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NCFS Support of SWGDE and T/SWGFEX Projects/Activities – T/SWGFEX Portion

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Abstract

The TWGFEX Portion of the National Center for Forensic Science (NCFS) Support of SWGDE and TWGFEX Projects/Activities was comprised of three major tasks: (1) Maintenance and Expansion of the Ignitable Liquids Reference Collection and Database, (2) Smokeless Powders Database development and (3) Implementation of a Searchable Ignitable Liquid Database. An additional 120 reference ignitable liquids were incorporated into the collection expanding the number of database entries to 614 making the ILRC the largest collection of reference ignitable liquids with an associated database of analytical data. The collection contains reference ignitable liquids spanning all ASTM E1618 classes and carbon ranges from various U.S. geographical areas and time frames. Part of this project involved the development of a new database containing data from the pyrolysis of common substrates (building materials). The Substrate Database benefits the fire debris analyst in their evaluation of whether the residue extracted from fire debris evidence contains an ignitable liquid and in the classification of any ignitable liquid present.

The Smokeless Powder Database is a regularly updated reference collection of powders obtained from various sources including vendors and manufacturers. It is designed to assist the forensic explosives analyst in characterizing, classifying, and comparing smokeless powder samples based on their physical and chemical properties. The database opened in 2011 with 100 records. Approximately 800 total records are being processed for release. These records come primarily from three sources, the Federal Bureau of Investigation, the Orange Co. California Crime Laboratory, and samples analyzed at the National Center for Forensic Science.

A searchable ignitable liquids database tool has been developed based on the use of the total ion spectrum (TIS) for finding the best match between a fire debris sample and ignitable liquids in the Ignitable Liquids Reference Collection (ILRC). The match criterion is based on the use of several distance and similarity metrics. A search of the entire ILRC is completed in a fraction of a second. Similarly, a sample can be searched against the Substrates Library. A sample may also be compared to the best-fit combination of an ILRC liquid and a single substrate. The searchable ignitable liquids database tool was developed in collaboration with several forensic laboratories. The software developed under this project has been tested in three forensic laboratories (South Carolina Department of Law Enforcement, Columbia SC; Tucson Police Department, Tucson AZ; Department of Public Safety, Burlington, VT) during the development phase. The search techniques are covered by a U.S. Patent and the software will be distributed electronically to forensic fire debris analysis laboratories.

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Executive Summary

The Technical/Scientific Working Group for Fire and Explosion (T/SWGFEX) was established in the mid 1990's as an organization of forensic scientists dedicated to the investigation of crimes involving fire and explosions. Over the years, T/SWGFEX members have maintained a strong interaction with the National Center for Forensic Science (NCFS) at the University of Central Florida (UCF). T/SWGFEX members comprise practicing professionals involved in the investigation, training, education and laboratory analysis of fire and explosion scenes. The Ignitable Liquids Reference Collection Committee is a T/SWGFEX committee of fire debris analysts who work with the National Center for Forensic Science (NCFS) in developing and maintaining the Ignitable Liquids Reference Collection (ILRC) and Database. The ILRC is a collection of ignitable liquids from refineries and distributors, but mostly commercially available products from around the United States. These reference ignitable liquids are compared to ignitable liquid residues extracted from fire debris collected at a fire scene. The reference ignitable liquids are analyzed at NCFS following established protocols and the analysis results are submitted to the committee for review and classification according to ASTM E1618. The product information, analytical data, and classification information for each reference liquid are uploaded onto the ILRC database, which is administered by NCFS. The three tasks incorporated into this project share a common focus on T/SWGFEX databases. The tasks include: (1) maintenance and expansion of the Ignitable Liquids Reference Collection and Database, (2) development of a smokeless powders database and (3) implementation of a searchable ignitable liquid database.

Maintenance and Expansion of the ILRC: The ILRC is utilized by forensic fire debris analysts from around the world for the analysis of casework samples. The ILRC is an important resource which is typically unmatched by single-laboratory databases. Maintaining and expanding the ILRC database enhances its' value to the forensic community. Despite the utility of the database in identifying ignitable liquid residue, there are frequently cases when the sample contains chromatographic peaks that cannot be explained by the ignitable liquid, but may be explained by pyrolysis products derived from decomposition of common building materials. The T/SWGFEX ILRC Committee identified the need for a database of substrate material decomposition products. A modified destructive distillation method developed by the State of Florida Bureau of Forensic Fire and Explosives Analysis was investigated for burning of substrate materials to create simulated fire debris. The study's objective was to determine if the method would consistently produce fire debris typically found at a fire scene. The experimental parameters developed for the modified destructive distillation method are suitable for creating a substrate database. A database of burned materials commonly found in fire debris provides information to assist the fire debris analyst in determining whether the compounds extracted contain any ignitable liquid residues and aid in classification. The second major task of this project was to develop and populate a Substrate Database. The ILRC committee collaborates with NCFS by assisting in the design of the substrate database and by reviewing the data. NCFS obtains, prepares, and analyzes the materials for incorporation into the database as well as administers the database. The Substrate Database design is a modified version of the ILRC database and the two databases are linked to one another. There is no reference sample collection of substrate materials since it is not required by ASTM E1618. The information gathered in the Substrate Database is utilized in training and aides in ascertaining which components extracted in the fire debris are from an ignitable liquid residue.

Maintenance and expansion of the Ignitable Liquids Reference Collection (ILRC) was accomplished by obtaining liquids throughout the United States and Canada from the collections of forensic laboratories performing fire debris analysis. There were 120 liquids analyzed and uploaded into the database from January 2009 to the present bringing the total number of ignitable liquids in the database to 614. The ILRC committee reviewed and classified 90 of the 120 liquids which were then placed in the public section of the database. There were a total of 277,713 hits on the database from January 2009 through June 2011 with a total of 683,816 since September 1, 2007. Hits refer to the number of times a user accessed the database. The count of database hits prior to September 1, 2007 (new database) is inaccurate or unknown. A total of 6,448 reference ignitable liquids were sold at minimal cost during this period. Since 2002, there have been 181 sales of 15,853 reference ignitable liquids to 88 customers in 42 states within the United States and six international countries.

A timeline for the completion of project goals was established and reported to the ILRC committee in November 2008. NCFS and ILRC committee discussed the design of the new Substrate Database. The requirements and functionality of the database were communicated to the database designer Tony Elmiger of TeGlobal. A prototype of the database was available for review on March 1, 2009 on the TeGlobal server. One hundred entries were uploaded into the prototype database consisting un-burned and burned debris from 50 materials. Committee members and NCFS personnel met in Atlanta with Tony Elmiger on May 4th and 5th, 2009 to discuss modifications to the database. The requested modifications were completed within two weeks. The data in the database was edited to fit the modified database format. The committee assessed the functionality of the database to determine if any further modifications would be required. Committee members and NCFS personnel met in Orlando on September 14th to discuss any final changes to the prototype. No major modifications were required and the Substrate Database was transferred to NCFS server in October 2009. Committee members reviewed the data and information for the 100 entries in preparation for the public release of the database. The Substrate Database was released to the public on July 1st, 2010 with 60 database entries. The Substrate Database has a total of 212 entries with 100 entries reviewed and available for public viewing (112 in committee review). There have been 22,378 hits on the database.

Smokeless Powders Database: The National Research Council (NRC) issued a report "Black and Smokeless Powders: Technologies for Finding Bombs and the Bomb Makers" in 1998. Key findings in the report include facts that 90% of the deaths and 80% of the injuries caused by pipe bombs containing black or smokeless powders occurred away from security screen points. This stresses the importance of the forensic investigation on determining the identity of the explosive material and the bomb maker. The report also emphasized that the materials recovered from the explosion scene such as unburned powder can aid in the identification of the product type and manufacturer provided that resources are available to the forensic scientist. Suitable resources include databases of products Information, physical characteristics, analytical data, and libraries of spectral identification of the components. The report indicates the fact that existing databases of black and smokeless powders were used extensively in bombing investigations yet were incomplete. Also, only large federal organization such as the Federal Bureau of Investigation, the Bureau of Alcohol and Tobacco and Firearms, and the U.S. Postal Service had the resources to develop these databases. Smaller state and local laboratories did not have the resources, time, and manpower to develop their own databases and did not have access to existing databases.

The database was developed by the Explosives Database Committee of the Technical/Scientific Working Group for Fire and Explosions (T/SWGFEX) and the National Center for Forensic Science (NCFS) at the University of Central Florida where the database in maintained. The Explosives committee identified the analytical methods most commonly used in the analysis of smokeless powders and assisted NCFS with developing procedures for these analytical methods. Some design aspects and data incorporated in the Smokeless Powders Database are contributions from the Federal Bureau of Investigation. The database design is divided into three sections; product information, physical characteristics, and chemical composition. The user can search multiple criteria from all three sections of the database where the user can decide what image (powder, container, FTIR spectrum, or GC-MS TIC) returns with the list of search results. The database is open access and is provided as a free service to the forensic science community. Since the intent of a smokeless powders database was for investigative purposes, the Explosives Database committee and NCFS decided a collection of reference samples available for purchase was not necessary.

The Smokeless Powders Database was released January 1, 2011 with 100 entries and can be viewed at http://www.ilrc.ucf.edu/powders. In 2011, development of the analytical methods was completed and standard operating procedures were written. NCFS has obtained 87 smokeless powders which are in the process of being analyzed. Mr. Ron Kelly from the FBI has provided product information and analytical data for an additional 650 smokeless powders from their agency database. As of September 15th, 2011 there have been 2,421 database hits.

Implementation of a Searchable Ignitable Liquid Database: Most fire debris analysis laboratories in the United States, and many laboratories in other countries, follow the ASTM E1618 Standard Method for the analysis of fire debris GC-MS data. The general procedure followed by these laboratories involves examining the fire debris sample in accordance with the procedures outlined in E1618 to arrive at a tentative ASTM classification. The ILRC is searchable by ASTM class, carbon range, etc. and the analyst must examine each of the entries having the assigned class to visually identify a chromatographic pattern that closely matches the fire debris sample's chromatographic profile. Before the ILRC was available online, this same process was used to analyze and classify samples; however, the each laboratory had a local method for database organization and searching. The advent of the ILRC brought a common interface and database search process to all laboratories that were willing to utilize this online resource. Standardization of the organization and search routine was a step forward for fire debris analysis through a partnership between the forensic community and the NCFS. The ILRC search method has been in place since the system became available online in 2002. The goal of this project was to implement a computer-based search routine to facilitate the analysis of fire debris casework samples. The goal of the project does not encompass a way to circumvent the use of E1618 and does not intend to replace this ASTM method, but rather, this project is intended to by synergistic with ASTM E1618.

The visual pattern recognition used by fire debris analysts for sample identification relies on chromatographic patterns, which vary significantly between laboratories. Peak alignment routines and retention indices offer potential methods to overcome the retention time variation challenge; however, variation in analytical column composition and differing oven temperature programs may lead to nonlinear relationships between retention times and significantly complicate the implementation of a strong electronic search routine. This project made use of an alternative approach which grew out of NASA research which investigated the use of electron ionization for the identification of organic analytes. Research at NCFS has shown that the total ion spectrum (TIS), equivalent to the average mass spectrum across the chromatographic profile, is sufficient for the identification of complex mixtures, of the type that are indicative of many ignitable liquids. Rapid advances in computing power with the advent of multi-core processors, massive memory blocks, stable operating systems and fast internet connections have created a climate that is much more favorable to the deployment of computationally intensive automated search methods and the general acceptance and use of those systems in forensic fire debris analysis laboratories.

The searchable ignitable liquids database tool was developed based on the use of the total ion spectrum (TIS) for finding the best match between a fire debris sample and ignitable liquids in the Ignitable Liquids Reference Collection (ILRC). The match criterion is based on the use of several distance and similarity metrics. A search of the entire ILRC is completed in a fraction of a second. Similarly, a sample can be searched against the Substrates Library. A sample may also be compared to the best-fit combination of an ILRC liquid and a single substrate. These searches can be completed in a fraction of a second on standard laboratory computers.

The searchable ignitable liquids database tool was developed in collaboration with several forensic laboratories. The software developed under this project has been tested in three forensic laboratories (South Carolina Department of Law Enforcement, Columbia SC; Tucson Police Department, Tucson AZ; Dept of Public Saftey,Burlington, VT) during the development phase. The original version of the software was personally delivered to the laboratories by NCFS staff, installed on forensic laboratory computers and training was provided to laboratory personnel. Subsequent versions of the software were delivered over the internet and installed by laboratory personnel. Internet delivery of the software will be used for future delivery to laboratories wishing to use the software.

The searchable ignitable liquids database tool was originally developed as a compiled program to run under Microsoft Windows. The software was upgraded by reprogramming in MatLab, to run under the MatLab runtime compiler, which is distributed free of charge with the software. The user does not need to purchase MatLab to use the software developed under this program.

The accuracy of the algorithms has been tested on laboratory burn and large – scale burn samples obtained in collaboration with the Florida Fire College and leveraging other funding from the National Institute of Justice (Application of Chemometrics for the Identification of Ignitable Liquids in Fire Debris Samples, Grant Number 2008-DN-BX-K069). One US patent ("Systems and Methods for Identifying Substances Contained in a Material", application 12/244,281) covering the search method has been approved and the patent should issue in 2011.

