Optimizing a method for extraction of explosives from soil using gas chromatography/mass spectrometry

Kendall Archer
Mentored by Elizabeth Corriveau, Alex Jestel, Dr. Stan Ostazeski, and Carmen Adrover

Introduction

The purpose of this study is to create an efficient method for extracting explosives from soils in order to detect and analyze them. A successful method will have real-world implications such as bomb damage analysis. It is based on the premise that explosives can successfully be extracted from different matrices using organic solvents such as acetone and hexane. These explosives can be detected by a gas chromatograph equipped with a mass spectrometer detector (GC-MS). Most often there will only be trace levels of the chemicals in the soil samples so enough must be extracted in order for the instrument to detect it. The perfect method would extract 100% of the present chemical with little to no "background noise" (i.e. extra chemicals). Thurman and Ferrer (2012) managed to get an 80% recovery or above for 11 chemicals in a similar study. However, they were using contaminated water samples, so their method cannot be used for this. The amount of time the compounds are in the soil and the composition of the soil such as the presence of organic matter are factors that may affect the extraction. This method can improve the ability of U.S. military personnel to analyze soil samples efficiently for the detection of explosives.

Materials and Methods

In order to determine the best possible method in removing explosives from soil first the type of solvent to be used was determined. Then the method was tested for versatility by reproducing the experiment using different soil types and by executing a time series. First, the spiking solution was created, which is 10 mL of a 50 µg/mL combination of 17 explosive related compounds; the compounds are shown in Figure 1. Based on the target concentration the appropriate amount of each solution was added to a Class A 10 mL volumetric flask and then diluted with acetone. From this spiking solution, nine calibration standard solutions ranging from 20 µg/mL to 0.1 µg/mL were made. The method to create these is very similar to the method for creating the spiking solution. The procedure to set up and perform the extractions was the same for each test. One gram of local soil from an APG test range, acid washed sand from Alfa Aesar, or organic rich potting soil was weighed out and placed in a 12 mL vial. The soil was then spiked with 20 µL of the 50 µg/mL spiking solution using a 100 µL pipette. Each variable being tested will have three replicates along with one spiked blank (no soil) to show 100% recovery and one method blank (no spike) to ensure the soil had no original contaminants. Two mL of solvent were added and mixed to the vial and then the liquid layer was extracted with a Pasteur pipette. This was done twice. The extractions were filtered and concentrated to 1 mL using a nitrogen evaporation machine. The samples were then run on the GC-MS.

Materials and Methods (cont.)

Explosive related compounds

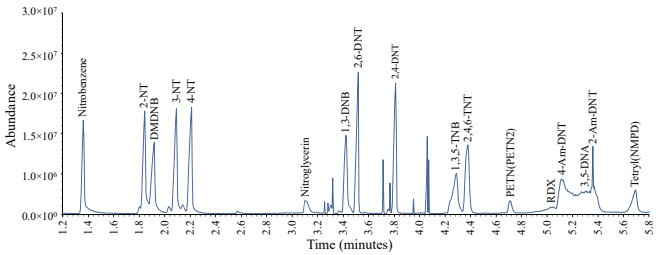
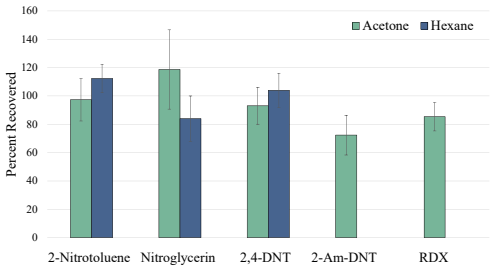


Figure 1 (above): A chromatogram showing peaks for the 17 explosive related compounds in the 50 µg/mL solution.

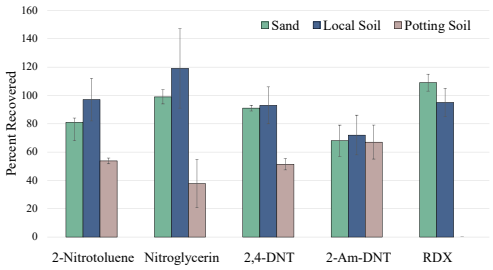
Results

Compound recovered by solvent



Graph 1 (left): Shows the relationship between the organic solvent used for extraction and the percent of the compound recovered from local soil. The error bars show one standard deviation. These five compounds represent overall conclusions.

Compound recovered by soil type



Graph 2 (left): Shows the relationship between soil type and percent of the compound recovered. Decrease in percent recovered can be attributed to the increase of organic matter in the soil. These compounds represent overall conclusions.

Over 100% recovery can be attributed to solvent evaporating during extraction or analysis while the vials are sitting on the GC-MS.

Results (cont.)

In the first experiment, it was determined that acetone was more successful than hexane in extracting the 17 explosive related compounds as shown by Graph 1. The percent recovery for many of the compounds were very similar in the two solvents, but 5 of the 17 were not present at all in the hexane extractions, so acetone is the preferred solvent. Graph 2 displays that extractions from the sand and local soil did not show a significant difference in recoveries, however the potting soil samples showed much lower amounts of recovered compounds. This difference can be attributed to the presence of more organic matter. A time series was conducted on potting soil which showed that the amount of time that a spike sits on the potting soil before extraction decreases percent recovery. This possibly is because the compounds interact with the organic matter and either degrade or do not go into the solvent. The same time series study was done with the local soil however time did not appear to have a significant impact on the percent recovered.

Conclusion

The purpose of creating and testing this method was to produce a method to help with real world problems such as bomb damage analysis or the remediation of old test ranges. The determined extraction method is relatively efficient since it does not use an excessive amount of resources such as solvent, soil, and time. Acetone was discovered to be the more successful solvent when trying to extract the 17 compounds. Our original hypothesis assumed that 80% recovery could be achieved using this method and the results support this for sand and local soil. The time that a spike sits on the local soil did not greatly affect the percent that was recovered, but a decrease in recovery was seen for the potting soil time series. These results suggest that when using this method for an unknown sand or soil sample with little organic matter, about 80% of the present compounds will be extracted if the compounds are one of the 17 compounds tested in these experiments. The method can be tested more in the future to further understand its uses. There are many more solvents that can be tested other than just acetone and hexane. While a large range of soils were tested from sand to potting soil, there are many types in between. This could determine the maximum level of organic matter in the soil that still provides 80% recovery. Finally, different environmental conditions such as humidity could be looked at to provide a more realistic extraction process.

References

Thurman, E. M., & Ferrer, I. (2012). *Adapting EPA Method 8330B for Analysis of Explosives in Water to SPE and LC/MS/MS.* Boulder, CO: Agilent Technologies.