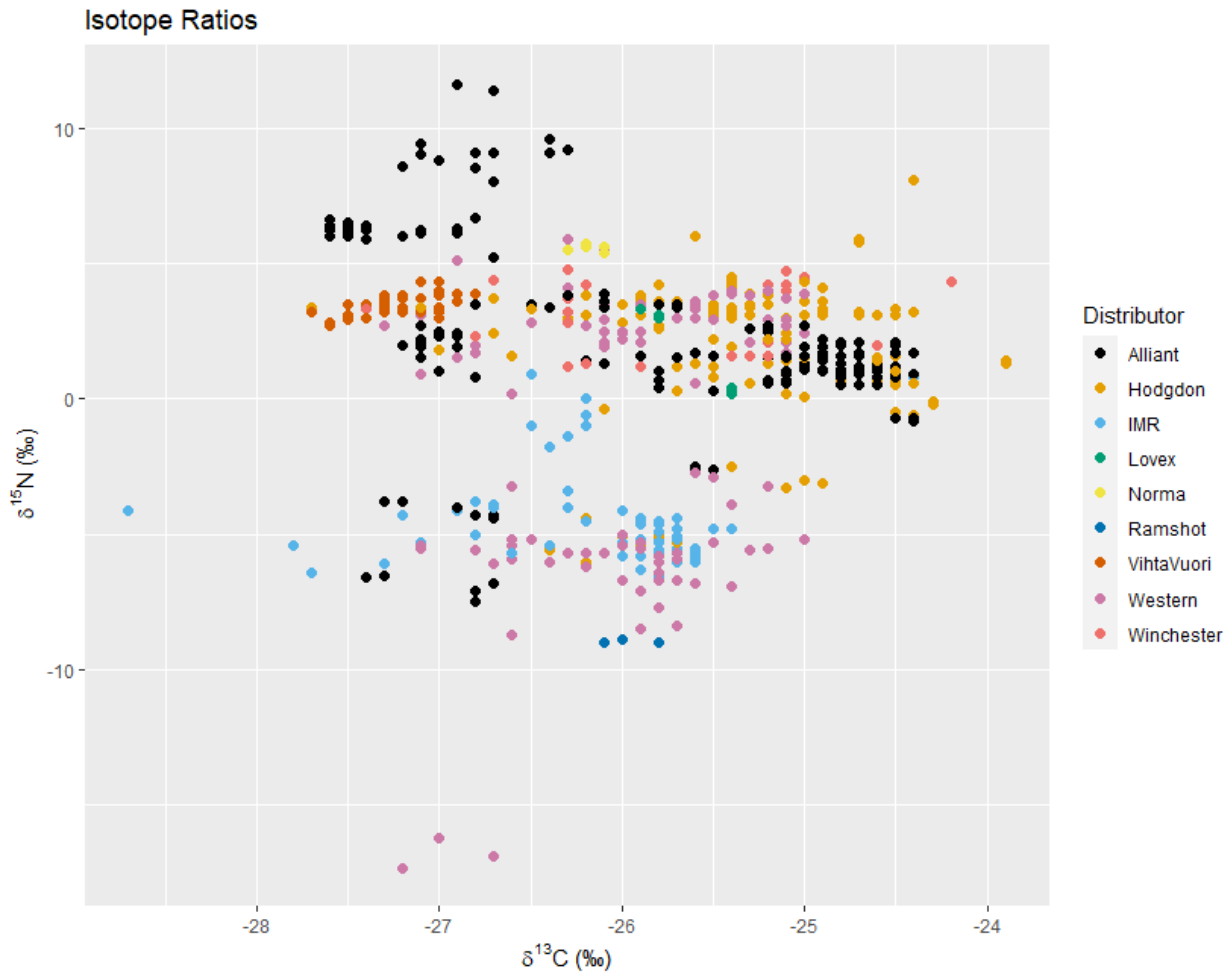


Figure 3. Nitrogen and carbon isotopic ratios for the SAP samples colored by distributor.