Current fire debris analysis practice may benefit significantly from the use of the software provided under this project. The software will be further introduced to the fire debris analysis community through T/SWGFEX and future AAFS conferences. In addition to the immediate use of the software, we will also use the software as a platform for introducing research results into practicing fire debris analysis laboratories following their publication under peer – review.

Conclusions: This project has focused on three tasks, each concerned with providing services and resources to the forensic fire and explosion analysis communities. Each task has produced a product that is currently accessed by the forensic community. The Ignitable Liquids Reference Collection and Database and the associated Substrates Database have been improved under this funding and they are consulted on a daily basis for the analysis of casework samples. The development of a Smokeless Powders Database fulfills a need that was identified by the National Research Council in 1998, but not acted on by any agency to create a resource that was freely available across the forensic community. Finally, the task of providing a computer searchable ignitable liquids database resource has been achieved; however, like the databases maintained by NCFS, we regard this software product as a living project which will continue to expand and provide a platform for leveraging future research and disseminating the results to the forensic community. All of these tasks have been accomplished through close and continued collaboration with the forensic community.

Ignitable Liquids Database and Reference Collection: Maintenance and Expansion

ILRC Maintenance and Expansion Introduction

The Technical/Scientific Working Group for Fire and Explosion (T/SWGFEX) was established in the mid 1990's as an organization of forensic scientists dedicated to the investigation of crimes involving fire and explosions. T/SWGFEX members comprise practicing professionals involved in the investigation, training, education and the laboratory analysis of fire and explosion scenes. The Ignitable Liquids Reference Collection Committee is a T/SWGFEX committee of fire debris analysts who work with the National Center for Forensic Science (NCFS) in developing and maintaining the Ignitable Liquids Reference Collection (ILRC) and Database. The ILRC is a collection of ignitable liquids from refineries and distributors, but mostly commercially available products from around the United States. These reference ignitable liquids are compared to ignitable liquid residues extracted from fire debris collected at a fire scene. These reference ignitable liquids are analyzed at NCFS following established protocols and the analysis results are submitted to the committee for review and classification according to ASTM E1618.[1] The product information, analytical data, and classification information for each reference liquid are uploaded onto the ILRC database, which is administered by NCFS.

ASTM E1618 method requires laboratories conducting fire debris analysis to have available reference ignitable liquids that represent the various classes and carbon ranges. The ILRC provides a valuable resource to the fire debris analysis community in fulfilling that requirement. The database is open access and is provided as a free service to the forensic science community. Fire debris analysts have utilized the database for casework and training of personnel. Development of the ILRC database began in 2000 after a 1998 national survey of forensic laboratories indicated a need by fire debris analysts for a collection of reference ignitable liquids and an associated database of gas chromatography-mass spectrometry (GC-MS) analytical data.[2] The ILRC database was accessible to the public with 100 entries in January 2002. As the size of the database increased, the performance of the database suffered and a new database was designed and implemented in 2007 with approximately 400 entries. Maintaining and expanding the database by obtaining reference ignitable liquids from different geographical areas and incorporating GC-MS data from other countries was one of the tasks for this project.

In the course of a fire, many of the common furnishing and building materials will undergo pyrolysis and combustion processes producing small to medium sized hydrocarbons that may be extracted from the fire debris along with any possible ignitable liquid residue. These hydrocarbons can interfere in determining the presence of an ignitable liquid residue in the fire debris and the subsequent classification of the residue if present.[3] Distinguishing between hydrocarbons produced as a result of pyrolysis and combustion of materials and those from an ignitable liquid residue is a very challenging aspect of fire debris analysis. GC-MS data of unburned and burned materials commonly found as substrate in fire debris provides valuable information to the fire debris analyst. The reference substrate data assists the analyst in ascertaining the presence of an ignitable liquid residue in the presence of substrate materials. An analyst will be able to explain the presence of compounds not generally consist with those of ignitable liquids.

In 2007, NCFS began studying the feasibility of establishing a substrate database at the request of the ILRC committee. A modified destructive distillation method developed by the State of Florida Bureau of Forensic Fire and Explosives was investigated for burning of substrate materials into fire debris. The study's objective was to determine if the method would consistently produce fire debris typically found at a fire scene. The experimental parameters developed for the modified destructive distillation method produced a relatively consistent set of fire debris data. The ILRC committee's opinion was the method produced TIC patterns consistent with what they see in casework. A database of burned materials commonly found in fire debris provides information to assist the fire debris analyst in determining whether the compounds extracted contain any ignitable liquid residues and aid in classification. The second major task of this project was to develop and populate a Substrate Database. The ILRC committee collaborates with NCFS by assisting in the design of the substrate database and by reviewing the data. NCFS obtains, prepares, and analyzes the materials for incorporation into the database as well as administers the database. The Substrate Database design is a modified version of the ILRC database and the two databases are linked to one another. There is no reference sample collection of substrate materials since it is not required by ASTM E1618. The information gathered in the Substrate Database is utilized in training and aides in ascertaining which components extracted in the fire debris are from an ignitable liquid residue.

ILRC Maintenance and Expansion Methods

Ignitable Liquids Reference Collection and Database

Maintenance and expansion of the existing Ignitable Liquids Reference Collection Database consisted of following previously established methods. Ignitable liquids obtained were obtained from retail distributors throughout the United States. Twenty microliters of the ignitable liquid is diluted by one milliliter of carbon disulfide. One microliter of the diluted ignitable liquid is injected into a 250°C injection port of a gas chromatograph mass spectrometer at a split ration of 50:1. Helium gas at a constant flow rate of 0.8 ml/min and a linear velocity of approximately 36 cm/sec passes the vaporized ignitable liquid through a gas chromatographic column. The column is 25 meters in length with an inner diameter of 0.20 mm and a 0.5 micron film thickness of 100% polydimethylsiloxane. The initial oven temperature is held at 50°C for 3 minutes and then increased at a rate of 10°C/min to a final temperature of 280°C which is held for 4 minutes. The transfer line from the gas chromatograph to the electron ionization mass spectrometer is at 280°C, the ionization source is at 230°C and the quadruple is at 150°C. The mass spectrometer scans 2 -3 scans per second over a scan range of 30 to 350 m/z. The ionization source is turned off between 1.54 and 2.00 minutes during solvent elution. The data file, total ion chromatogram and extracted ion chromatograms are uploaded to the database with the product name and description. The ILRC committee reviews the data and identifies the carbon range, determines the predominant ion profile, identifies the major components, and classifies the ignitable liquid according to ASTM E1618.

Substrate Database

Materials obtained for the Substrate Database were typically building materials, flooring, furniture and household items. Each material is prepared in two different manners; un-heated and heated. Sample sizes are approximately 6 cm x 6 cm. The heated samples underwent a destructive distillation process to produce combustion and pyrolysis products upon degradation of the materials. The modified destructive distillation method was developed by the National Center for Forensic Science and was based on a method described by State of Florida Bureau of Forensic Fire and Explosives laboratory. The sample materials are placed up-side down in an un-lined metal quart paint can on which a lid with nine 0.5 cm diameter holes is loosely placed onto the can. Heat is applied to the bottom of the can by a propane torch held 4 cm from the bottom of the can. Once smoke appeared from the can, heat continued to be applied for an additional 2 minutes. After the 2 minutes, the heat is removed and the lid was replaced with a solid lid to allow vapors within the headspace to condense as the can temperature returned to room temperature. The process of creating fire debris by modified destructive distillation is illustrated in figure 1.

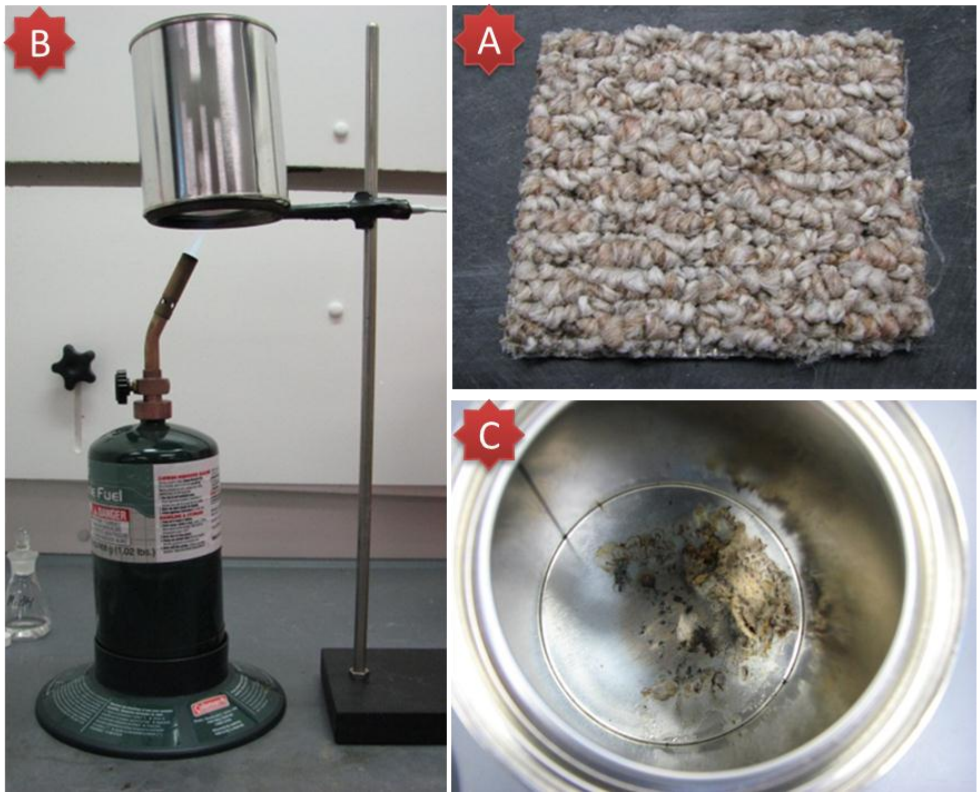


Figure 1: Process of creating fire debris by modified destructive distillation method using carpet; a) carpet prior to heating, b) heating, c) subsequent fire debris after heating carpet.

Interfering products from the heated and un-heated substrate materials are collected by passive headspace adsorption onto activated carbon following the ASTM E1412 standard method.[4] An activated carbon strip (10 mm x 22 mm) is suspended into the headspace of the can by a paperclip and un-waxed dental floss. The sealed can is heated for 16- 18 hours at 66ºC. Once cooled to room temperature, the activated carbon strip is removed from the can and cut in half lengthwise. One half is archived and the other is deposited into a vial with 1 ml of carbon disulfide for analysis. Analysis of the extracted fire debris components is performed by gas chromatography mass spectrometry using the ILRC method described previously. The data file, total ion chromatogram and extracted ion chromatograms are uploaded to the Substrate database with the product name and description. The ILRC committee reviews and identifies the carbon range, determines the predominant ion profile, and identifies the major components. The committee also determined whether a substrate material produced any interfering products similar to an ignitable liquid. If so, it was classified according to ASTM E1618.

Quality Control

Quality control procedures are performed on all aspects of the Ignitable Liquids Reference Collection Database and the Substrate Database. A flame test is performed to determine whether a potential liquid is ignitable prior to being incorporated into the collection. On a daily basis the instrument is tuned and a fire debris standard is analyzed to determine instrument performance. Control charts are kept on the retention times of n-octane and n-eicosane from the fire debris standard to confirm the instrument method remains within the established limits. Sample preparation and analysis of each ignitable liquid is performed twice. Sample preparation, extraction, and subsequent analysis of the substrate materials are performed only once since the burning process is inherently unpredictable. All of the data uploaded into the database is reviewed by the ILRC committee during the classification process. The classification process includes determining the carbon range, predominant ion profile, identification of major components, and the ASTM classification of any ignitable liquid present. This process is performed by two committee members, two committee chairs, and reviewed by NCFS personnel.

ILRC Maintenance and Expansion Results

Ignitable Liquids Reference Collection and Database

Maintenance and expansion of the Ignitable Liquids Reference Collection (ILRC) was accomplished by obtaining liquids throughout the United States and Canada from the collections of forensic laboratories performing fire debris analysis. There were 120 liquids analyzed and uploaded into the database from January 2009 to the present bringing the total number of ignitable liquids in the database to 614. The ILRC committee reviewed and classified 90 of the 120 liquids which were then placed in the public section of the database. There were a total of 277,713 hits on the database from January 2009 through June 2011 with a total of 683,816 since September 1, 2007.

Table 1: Summary of ILRC maintenance and expansion progress during the project period.

ILRC	January – June 2009	July – December 2009	January – June 2010	July – December 2010	January – June 2011	Total
Liquids Analyzed	40	10	0	30	40	120
Database Entries	30	20	10	20	10	90
Database Hits (since 9/1/2007)	72,381 (478,484)	85,974 (564,458)	78,099 (642,557)	35,926 (678,483)	5,333 (683,816)	277,713
Liquids Sold	2,254	672	1,373	532	1,617	6,448

The count of database hits prior to September 1, 2007 (new database) is inaccurate or unknown. A total of 6,448 reference ignitable liquids were sold at minimal cost during this period. Since 2002, there have been 181 sales of 15,853 reference ignitable liquids to 88 customers in 42 states within the United States and six international countries, see figure 2.

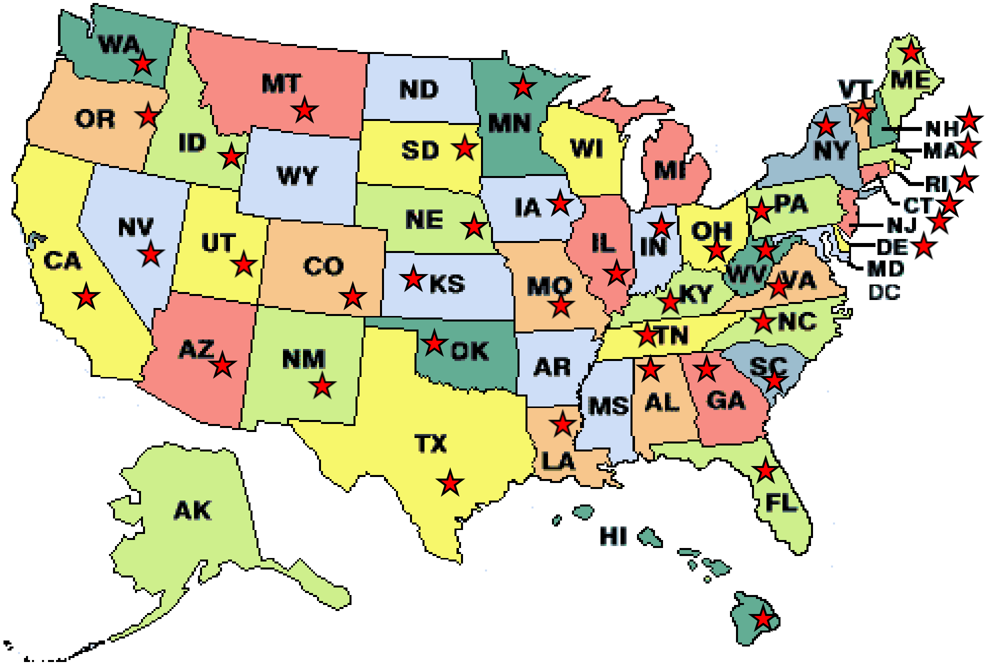
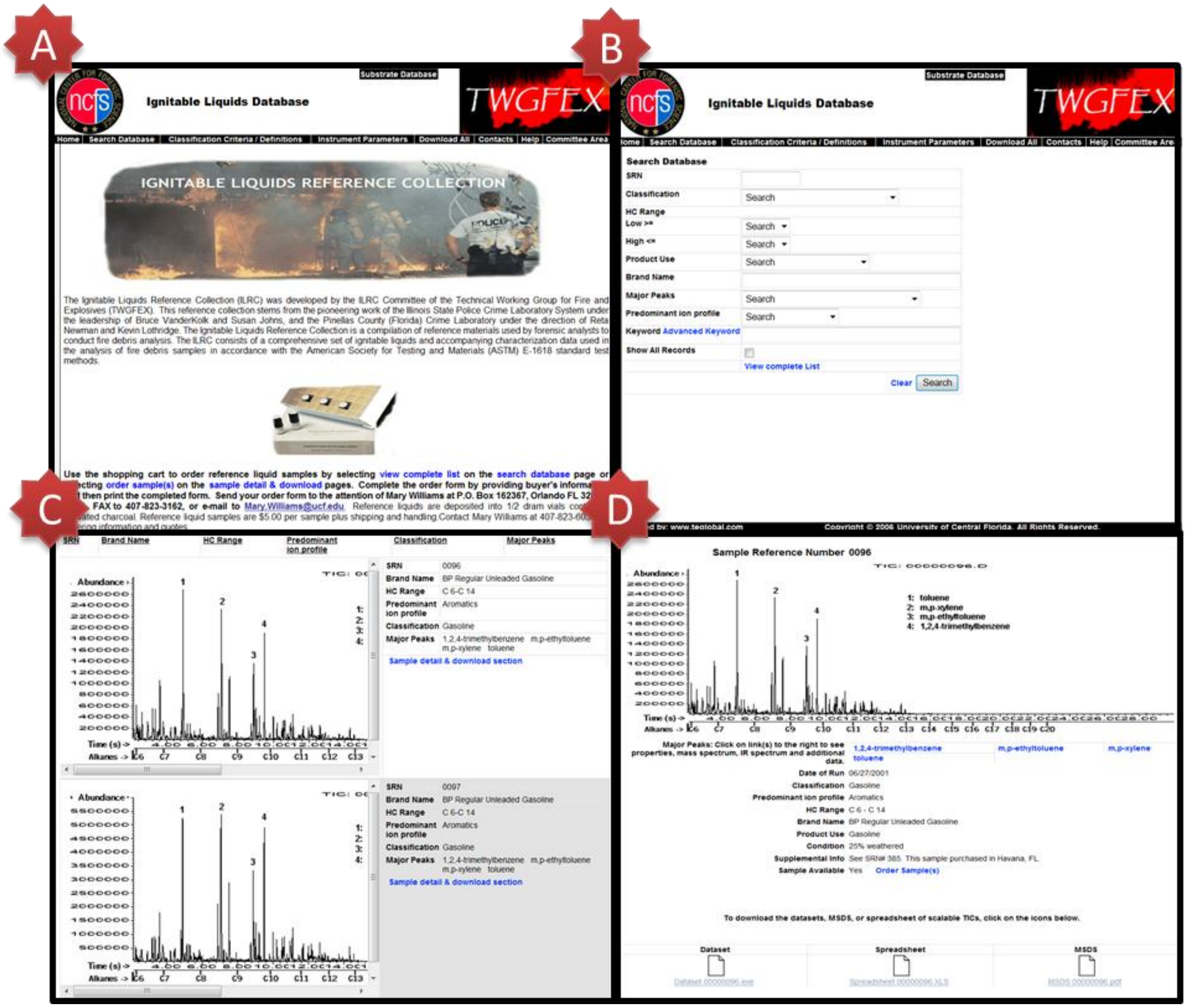


Figure 2: The 42 states within the United States from which the 88 ILRC customers reside.

The Ignitable Liquids Reference Collection Database was designed primarily by ILRC committee members who are practicing fire debris analysts. The design enables a fire debris analyst to search the database using parameters typically evaluated when utilizing the ASTM E1618. The user may search all of the parameters; classification, carbon range, product use, brand name, major peaks (components), and predominant ion profile or select just a few parameters. The search results provide the user a list of all reference ignitable liquids meeting the criteria requested in the search. An image of the total ion chromatogram (TIC) is provided with some information in the search results. The user can acquire more information and download the data files from the sample detail and download page of the desired reference ignitable liquid. Figure 2 shows screenshot of the various portions of the Ignitable Liquids Reference Collection Database.



The Ignitable Liquids Reference Collection (ILRC) was developed by the ILRC Committee of the Technical Working Group for Fire and Explosives (TWGFEX). This reference collection stems from the pioneering work of the Illinois State Police Crime Laboratory System under the leadership of Bruce VanderKolk and Susan Johns, and the Pinellas County (Florida) Crime Laboratory under the direction of Reta Newman and Kevin Lothridge. The Ignitable Liquids Reference Collection is a compilation of reference materials used by forensic analysts to conduct fire debris analysis. The ILRC consists of a comprehensive set of ignitable liquids and accompanying characterization data used in the analysis of fire debris samples in accordance with the American Society for Testing and Materials (ASTM) E-1618 standard test methods.

Use the shopping cart to order reference liquid samples by selecting view complete list on the search database page or …cting **order sample(s) on the sample detail & download pages. Complete the order form by providing buyer's informa…** …**then print the completed form. Send your order form to the attention of Mary Williams at P.O. Box 162367, Orlando FL 32…** … **FAX to 407-823-3162, or e-mail to** Mary.Williams@ucf.edu. Reference liquids are deposited into 1/2 dram vials co… …ated charcoal. Reference liquid samples are $5.00 per sample plus shipping and handling Contact Mary Williams at 407-823-60…

Search Database

SRN	
Classification	Search
HC Range	
Low >=	Search
High <=	Search
Product Use	Search
Brand Name	
Major Peaks	Search
Predominant ion profile	Search
Keyword Advanced Keyword	
Show All Records	☐
	View complete List

Clear Search

SRN	Brand Name	HC Range	Predominant ion profile	Classification	Major Peaks

SRN	0096
Brand Name	BP Regular Unleaded Gasoline
HC Range	C 6-C 14
Predominant ion profile	Aromatics
Classification	Gasoline
Major Peaks	1,2,4-trimethylbenzene m,p-ethyltoluene m,p-xylene toluene

Sample detail & download section

SRN	0097
Brand Name	BP Regular Unleaded Gasoline
HC Range	C 6-C 14
Predominant ion profile	Aromatics
Classification	Gasoline
Major Peaks	1,2,4-trimethylbenzene m,p-ethyltoluene m,p-xylene toluene

Sample detail & download section

Sample Reference Number 0096

1: toluene
2: m,p-xylene
3: m,p-ethyltoluene
4: 1,2,4-trimethylbenzene

Major Peaks: Click on link(s) to the right to see properties, mass spectrum, IR spectrum and additional data.	1,2,4-trimethylbenzene m,p-ethyltoluene m,p-xylene toluene
Date of Run	06/27/2001
Classification	Gasoline
Predominant ion profile	Aromatics
HC Range	C 6 - C 14
Brand Name	BP Regular Unleaded Gasoline
Product Use	Gasoline
Condition	25% weathered
Supplemental Info	See SRN# 385. This sample purchased in Havana, FL.
Sample Available	Yes Order Sample(s)

To download the datasets, MSDS, or spreadsheet of scalable TICs, click on the icons below.

Dataset	Spreadsheet	MSDS
Dataset 00000096.exe	Spreadsheet 00000096.XLS	MSDS 00000096.pdf

d by: www.tealobal.com Copyright © 2006 University of Central Florida. All Rights Reserved.

Figure 3: Ignitable Liquids Reference Collection Database; A) Home page, B) Search page, C) Search Results page, D) Sample Detail and Download page.

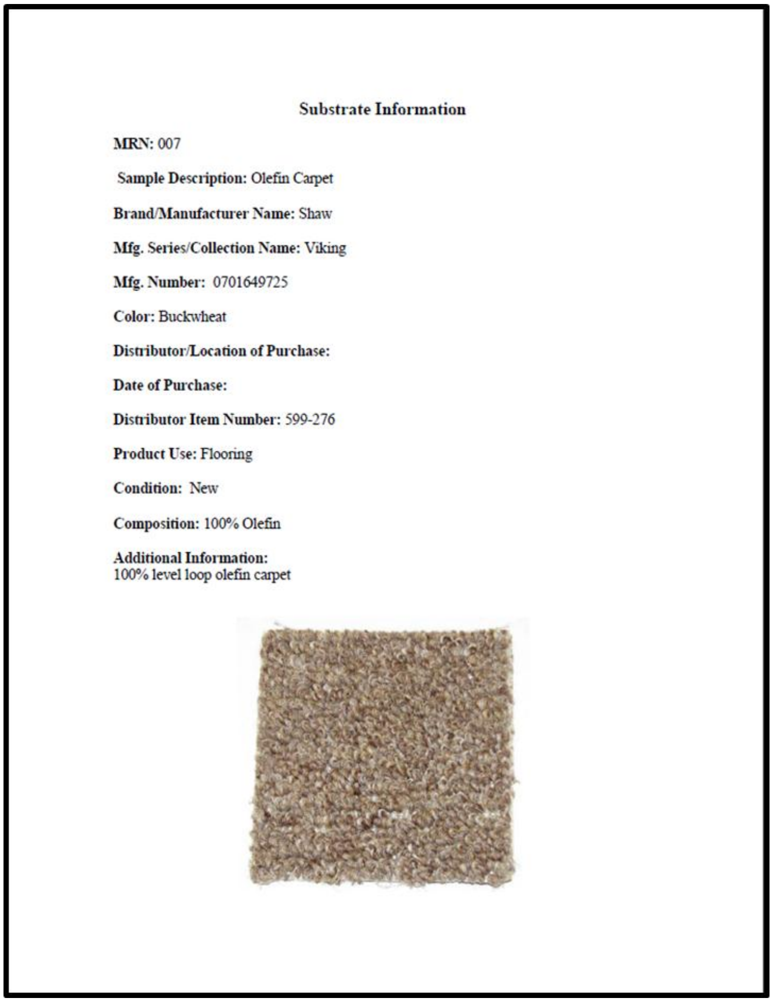
Substrate Database

A timeline for the completion of project goals was established and reported to the ILRC committee in November 2008. NCFS and ILRC committee discussed the design of the new Substrate Database. The requirements and functionality of the database were communicated to the database designer Tony Elmiger of TeGlobal. A prototype of the database was available for review on March 1, 2009 on the TeGlobal server. One hundred entries were uploaded into the prototype database consisting un-burned and burned debris from 50 materials. Committee members and NCFS personnel met in Atlanta with Tony Elmiger on May 4th and 5th, 2009 to discuss modifications to the database. The requested modifications were completed within two weeks. The data in the database was edited to fit the modified database format. The committee assessed the functionality of the database to determine if any further modifications would be required. Committee members and NCFS personnel met in Orlando on September 14th to discuss any final changes to the prototype. No major modifications were required and the Substrate Database was transferred to NCFS server in October 2009. Committee members reviewed the data and information for the 100 entries in preparation for the public release of the database. The Substrate Database was released to the public on July 1st, 2010 with 60 database entries. The Substrate Database has a total of 212 entries with 100 entries reviewed and available for public viewing (112 in committee review). There have been 22,378 hits on the database.