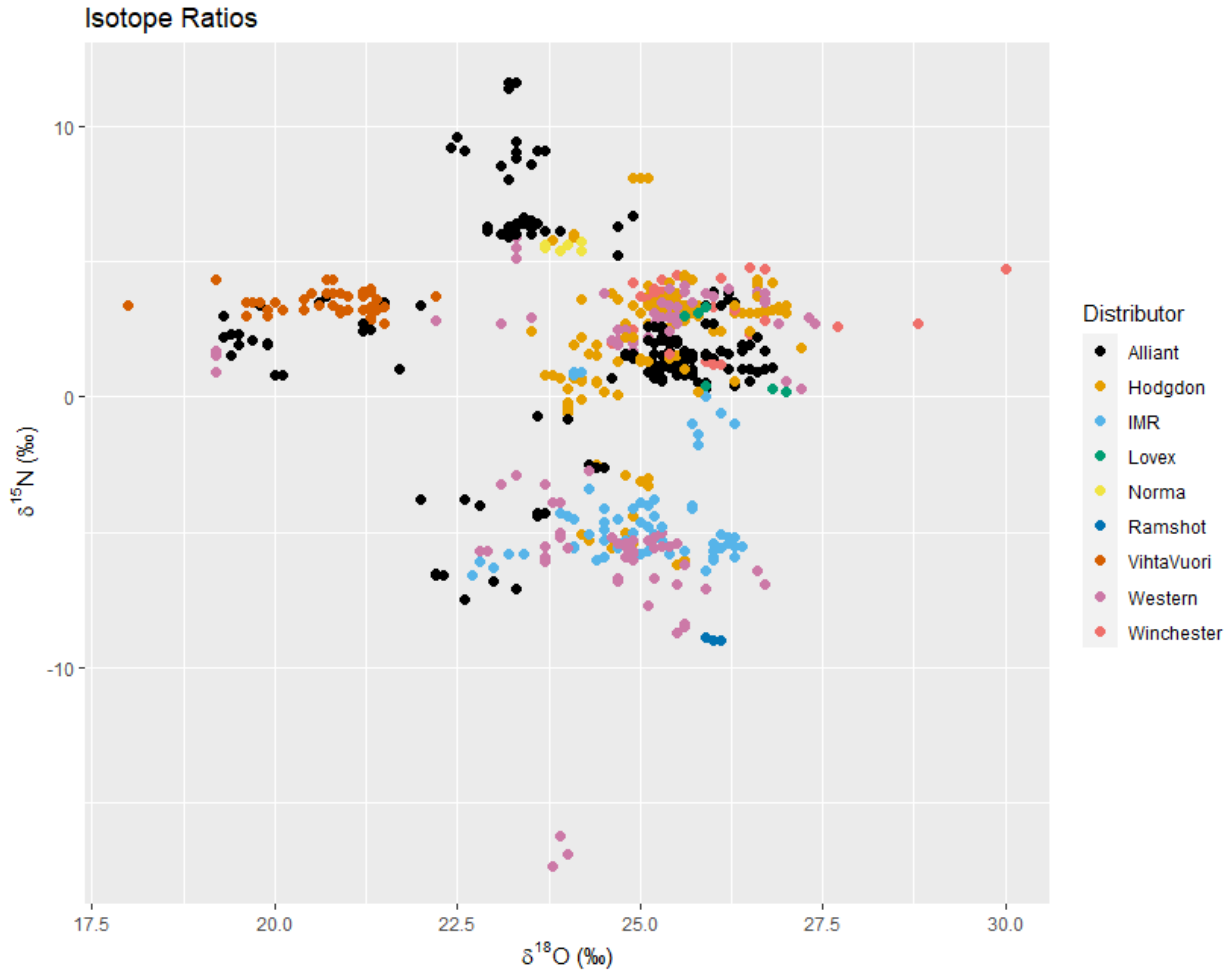


Figure 4. Nitrogen and oxygen isotopic ratios for the SAP samples colored by distributor.

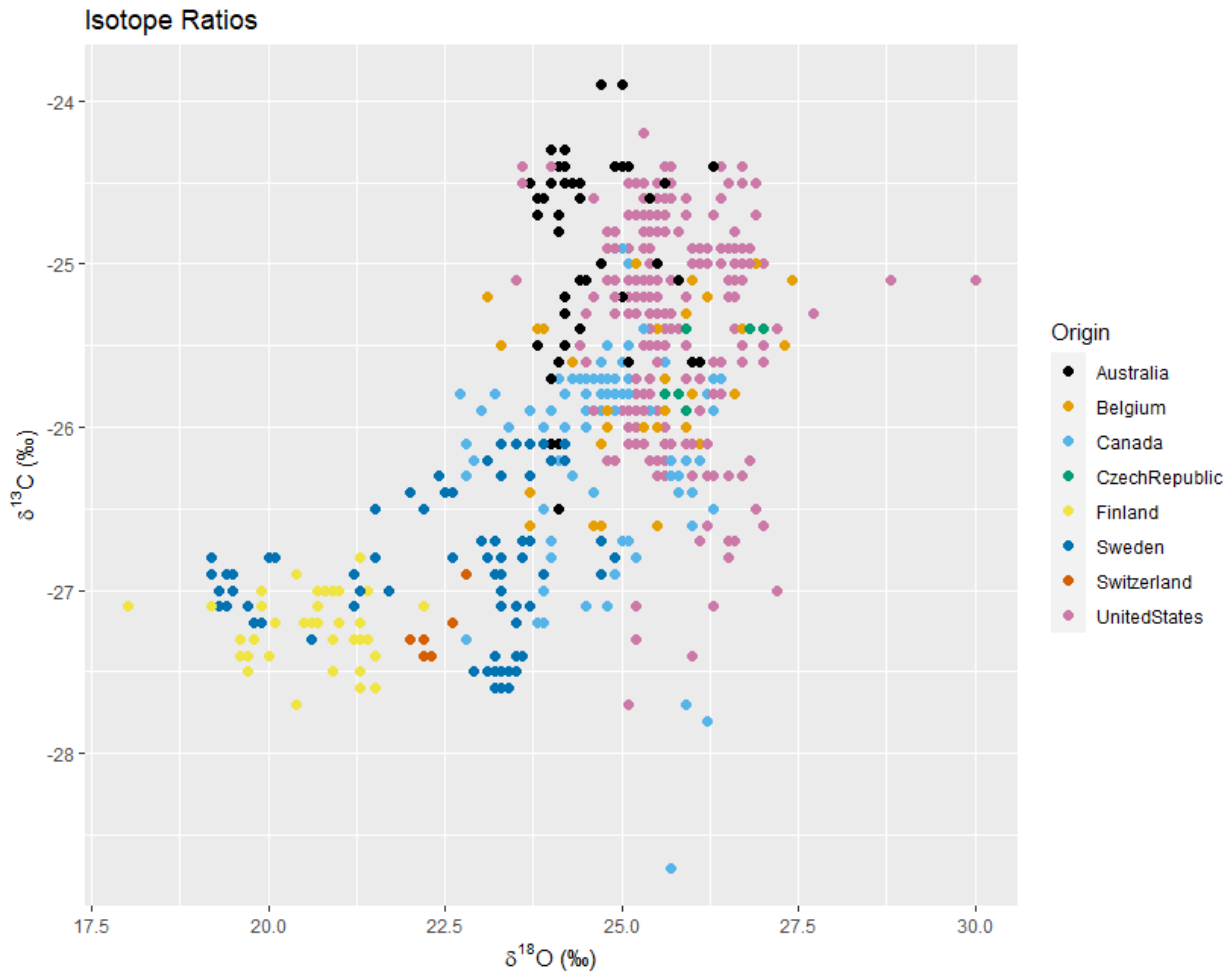


Figure 5. Oxygen and carbon isotopic ratios for the SAP samples colored by country of manufacture.