Table 2: Summary of Substrate Database development

Substrate	January June 2009	July December 2009	January June 2010	July December 2010	January September 2011	Total
Database	Prototype 1st Meeting 100 entries	2nd Meeting Database design finalized Database transferred to NCFS	60 entries reviewed	Open to Public July 1st		
Materials Analyzed	100		34	6	72	212
Database Entries			60	20	20	100
Database Hits (total)				16,239	6,139	22,378

The Substrate Database was designed to emulate the Ignitable Liquids Reference Collection Database with some modifications. The focus of the search in the Substrate Database is centered on the product information rather than the ASTM E 1618 classification of the material which is for ignitable liquids. The major search parameters in the Substrate Database are material use class, material use sub-class, material composition, and liquid present rather than the ASTM E 1618 classification. The parameters typically evaluated when utilizing the ASTM E1618 such as ASTM E1618, carbon range, predominant profile, and identification of major peaks (components) are nevertheless still evaluated to determine whether the interfering products produced by the substrate material are similar to an ignitable liquid residue. The user may search all of the parameters or select just a few parameters. The search results provide the user a list of all reference substrate materials meeting the criteria requested in the search. An image of the total ion chromatogram (TIC) is provided with some information about the substrate material. The user can acquire more information and download the data files from the sample detail and download page of the desired reference substrate material. If the user desires more product information in order to purchase and analyze the material themselves, they can download the Substrate Information page, see figure 4. Figure 5 shows screenshot of the various portions of the Substrate Database.



Substrate Information

MRN: 007

Sample Description: Olefin Carpet

Brand/Manufacturer Name: Shaw

Mfg. Series/Collection Name: Viking

Mfg. Number: 0701649725

Color: Buckwheat

Distributor/Location of Purchase:

Date of Purchase:

Distributor Item Number: 599-276

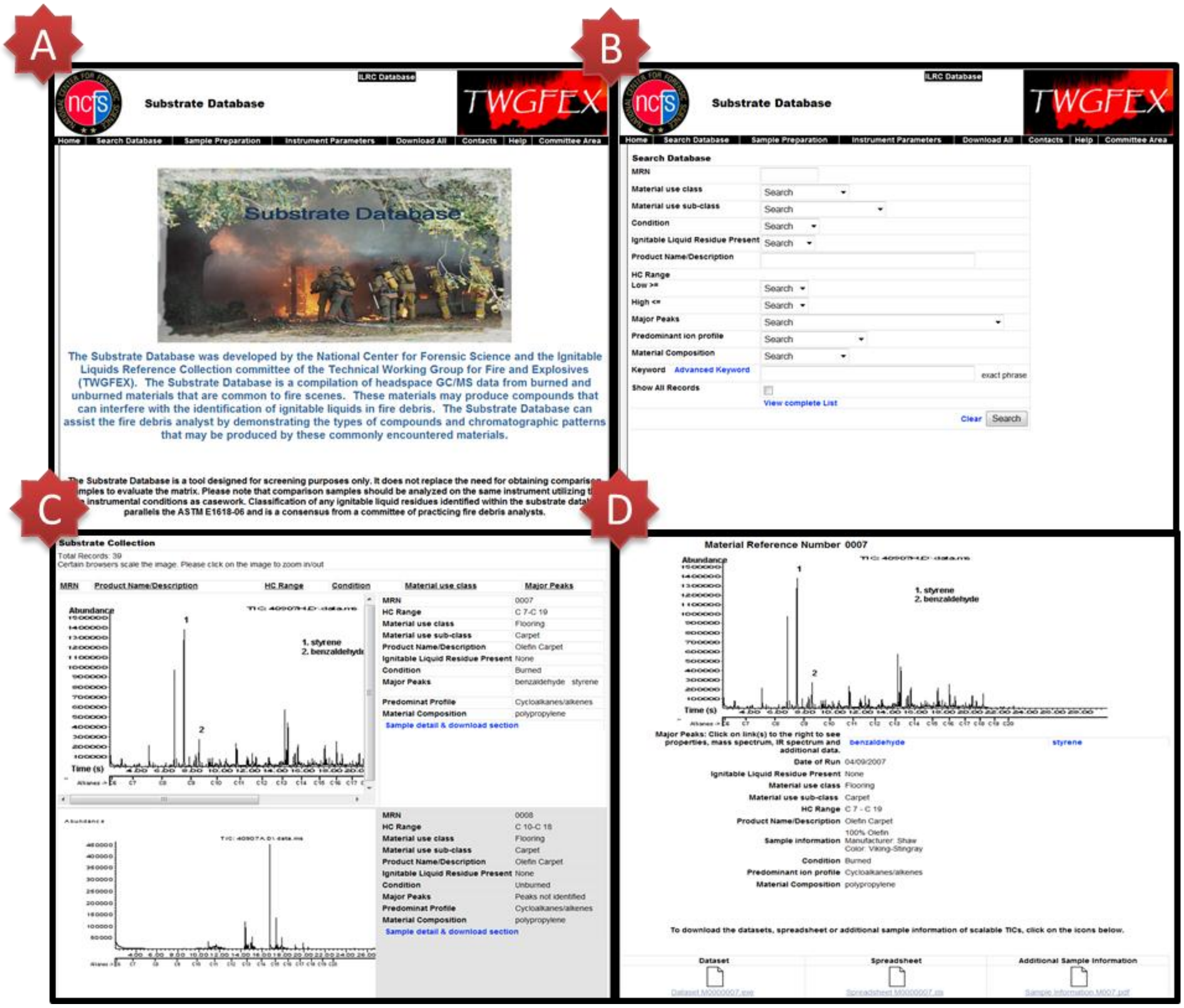
Product Use: Flooring

Condition: New

Composition: 100% Olefin

Additional Information:
100% level loop olefin carpet

Figure 4: Substrate Information page



The Substrate Database was developed by the National Center for Forensic Science and the Ignitable Liquids Reference Collection committee of the Technical Working Group for Fire and Explosives (TWGFEX). The Substrate Database is a compilation of headspace GC/MS data from burned and unburned materials that are common to fire scenes. These materials may produce compounds that can interfere with the identification of ignitable liquids in fire debris. The Substrate Database can assist the fire debris analyst by demonstrating the types of compounds and chromatographic patterns that may be produced by these commonly encountered materials.

e Substrate Database is a tool designed for screening purposes only. It does not replace the need for obtaining comparis
mples to evaluate the matrix. Please note that comparison samples should be analyzed on the same instrument utilizing t
instrumental conditions as casework. Classification of any ignitable liquid residues identified within the substrate data
parallels the ASTM E1618-06 and is a consensus from a committee of practicing fire debris analysts.

Search Database

MRN	
Material use class	Search
Material use sub-class	Search
Condition	Search
Ignitable Liquid Residue Present	Search
Product Name/Description	
HC Range Low >=	Search
High <=	Search
Major Peaks	Search
Predominant ion profile	Search
Material Composition	Search
Keyword Advanced Keyword	exact phrase
Show All Records	☐ View complete List
	Clear Search

Substrate Collection

Total Records: 39

Certain browsers scale the image. Please click on the image to zoom in/out

MRN Product Name/Description HC Range Condition Material use class Major Peaks

MRN	0007
HC Range	C 7-C 19
Material use class	Flooring
Material use sub-class	Carpet
Product Name/Description	Olefin Carpet
Ignitable Liquid Residue Present	None
Condition	Burned
Major Peaks	benzaldehyde styrene
Predominat Profile	Cycloalkanes/alkenes
Material Composition	polypropylene

Sample detail & download section

MRN	0008
HC Range	C 10-C 18
Material use class	Flooring
Material use sub-class	Carpet
Product Name/Description	Olefin Carpet
Ignitable Liquid Residue Present	None
Condition	Unburned
Major Peaks	Peaks not identified
Predominat Profile	Cycloalkanes/alkenes
Material Composition	polypropylene

Sample detail & download section

Material Reference Number 0007

Major Peaks: Click on link(s) to the right to see properties, mass spectrum, IR spectrum and additional data.	benzaldehyde styrene
Date of Run	04/09/2007
Ignitable Liquid Residue Present	None
Material use class	Flooring
Material use sub-class	Carpet
HC Range	C 7 - C 19
Product Name/Description	Olefin Carpet
Sample information	100% Olefin Manufacturer: Shaw Color: Viking-Stingray
Condition	Burned
Predominant ion profile	Cycloalkanes/alkenes
Material Composition	polypropylene

To download the datasets, spreadsheet or additional sample information of scalable TICs, click on the icons below.

Dataset	Spreadsheet	Additional Sample Information
Dataset M0000007.exe	Spreadsheet M0000007.xls	Sample Information M007.pdf

Figure 5: Substrate Database; A) Home page, B) Search page, C) Search Results page, D) Sample Detail and Download page.

ILRC Maintenance and Expansion Conclusions

The development and maintenance of the Ignitable Liquids Reference Collection and Database have been a worthwhile and appreciated source of reference ignitable liquid GC-MS analytical data, ASTM E 1618 classification information and reference materials for fire debris analysts in the past nine years. An additional 120 reference ignitable liquids were incorporated into the collection expanding the number of database entries to 614 making the ILRC the largest collection of reference ignitable liquids with an associated database of analytical data. The collection contains reference ignitable liquids spanning all ASTM E1618 classes and carbon ranges from various U.S. geographical areas and time frames. Ignitable liquids from other countries were desirable, but, transportation regulations make it prohibitive to ship hazardous materials internationally. An alternative approach was to gather the GC-MS data from reference ignitable liquids obtained and analyzed from laboratories in other countries, however, there was a consensus from European nations not to pursue a European database.[5] This work has inspired the forensic fire debris community to request development of a database consisting of weathered and bacterial degraded ignitable liquids. NCFS and T/SWGFEX have been awarded funding for a Degraded Ignitable Liquids Database to study weathering and bacterial degradation on chromatographic patterns of ignitable liquids and incorporate this data into a database (2011DNBXK539).

The Substrate Database benefits the fire debris analyst in their evaluation of whether the residue extracted from fire debris evidence contains an ignitable liquid and in the classification of any ignitable liquid present. Many building materials and furnishings decompose to yield combustion and pyrolysis products that are concurrently extracted with any ignitable liquid residue. Having a reference data collection of both ignitable liquids and substrate materials that have undergone heating aides the analyst in determining which chromatographic patterns can be attributed to the ignitable liquid or the substrate material. Conditions of a fire scene and subsequent suppression efforts vary the components and their relative ratios within fire debris; therefor the database contains representative fire debris and doesn’t endeavor to represent all possible fire debris samples. The materials and subsequent fire debris sample data The Substrate Database was developed to search parameters employed by fire debris analysts evaluating fire debris according to ASTM E1618 and populated with substrate materials commonly found in fire debris. On July 1, 2010, the database was available to the forensic community with 60 entries. Currently, there are 50 substrates materials both unburned and burned for a total of 100 database entries in the public section of Substrate Database. There are another 112 entries being reviewed by the ILRC committee that will bring the total to 212 entries.

ILRC Maintenance and Expansion References

[1] ASTM E 1618-10 “Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry,” ASTM International, West Conshohocken, PA, 2010.

[2] Allen, Stephen P., Case, Stuart W., Survey of Forensic Science Laboratories by the Technical Working Group for Fire and Explosions (TWGFEX), Forensic Science Communications, 2(1), Jan 2000.

[3] Stauffer, E., Basic Concept of Pyrolysis for Fire Debris Analysts, Science and Justice, 43(1), p 29-40.

[4] ASTM E1412-07 “Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration,” ASTM International, West Conshohocken, PA, 2007.

[5] Personal communication from a member of European Network of Forensic Science Institutes.

Dissemination of ILRC Maintenance and Expansion Research Findings

ILRC Database: http://ilrc.ucf.edu/

Substrate Database: http://ilrc.ucf.edu/substrate/

In February 2011 at the American Academy of Forensic Sciences in Chicago, NCFS presented a poster regarding the ILRC and Substrate Databases developed and maintained by the National Center for Forensic Science and the Technical/Scientific Working Group for Fire and Explosions.

In September 2011 at the American Society of Crime Laboratory Directors in Denver, NCFS presented a poster regarding the ILRC and Substrate Databases developed and maintained by the National Center for Forensic Science and the Technical/Scientific Working Group for Fire and Explosions.