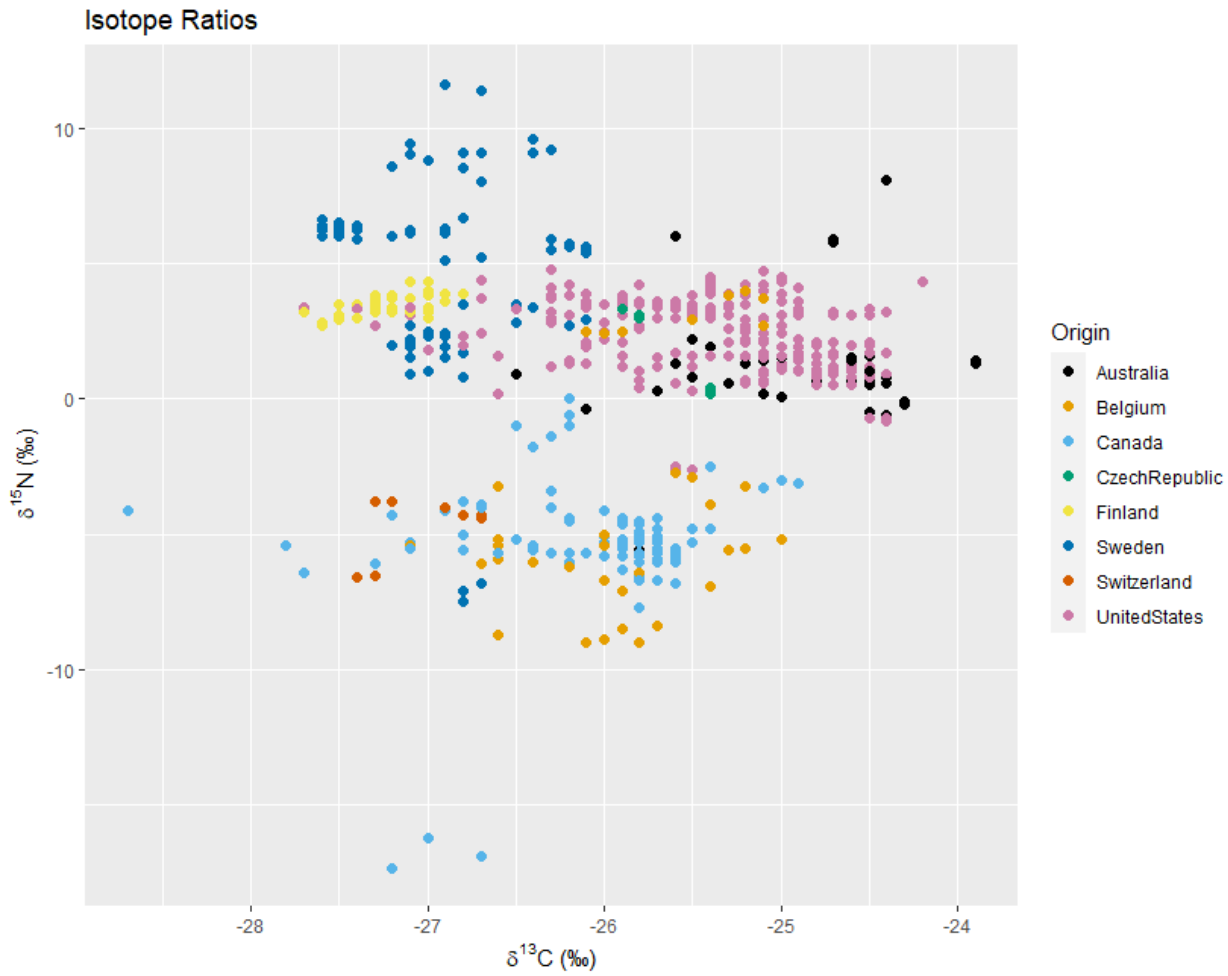


Figure 6. Nitrogen and carbon isotopic ratios for the SAP samples colored by country of manufacture.

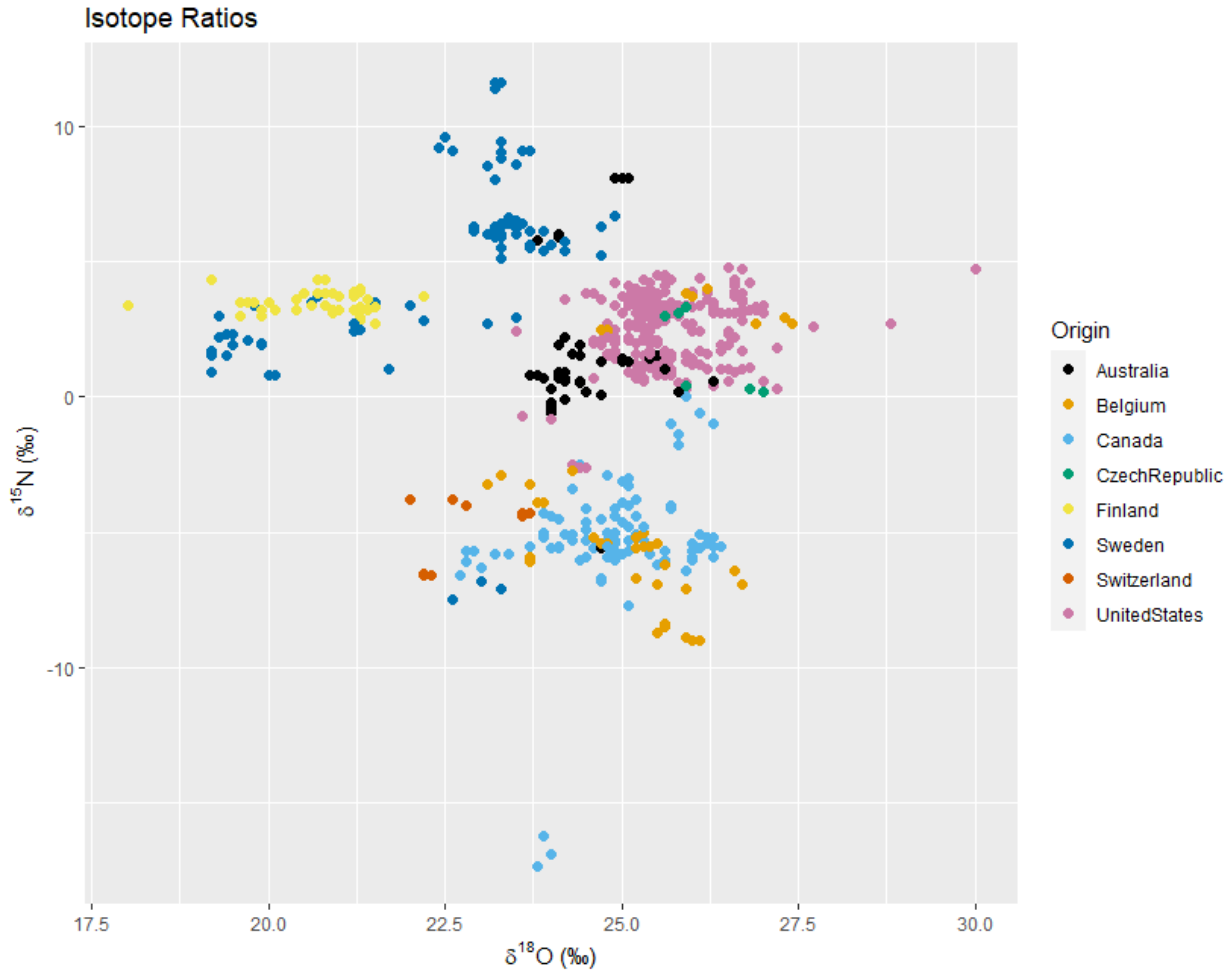


Figure 7. Nitrogen and oxygen isotopic ratios for the SAP samples colored by country of manufacture.

As previously discussed, 14 samples that could not be differentiated by combining micromorphometry and GC/MS additive profiling. To assess the utility of stable isotope signatures to potentially differentiate between the 14 samples, a MANOVA analysis was undertaken (Table 4). The results from this analysis showed that 8 of the misclassified pairs had F-statistics and associated p-values that resulted in rejecting the null hypothesis and thus support the inference that they are from different sources. The MANOVA results for the remaining 6 samples failed to reject the null hypothesis; thus, the isotope data failed to differentiate these samples.

Table 4. Results for the comparison of the misclassified pairs using MANOVA.

| Misclassified Pair | | F-statistic | p-value | Interpretation |
|---|---|---|---|---|
| Reloader 19 | Reloader 22 | 195.13 | 8.09E-08 | excluded |
| SP_007.C | SP_032.E | 883.33 | 0.001131 | excluded |
| 8208XBR | Benchmark | 234.11 | 0.004256 | excluded |
| Ramshot Silhouette | Accurate No.5 | 116.05 | 0.008556 | excluded |
| SP_007.D | SP_032 | 35.71 | 0.02736 | excluded |
| SP_007.C | SP_032 | 37.333 | 0.0262 | excluded |
| International | Clays | 31.41 | 0.03101 | excluded |
| Power Pistol | BE-86 | 78.833 | 0.01255 | excluded |
| 231 | HP-38 | 10.769 | 0.08616 | included |
| International | HI-Skor700-X | 10.899 | 0.08521 | included |
| CFEBLK | Accurate1680 | 15.463 | 0.06135 | included |
| Herco | Blue Dot | 6.5486 | 0.1353 | included |
| N550 | N540 | 4.3333 | 0.1932 | included |
| Unique | Blue Dot | 2.3974 | 0.3079 | included |