Development and population of the Substrate Database supported the thesis research of a student at the University of Central Florida in receiving a Master of Science in Forensic Science.

Smokeless Powders Database

Smokeless Powder Database Introduction

Smokeless powders are combustible materials containing oxygen which do not detonate, but actually deflagrate. Combustion of these propellants or low explosives produces gases which produce an explosion.[1] These solid propellants were designed to accelerate a projectile from its position of rest at the breech of a weapon to its full velocity as it exits the tube or barrel. Smokeless powders replaced black powder as a military propellant because black powder produced a dense black cloud upon firing the weapon. The adverse effects of black powder were the ease of locating the firing position and creating chaos and confusion on the battle field. Developments in the manufacturing of smokeless powders has been primarily to improve stability, decrease erosion of the weapon's barrel, control pressures, decrease the amount of smoke expelled, and decrease muzzle flash upon firing the weapon.[2] Smokeless powders are used in the manufacturing of military and civilian ammunition. In addition, they are sold in the retail market for sport and recreational purposes, primarily for reloading of ammunition. Smokeless powders may also be used to manufacture improvised explosive devices such as pipe bombs and as such are of interest to the law enforcement community.

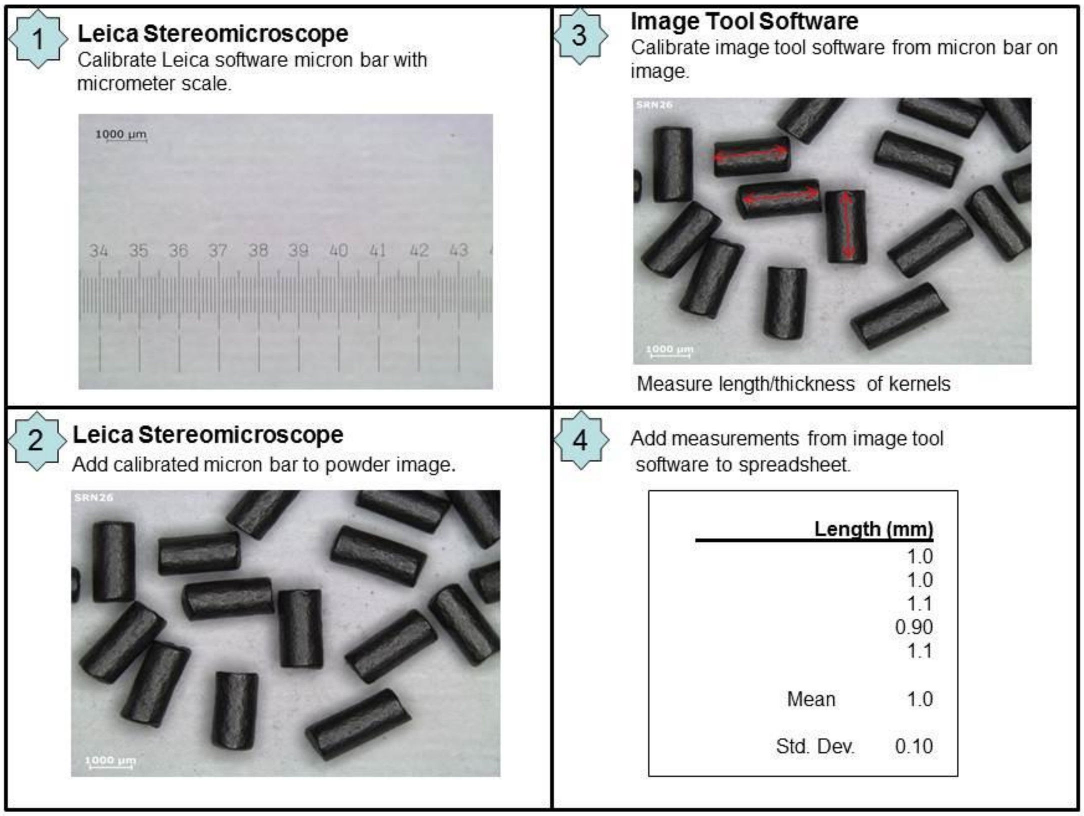
The National Research Council (NRC) issued a report "Black and Smokeless Powders: Technologies for Finding Bombs and the Bomb Makers" in 1998.[3] Key findings in the report include facts that 90% of the deaths and 80% if the injuries caused by pipe bombs containing black or smokeless powders occurred away from security screening points. This stresses the importance of the forensic investigation on determining the identity of the explosive material and the bomb maker. The report also emphasized that the materials recovered from the explosion scene such as unburned powder can aid in the identification of the product type and manufacturer provided that resources are available to the forensic scientist. Suitable resources include databases of products information, physical characteristics, analytical data, and spectral libraries for identification of the components. The report indicates the fact that existing databases of black and smokeless powders were used extensively in bombing investigations yet were incomplete. Also, only large federal organization such as the Federal Bureau of Investigation, the Bureau of Alcohol and Tobacco and Firearms, and the U.S. Postal Service had the resources to develop these databases. Smaller state and local laboratories did not have the resources, time, and manpower to develop their own databases and did not have access to existing databases.

The database was developed by the Explosives Database Committee of the Technical/Scientific Working Group for Fire and Explosions (T/SWGFEX) and the National Center for Forensic Science (NCFS) at the University of Central Florida where the database is maintained. The Explosives committee identified the analytical methods most commonly used in the analysis of smokeless powders and assisted NCFS with developing procedures for these analytical methods. Some design aspects and data incorporated in the Smokeless Powders Database are contributions from the Federal Bureau of Investigation. The database design is divided into three sections; product information, physical characteristics, and chemical composition. The user can search multiple criteria from all three sections of the database where the user can decide what image (powder, container, FTIR spectrum, or GC-MS TIC) returns with the list of search results. The database is open access and is provided as a free service to the forensic science community. Since the intent of a smokeless powders database was for investigative purposes, the Explosives Database committee and NCFS decided a collection of reference samples available for purchase was not necessary.

Smokeless Powder Database Methods

Three analytical methods are utilized to examine the smokeless powders. Attenuated Total Reflectance Fourier Transform Infrared (ATR-FTIR) Spectroscopy and Gas Chromatograph – Mass Spectrometry (GC-MS) are the two chemical analysis methods and micrometry is the only physical analysis method. Since the smokeless powder data is a contribution from more than one source, not all methods are utilized and the methods vary in sample preparation and instrument parameters. For example, the data provided by the Federal Bureau of Investigation did not include FTIR data. The three analytical methods described here are those performed on smokeless powders obtained and analyzed by the National Center for Forensic Science (NCFS).

The only physical analysis method employed is micrometry, the physical measurement of the smokeless powder kernels by microscopy. Fifty smokeless powder kernels are placed on a white background and imaged at X10.1 magnification using a Leica S8APO stereomicroscope with a Leica DFC290 camera. The micron bar in the camera software is calibrated with a 50mm micrometer scale with 0.1mm divisions from Graticles, Ltd. (serial number PS50/124). ImageTool software is used to measure the length and thickness of each kernel in the micrograph. The software may be downloaded for free at http://ddsdx.uthscsa.edu/dig/itdesc.html. The Image Tool software measuring tool is calibrated using the micron bar in the micrograph. Calibration of the micron bar is done at an image zoom of 2:1 and the kernel measurements are performed at an image zoom of 1:1. Measurements are made in millimeters rounded to one decimal place. The average and standard deviation of the measurements are calculated and the maximum and minimum measurements noted, see figure 6.



Leica Stereomicroscope
Calibrate Leica software micron bar with micrometer scale.

Image Tool Software
Calibrate image tool software from micron bar on image.

Measure length/thickness of kernels

Leica Stereomicroscope
Add calibrated micron bar to powder image.

Add measurements from image tool software to spreadsheet.

	Length (mm)
	1.0
	1.0
	1.1
	0.90
	1.1
Mean	1.0
Std. Dev.	0.10

Figure 6: Steps in measuring smokeless powder kernels; 1) calibrate stereomicroscope software with micrometer scale, 2) add micron bar to image, 3) calibrate Image Tool Software using micron bar, 4) make measurements.

The chemical analysis method ATR-FTIR measures the absorption of energy due to fundamental molecular vibrations. This chemical analysis is commonly employed to detect nitrocellulose, the most abundant compound in the smokeless powder to confirm that the material is in fact a smokeless powder. Two types of sample preparation were studied; inner portion of kernel or creating a film constituting all of the powder components. Contact of the internal reflecting element (crystal) with the smokeless powder kernel was problematic; a more affective sample preparation was to disperse the kernel components using a solvent. It was determined that the most affective solvent for dispersing the kernel components was acetone. Sample preparation consists of placing 80 µl of acetone in approximately 2.5 mg of smokeless powder kernels for 30 minutes. The resulting supernatant is spotted onto a microscope slide slowly allowing the solvent to evaporate to form a film. Analysis of the smokeless powder film is conducted on an ATI Mattson Infinity Series FTIR with Spectra Tech IR Plan Advantage IR microscope. To perform attenuated total reflectance absorbance measurements, the silicon internal reflectance element contacts the smokeless powder film deposited onto the microscope slide. The spectra are an average of 32 scans at a resolution of 4 cm^{-1}. An ATR correction is applied using the OMNIC software which corrects for the variance in the penetration depth. The spectra are collected in absorbance, but viewed in percent transmittance in the database, see figure 7.

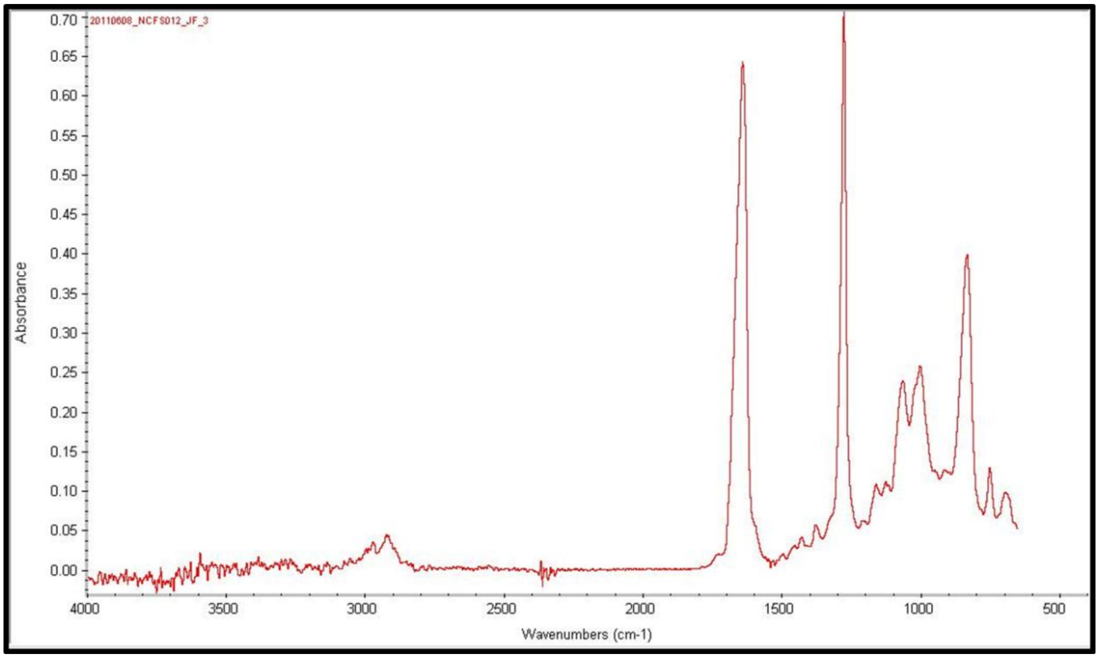


Figure 7: FTIR-ATR spectrum of a smokeless powder in absorbance.

The second chemical analysis method is GC-MS which separates the components of the mixture and then fragments the molecules of each component into ions (m/z) which are subsequently detected. The total ion chromatogram displays the components of the smokeless powders as peaks eluted in time with a relative abundance. Various methods for the extraction of the volatile chemical components from the carbon and nitrocellulose of the smokeless powders were investigated. Initially, acetone was used in differing volumes and extraction time periods since it disperses the smokeless powder kernel components. Unfortunately, eliminating nitrocellulose completely from the solution is not possible. Nitrocellulose is not volatile therefor it will eventually obstruct the injection port liner on the GC-MS. The alternative solvent investigated was methylene chloride since all of the volatile components are soluble in this solvent. Unfortunately, methylene chloride doesn't disperse the smokeless powder kernels making it uncertain whether all of the components are extracted. Further studies using methylene chloride included bisecting the kernels and varying the extraction time period to confirm all of the volatile components were extracted. Sample preparation consists of cutting 10 mg of smokeless powder kernels to expose the inner portion of the kernel. Research indicates that cutting the kernels necessary for the optimal extraction of the analytes. The analytes are extracted with 300 μl of methylene chloride with 10 ppm undecane as an internal standard. Complete extraction requires three hours.