**Summary**

The results from this study show that non-destructive micromorphometry provides valuable information to help limit the number of potential brands for a questioned SAP. As this analysis is non-destructive, it is recommended to be performed first in the sequence of SAP analysis. The

next step in the process was additive profiling by GC/MS. This method provided valuable information that complemented the micromorphometry results. It was noted that several distributors and brands overlapped in their additive profiles. Thus, the discriminatory power of additive profiling is lower than micromorphometry. The final step in the research analytical workflow was stable isotope ratio mass spectrometry. While these analyses did provide valuable information concerning distributor and country of origin (see Figures 2-7), the discriminatory power of the method needs additional statistical analysis. However, the data from the three isotope systems did provide additional discrimination for eight of the fourteen samples that could not be separated by micromorphometry and additive profiling. The results from each of three phases of the research are currently being prepared for submission for publication.

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**Artifacts**

Publications:

1. Casey M. Jarvis, Devin Kress, Wayne Moorehead, and Jack Hietpas. **in review.** Assessing the strengths and limitations of quantitative micromorphometry for the forensic examination of small arms propellant toward brand identification. Forensic Science International.
2. Claire Page, Otyllia Abraham, Ryan Schonert, Wayne Moorehead, and Jack Hietpas**.** ***In preparation.*** Assessing the utility of additive profiling for the differentiation of small arms propellants.
3. Claire Page, Ryan Schonert, Wayne Moorehead, and Jack Hietpas. ***In preparation.*** Assessing the utility of stable isotope signatures to discriminate small arms propellants.

Conference platform presentations:

1. Hietpas, J., Brown, C., Paige, C., Kress, D., Sowers, T, and Moorehead, W. 2020. The chemical,
   micro-morphological and isotopic characterization of smokeless powders. Session Number: 4-3-2, Pittcon Conference and Expo, Chicago, IL
2. Page, C., Schonert, R., Sowers, T., Moorehead, W., and Hietpas, J. 2020. Smokeless powder additive profiles and compound-specific stable isotope signatures for potential brand identification and sample discrimination. Am. Acad. of For. Sci. (AAFS) meeting. Anaheim, CA
3. Page, C., Hietpas, J., Moorehead, W., Schonert, R., and Sowers, T., 2019. Smokeless powder analysis. Northeast Association of Forensic Scientists meeting. Lancaster, PA.
4. Deibel, S., Kress, D., Brown, C.M., Moorehead, W., and Hietpas, J. 2019. Assessing the utility of smokeless powder micromorphometry for brand identification. Inter/Micro. Chicago, IL.
5. Brown, C.M., Schonert, R., Moorehead, W., and Hietpas, J. 2019. A quantitative comparison and
   differentiation of smokeless powders. Am. Acad. of For. Sci. (AAFS) meeting. Baltimore, MD.
6. Brown, C. and Hietpas, J. 2018. Semi-automated micromorphometry of small arms propellants for brand identification. Abstracts with Programs. Inter/Micro. Chicago, IL

Undergraduate thesis:

1. Samantha Deibel, 2019. Smokeless powder cross section analysis for brand identification. The Pennsylvania State University Schreyer Honors College.

MPS theses:

1. Claire Page, 2021. Smokeless powder additive profiles and stable isotope signatures for potential brand identification and sample discrimination. The Pennsylvania State University.
2. Casey Brown, 2018. Semi-automated micromorphometry of small arms propellant for brand identification. The Pennsylvania State University.
3. Ryan Schonert, 2018. Analysis of Extracted Additives in Smokeless Powder Using Gas Chromatograph-Mass Spectrometry. The Pennsylvania State University.

Data Sets:

Three large data sets were generated during this project, these include:

1. Micromorphometry. The data set consists of a large matrix containing all particle micromorphometry data.
2. GC/MS additive profiles. The data consists of a large matrix containing additive peak area values for all smokeless powder sample replicates.
3. Stable isotope data. The data consists of a large matrix containing the carbon, nitrogen, and oxygen isotope ratios for all smokeless powder replicates.