The supernatant and a 1:10 dilution of the supernatant are analyzed using a Hewlett-Packard 6890 gas chromatograph interfaced to a 5973 mass spectrometer. Various injection temperatures were investigated to ensure all of the analytes eluted onto the column and did not decompose in the injection port. The initial oven temperature was modified based on the extraction solvent used in the sample preparation. The solvent delay on the mass spectrometer was also adjusted based on the solvent utilized in the method. Other parameters studied were the oven temperature ramp rate and oven final temperature of the gas chromatograph, the interface temperature between the gas chromatograph and mass spectrometer as well as the mass range of the mass spectrometer. The final GC-MS method consisted of injecting 1 μl of smokeless powder extract into a 170°C injection port of the gas chromatograph where helium gas at a constant flow rate of 1.2 ml/min and a velocity of 40 cm/sec passes the vaporized analytes onto the column. The analytes are separated on a 30 m capillary column with a 5% phenyl methyl siloxane film of 0.25 μm thickness and a nominal diameter of 250 μm. An initial oven temperature of 40°C is held for 1 minute, followed by a temperature ramp of 25°C/min to a final temperature of 280°C, which is held for three minutes. The transfer line from the gas chromatograph to the electron ionization mass spectrometer is at 250°C, the ionization source is at 230°C and the quadruple is at 150°C. The mass spectrometer scans 2 -3 scans per second over a scan range of 43 to 400 m/z. The ionization source remains off for the first 4 minutes during solvent elution. The analytes contained in smokeless powders are denoted as peaks in the total ion chromatogram shown in figure 8. Analyte identification criteria are; signal to noise ratio of 3 to 1, the presence of at least three major ions and the retention time must be the same as the standard. A standard of the most common analytes was analyzed prior to sample analysis as a quality assurance. Table 3 lists the most common analytes within smokeless powders.

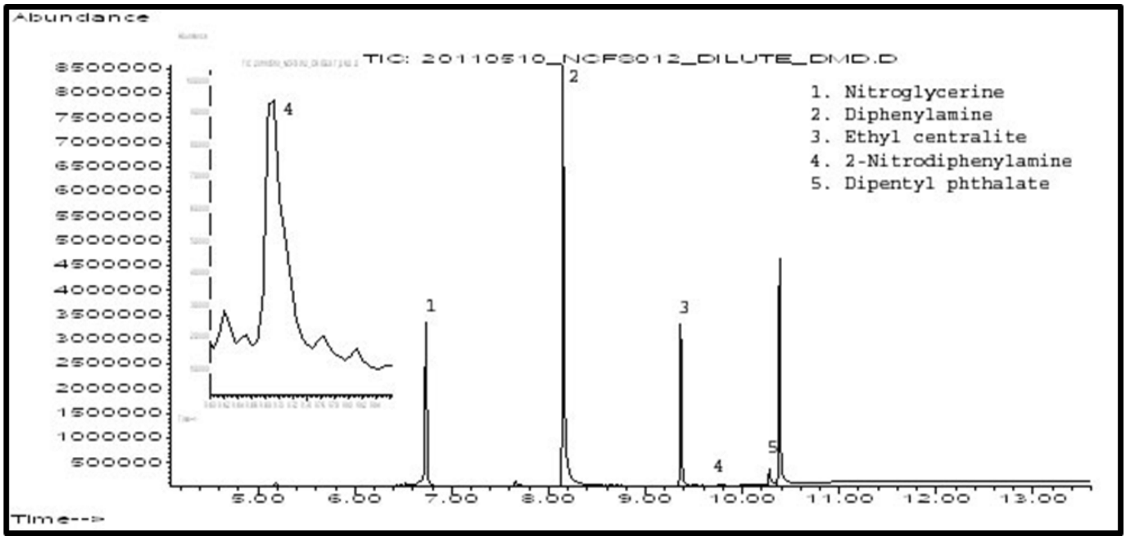


Figure 8: GC-MS total ion chromatogram of smokeless powder.

Table 3: List of analytes contained in GC-MS standard for the analysis of smokeless powders.

Analyte	CAS number	Mass to Charge	% Abundance
Nitroglycerin	55-63-0	46	100
		76	15
		43	13
Camphor	21368-68-3	95	100
		81	64
		108	41
Dibutyl Phthalate	84-74-2	149	100
		223	6
		205	5
Diethyl Phthalate	84-66-2	149	100
		177	26
		105	7
Dipentyl Phthalate	131-18-0	149	100
		150	9
		237	8
Diphenylamine	122-39-4	169	100
		84	10
		51	7
Dioctyl Phthalate	117-81-7	149	100
		167	35
		57	17
Ethyl Centralite	85-98-3	120	100
		148	81
		268	46

Methyl Centralite	611-92-7	134	100
Analyte	CAS number	Mass to Charge	% Abundance
		240	62
		106	45
2,4-Dinitrotoluene	121-14-2	165	100
		89	50
		63	27
4-Nitrodiphenylamine	836-30-6	214	100
		167	83
		184	26
2-Nitrodiphenylamine	119-75-5	214	100
		167	82
		180	37
4-Nitrosodiphenylamine	156-10-5	167	100
		198	72
		184	12

Smokeless Powder Database Results

A questionnaire was sent out to members of the T/SWGFEX explosives database committee to determine which analytical methods were most commonly used in the forensic laboratories analyzing smokeless powders. The consensus of the questionnaire was visual and microscopic examination for morphology, FTIR and GC-MS. The first meeting to discuss the Smokeless Powders Database was May 20th and 21st of 2009, those attending were NCFS personnel, members of the explosives database committee, and Tony Elmiger the database designer from TeGlobal. Database design and functionality would be provided and tested by the committee while population, maintenance, and analysis of smokeless powders would be accomplished by NCFS. The database would be designed into three major sections; powder information, physical description and chemistry. A statement of work was created for the database designer who then provided a quote. The prototype was available for review in July 2009 with 100 entries provided by the Federal Bureau of Investigation. NCFS began to develop micrometry, FTIR-ATR, and GC-MS methods for the analysis of smokeless powders with the assistance of two University of Central Florida Chemistry doctoral students.

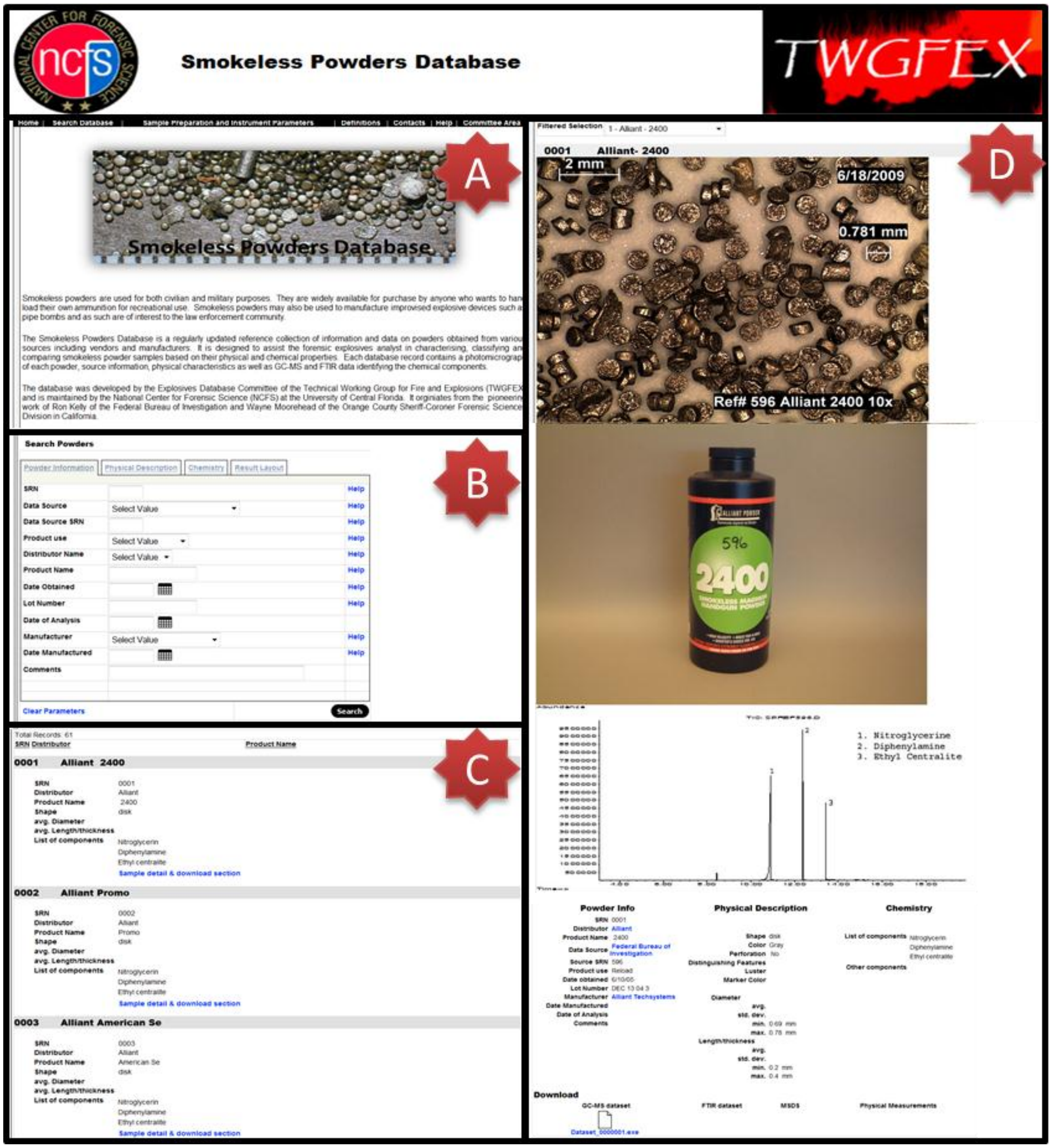
A second meeting was held on September 14th, 2009 to discuss the development of the analytical methods and any modifications required to the database prototype. Requested modifications to the prototype were completed by January 1, 2010. In 2010, the database prototype and the analytical methods were continually reviewed and modified. Final modifications to the prototype were completed on the TeGlobal server and the final database was transferred to the NCFS server. The Smokeless Powders Database was released January 1, 2011 with 100 entries and can be viewed at http://www.ilrc.ucf.edu/powders. In 2011, development of the three analytical methods was completed and standard operating procedures were written. NCFS has obtained 87 smokeless powders which have been analyzed and the data is the process of being added to the database. Ron Kelly from the FBI has provided product information and analytical data for an additional 650 smokeless powders from their agency database. An overview of the work accomplished in developing and populating the Smokeless

Powders Database is in table 4. As of September 15th, 2011 there have been 2,421 database hits.

Table 4: Goals met in developing and populating the Smokeless Powders Database.

2009	Questionnaire about smokeless powder analytical methods First meeting Database prototype with 100 entries Second meeting Analytical methods developed
2010	Modifications to database prototype Modifications to analytical methods Database finalized and transferred to NCFS
2011	Smokeless Powders Database launched January 1st Analytical methods finalized Standard operating procedures written NCFS obtained 87 smokeless powders Analyzed 87 smokeless powders Received information and data for 650 smokeless powders from the FBI

The Smokeless Powders Database was designed primarily by the Explosive Database committee members who are practicing explosives analysts. The database consists of four major areas; a home, search powders, search results, and sample detail and download page as shown in figure 9. The home page provides general information about the development and purpose of the database. The search powders function is divided into four sections; product information, physical description, chemistry, and results layout as shown in figure 10. Product information includes the source of the information and data, product use, product name, distributor, manufacturer, lot number, date manufactured, date obtained, and date of analyses. Physical description includes powder shape, color, luster, distinguishing features, marker color, perforation, and physical measurements. The chemistry section includes the presence of components detected by GC-MS. Result Layout allows the user to choose which image to return in the search results; GC-MS TIC, powder, canister, FTIR spectrum or no image. The Search Result lists those powders meeting the search criteria and layout chosen by the user. The list of results furnishes the image, sample reference number, product name, distributor, shape, measurements, and a list of components. The Sample Detail and Download section provides additional images, information about the smokeless powder and downloadable data files.



Smokeless Powders Database

Home | Search Database | Sample Preparation and Instrument Parameters | Definitions | Contacts | Help | Committee Area

Smokeless Powders Database

Smokeless powders are used for both civilian and military purposes. They are widely available for purchase by anyone who wants to han load their own ammunition for recreational use. Smokeless powders may also be used to manufacture improvised explosive devices such a pipe bombs and as such are of interest to the law enforcement community.

The Smokeless Powders Database is a regularly updated reference collection of information and data on powders obtained from variou sources including vendors and manufacturers. It is designed to assist the forensic explosives analyst in characterising, classifying an comparing smokeless powder samples based on their physical and chemical properties. Each database record contains a photomicrograp of each powder, source information, physical characteristics as well as GC-MS and FTIR data identifying the chemical components.

The database was developed by the Explosives Database Committee of the Technical Working Group for Fire and Explosions (TWGFEX and is maintained by the National Center for Forensic Science (NCFS) at the University of Central Florida. It orginiates from the pioneerin work of Ron Kelly of the Federal Bureau of Investigation and Wayne Moorehead of the Orange County Sheriff-Coroner Forensic Science Division in California.

Search Powders

Powder Information | Physical Description | Chemistry | Result Layout

SRN		Help
Data Source	Select Value	Help
Data Source SRN		Help
Product use	Select Value	Help
Distributor Name	Select Value	Help
Product Name		Help
Date Obtained		Help
Lot Number		Help
Date of Analysis		
Manufacturer	Select Value	Help
Date Manufactured		Help
Comments		

Clear Parameters   Search

Total Records: 61

SRN Distributor   Product Name

0001 Alliant 2400

SRN	0001
Distributor	Alliant
Product Name	2400
Shape	disk
avg. Diameter	
avg. Length/thickness	
List of components	Nitroglycerin Diphenylamine Ethyl centralite

Sample detail & download section

0002 Alliant Promo

SRN	0002
Distributor	Alliant
Product Name	Promo
Shape	disk
avg. Diameter	
avg. Length/thickness	
List of components	Nitroglycerin Diphenylamine Ethyl centralite

Sample detail & download section

0003 Alliant American Se

SRN	0003
Distributor	Alliant
Product Name	American Se
Shape	disk
avg. Diameter	
avg. Length/thickness	
List of components	Nitroglycerin Diphenylamine Ethyl centralite

Sample detail & download section

Filtered Selection: 1 - Alliant - 2400

0001 Alliant- 2400

Powder Info

SRN	0001
Distributor	Alliant
Product Name	2400
Data Source	Federal Bureau of Investigation
Source SRN	596
Product use	Reload
Date obtained	6/10/05
Lot Number	DEC 13 04 3
Manufacturer	Alliant Techsystems
Date Manufactured	
Date of Analysis	
Comments	

Physical Description

Shape	disk
Color	Gray
Perforation	No
Distinguishing Features	
Luster	
Marker Color	
Diameter	
avg.	
std. dev.	
min.	0.69 mm
max.	0.78 mm
Length/thickness	
avg.	
std. dev.	
min.	0.2 mm
max.	0.4 mm

Chemistry

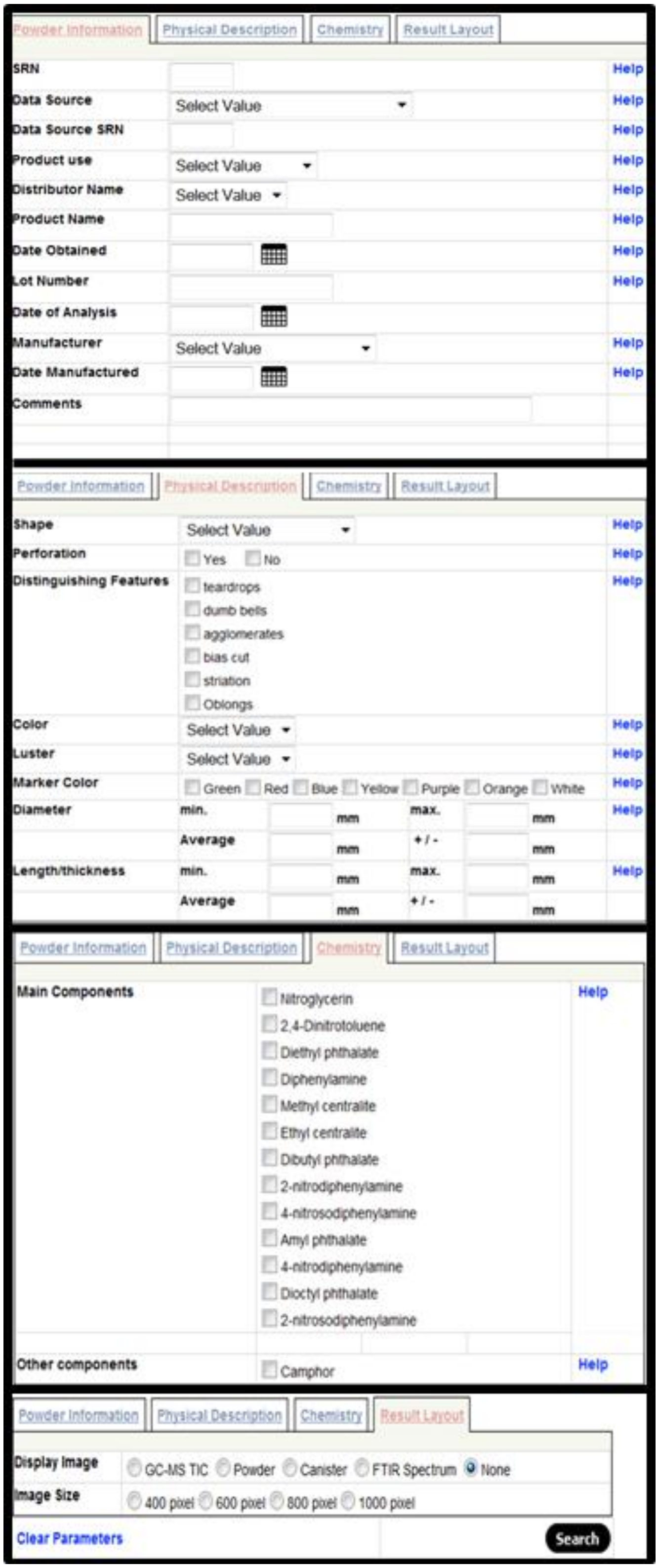
List of components	Nitroglycerin Diphenylamine Ethyl centralite
Other components	

Download

GC-MS dataset   FTIR dataset   MSDS   Physical Measurements

Dataset_0000001.exe

Figure 9: Four main areas of the Smokeless Powders Database, A) Home, B) Search powders, C) Search results, and D) Sample detail and download.



Display Image ○ GC-MS TIC ○ Powder ○ Canister ○ FTIR Spectrum ● None

Figure 10: Four main section of the search powders area; product information, physical description, chemistry, and results layout.

Smokeless Powder Database Conclusions

The Smokeless Powder Database is a regularly updated reference collection of powders obtained from various sources including vendors and manufacturers. Currently, the database contains information and analytical data of reloading smokeless powders with the intent to add OEM manufactured and military powders in the future. It is designed to assist the forensic explosives analyst in characterizing, classifying, and comparing smokeless powder samples based on their physical and chemical properties. Each database entry contains information about the powder, it physical characteristics, and chemical composition. Images of the commercial container, powder, FTIR-ATR spectrum, and GC-MS total ion chromatogram are also included in each database entry with micrometry measurements, FTIR-ATR data, and GC-MS data. The database will contain smokeless powder data and information from multiple sources. Each entry will identify the source (i.e. FBI, NCFS, etc.) of the data and information provided in the database along with the operating parameters of the analytical methods used to collect the data. The initial 100 entries were a contribution from the Federal Bureau of Investigation whose database is extensive and contains a significant amount of historical data and information on smokeless powders. NCFS will be contributing data and information from newer smokeless powders following the analytical methods described previously. In the future, contributions from the National Centre for Forensic Sciences in Ontario will be included in the database comprising smokeless powders distributed in Canada.

The database is open access and is provided as a free service to the forensic science community. It is a valuable tool for investigations of explosions caused by low explosives such as smokeless powders. The analytical methods developed will aid explosives analysts in forming consensus analytical procedures for the analysis of smokeless powders. The significant amount of data and information can be utilized in further research such as developing new analytical methods to characterize the smokeless powders and chemometric methods to provide a probability of a smokeless powder having certain physical and chemical characteristics.

Smokeless Powder Database References

[1] The Chemistry of Powder and Explosives, Davis, L., Tenney, CSG & Assoc., San Pedro, CA, 1943.

[2] Forensic Investigation of Explosions, Beveridge, Alexander, Taylor & Francis Inc., Bristol, PA, 1998.

[3] "Black and Smokeless Powders: Technologies for Finding Bombs and the Bomb Makers" Committee on Smokeless and Black Powders, National Research Council, National Academy Press, Washington, D.C. 1998.

Dissemination of Smokeless Powder Database Research Findings

Smokeless Powders Database: http://www.ilrc.ucf.edu/powders

In February 2011 at the American Academy of Forensic Sciences in Chicago, NCFS presented a poster regarding the Smokeless Powders Database developed and maintained by the National Center for Forensic Science and the Technical/Scientific Working Group for Fire and Explosions.

In May 2011 at the Florida Annual Meeting and Exposition in Palm Harbor Florida, one of the chemistry doctoral students presented a poster regarding the development of the smokeless powders analytical methods.

In September 2011 at the American Society of Crime Laboratory Directors in Denver, NCFS presented a poster regarding the Smokeless Powders Database developed and maintained by the National Center for Forensic Science and the Technical/Scientific Working Group for Fire and Explosions.

Implementation of a Searchable Ignitable Liquids Database Tool for the Fire Debris Analysis Community: Correction for Pyrolysis Products and Weathering

Searchable Ignitable Liquids Database Tool Introduction

Most fire debris analysis laboratories in the United States, and many laboratories in other countries, follow the ASTM E1618 Standard Method for the analysis of fire debris GC-MS data. The general procedure followed by these laboratories involves examining the fire debris sample in accord with the procedures outlined in E1618 to determine the presence of an ignitable liquid and arrive at a tentative ASTM classification. The ILRC is searchable by ASTM class, carbon range, etc. and the analyst must examine each of the entries having the assigned class to visually identify a chromatographic pattern that closely matches the fire debris sample's chromatographic profile. Before the ILRC was available online, this same process was used to analyze and classify samples; however, each laboratory had a local method for database organization and searching. The advent of the ILRC brought a common interface and database search process to all laboratories that were willing to utilize this online resource. Standardization of the organization and search routine was a step forward for fire debris analysis through a partnership between the forensic community and the NCFS. The ILRC search method has been in place since the system became available online in 2002. The goal of this project was to implement a computer-based search routine of reference ignitable liquids from multiple sources to facilitate the analysis of fire debris casework samples. The goal of the project does not encompass a way to circumvent the use E1618 and does not intend to replace this ASTM method, but rather, this project is intended to by synergistic with ASTM E1618.

The visual pattern recognition used by fire debris analysts for sample identification relies on chromatographic patterns, which vary significantly between laboratories. Peak alignment routines and retention indices offer potential methods to overcome the retention time variation challenge; however, variation in analytical column composition and differing oven temperature programs may lead to nonlinear relationships between retention times and significantly complicate the implementation of a strong electronic search routine. This project made use of an alternative approach which grew out of the NASA research which investigated the use of electron ionization for the identification of organic analytes. Research at NCFS has shown that the total ion spectrum (TIS), equivalent to the average mass spectrum across the chromatographic profile, is sufficient for the identification of complex mixtures, of the type that are indicative of many ignitable liquids.[1,2,3] Previous attempts at automated searching of databases include an early attempt by Chris Lennard and coworkers that was based on the use of target compounds;[4] however, as promising as this method appeared to be, it never achieved widespread use among the fire debris analysis community. Early attempts to utilize expert systems were also introduced by Bertch and coworkers.[5,6] These systems also failed to find their way into widespread use, which may be attributable to the general lack of computing power available in forensic laboratories at the time of their development. Rapid advances in computing power with the advent of multi-core processors, massive memory blocks, stable operating systems and fast internet connections have created a climate that is much more favorable to the deployment of computationally intensive automated search methods and the general acceptance and use of those systems in forensic fire debris analysis laboratories. The software developed under this project has been tested in three forensic laboratories (South Carolina Department of Law Enforcement, Columbia SC; Tucson Police Department, Tucson AZ; Dept of Public Saftey,Burlington, VT) during the development phase.

Searchable Ignitable Liquids Database Tool Methods

Ignitable liquid and substrate total ion spectra (TIS) comprising the two libraries are obtained from the GC-MS data files of the Ignitable Liquids Reference Collection database and the Substrate Database. Twenty microliters of ignitable liquid is diluted with 1000 µl of carbon disulfide. Decomposition products from the burning of the substrate materials are adsorbed onto activated carbon strips and then extracted with 1000 µl of carbon disulfide. The GC-MS method to analyze ignitable liquids and extracts from the decomposition of substrate materials consists of injecting 1 µl into a 250°C injection port of a gas chromatograph mass spectrometer at a split ration of 50:1. Helium gas at a constant flow rate of 0.8 ml/min and a linear velocity of approximately 36 cm/sec passes the vaporized ignitable liquid through a gas chromatographic column. The column is 25 meters in length with an inner diameter of 0.20 mm and a 0.5 micron film thickness of 100% polydimethylsiloxane. The initial oven temperature is held at 50°C for 3 minutes and then increased at a rate of 10°C/min to a final temperature of 280°C which is held for 4 minutes. The transfer line from the gas chromatograph to the electron ionization mass spectrometer is at 280°C, the ionization source is at 230°C and the quadruple is at 150°C. The mass spectrometer scans 2 -3 scans per second over a scan range of 30 – 350 m/z. The ionization source is turned off between 1.54 and 2.00 minutes during solvent elution.

The GC-MS data are exported in an AIA (CDF) file format containing the ion intensity of each mass / charge ratio at every mass scan (equates to time). Summing the ion intensity over all mass / charge ratios at each mass scan constitutes a total ion chromatogram (TIC). Total ion chromatograms are the most common form of GC-MS data displayed. Summing the ion intensity over all mass scans at each mass / charge ratio constitutes a total ion spectrum (TIS). A visual depiction of the data format is shown in figure 11. Each library consists of the total ion spectra of their respective reference materials; ignitable liquid or substrate. The total ion spectra are normalized (Z_N) to have a summed intensity of one. Comparison of the spectra is accomplished by a distance calculation. The distance calculation measures the difference in relative abundance between the respective mass to charge ion and then the differences are summed. Similarities between the spectra have been calculated since it is more intuitive to measure how similar the spectra are to one another. The various distance and similarity equations provided in the software are shown in table 5. In the case where a fire debris sample contains contributions from an ignitable liquid (IL) and contributions from the decomposition products of substrate material (SUB), Z_N can be written as a combination of the two contributing components, see equation 1. The sum of the multipliers *a* and *b* must be equal to one. This combination is easily achieved identifying the multipliers a and b by a maximum likelihood method (least squares). Each ignitable liquid in the library is combined with each pyrolysis sample and the mixture that is most similar to the fire debris sample is retained.

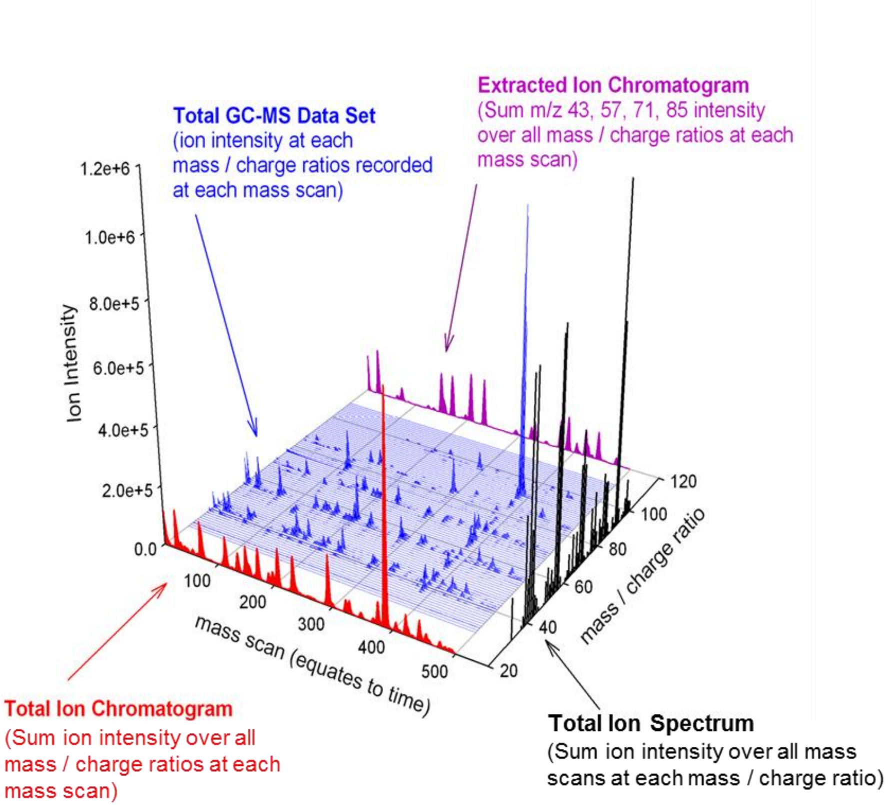


Figure 11: Depiction of GC-MS 3D data

Table 5: Distance and Similarity calculation in software

Minkowski Distance	NCFS Distance
$D = (\Sigma\lvert x_1 - x_2\rvert^p)^{1/p}$	$D = (\Sigma\lvert x_1 - x_2\rvert)/2$
Manhattan p = 1 Euclidean p = 2	
Minkowski distance boundaries	NCFS distance boundaries
[0, 2]	[0, 2]
Minkowski Similarity	NCFS Similarity
$S = 1/(1+D)$	$S = 1-D/D_{max} = 1-D \quad (D_{max} = 1)$
Minkowski similarity boundaries	NCFS similarity boundaries
[0.33, 2]	[0, 1]

$$Z_N = a*Z_{N,IL} + b*Z_{N,SUB} \qquad (1)$$

Searchable Ignitable Liquids Database Tool Results

Ignitable liquid residues extracted from fire debris are evaporated (weathered) compared to those ignitable liquids that are un-evaporated (un-weathered) in the library. The weathering of ignitable liquids and the effects of comparing weathered ignitable liquid residues to un-weathered ignitable liquids was studied. Sixty-five ignitable liquids from eight ASTM E1618 classes in various stages of weathering were compared to one another using the software. Similarities based on the NCFS distance equation were calculated for each comparison of the 65 ignitable liquids for a total of 2,080 comparisons. Systat 12, a statistical graphics software package (Cranes Software International, Ltd) was used to classify the ignitable liquids based on their calculated similarities. Hierarchical clustering using single linkage with a Euclidean distance clustered together those ignitable liquids with a high similarity. The letter preceding the ignitable liquid reference number indicates the ASTM E1618 classification of the liquid and the letter following indicates the percent weathering of the liquid; see table 6. Figure 12 is a graphical representation of the similarities between the 65 liquids. The diagonal of the graph is the color red representing a similarity of one; these are the comparisons of the ignitable liquids to themselves. Bordering the graph is a cluster tree diagram; another graph demonstrating the relative similarity of the ignitable liquids to one another. Both graphs demonstrate that ignitable liquids within a class are more similar to one another than to ignitable liquids of another class including the weathered liquids. The seven tables (tables 7 – 13) present the calculated similarities of the 65 liquids within each ASTM E1618 class. Calculated similarities between the un-weathered (0% weathered) and the 75% weathered ignitable liquids for the eight classes; gasoline 0.644 and 0.783, aromatic 0.887, HPD 0.876, isoparaffinic 0.922, LPD 0.928 and 0.843, MPD 0.913, 0.918, and 0.925, and naphthenic paraffinic 0.932 and 0.903. These similarities indicate that weathering of the ignitable liquid mostly affects the gasoline class. The simplest method of correcting for the differences between un-weathered and highly weathered gasoline was to incorporate reference gasoline at various stages of weathering to the ignitable liquids library.

Residues extracted from fire debris may have contributions from ignitable liquids and decomposition products from the heating of the materials at the fire scene.

Table 6: Key for identification of class and percent weathering of reference samples.

Symbol	Preceding number	Symbol	Following number
A	Aromatic	A	0% weathered
G	Gasoline	B	25% weathered
O	Oxygenated Solvent	C	50% weathered
M	Medium Petroleum Distillate	D	75% weathered
L	Light Petroleum Distillate	E	90% weathered
H	Heavy Petroleum Distillate	F	99% weathered
N	Naphthenic Paraffinic		
I	Isoparaffinic		

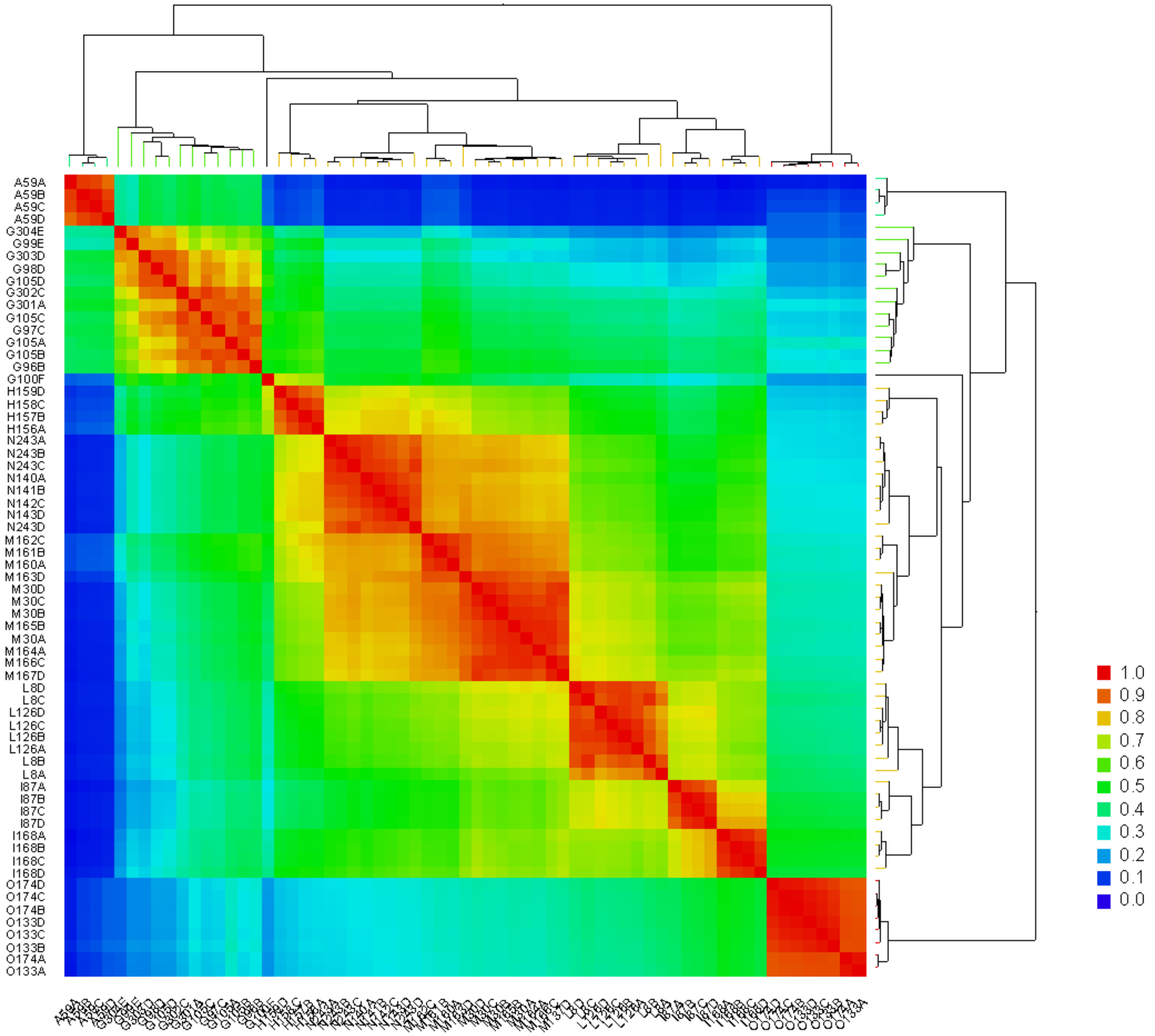


Figure 12: Graphical representation of the similarities between 65 ignitable liquids using hierarchical clustering in the study of ignitable liquid weathering.

Table 7: Similarities between reference gasoline samples at various stages of weathering

	G100F	G105A	G105B	G105C	G105D	G301A	G302C	G303D	G304E	G96B	G97C	G98D	G99E
G100F	1.000	0.502	0.544	0.558	0.562	0.484	0.526	0.492	0.476	0.504	0.528	0.527	0.561
G105A	0.502	1.000	0.931	0.893	0.783	0.909	0.869	0.761	0.624	0.888	0.892	0.776	0.663
G105B	0.544	0.931	1.000	0.940	0.824	0.895	0.897	0.783	0.651	0.903	0.921	0.813	0.701
G105C	0.558	0.893	0.940	1.000	0.878	0.897	0.941	0.837	0.698	0.866	0.919	0.858	0.750
G105D	0.562	0.783	0.824	0.878	1.000	0.800	0.891	0.919	0.812	0.767	0.828	0.919	0.843
G301A	0.484	0.909	0.895	0.897	0.800	1.000	0.903	0.790	0.644	0.877	0.904	0.804	0.680
G302C	0.526	0.869	0.897	0.941	0.891	0.903	1.000	0.875	0.725	0.849	0.910	0.888	0.764
G303D	0.492	0.761	0.783	0.837	0.919	0.790	0.875	1.000	0.843	0.735	0.798	0.920	0.828
G304E	0.476	0.624	0.651	0.698	0.812	0.644	0.725	0.843	1.000	0.608	0.658	0.797	0.882
G96B	0.504	0.888	0.903	0.866	0.767	0.877	0.849	0.735	0.608	1.000	0.927	0.780	0.658
G97C	0.528	0.892	0.921	0.919	0.828	0.904	0.910	0.798	0.658	0.927	1.000	0.844	0.714
G98D	0.527	0.776	0.813	0.858	0.919	0.804	0.888	0.920	0.797	0.780	0.844	1.000	0.830
G99E	0.561	0.663	0.701	0.750	0.843	0.680	0.764	0.828	0.882	0.658	0.714	0.830	1.000

Table 8: Similarities between reference samples classified as aromatic products at various stages of weathering.

	A59A	A59B	A59C	A59D
A59A	1.000	0.940	0.931	0.887
A59B	0.940	1.000	0.981	0.932
A59C	0.931	0.981	1.000	0.951
A59D	0.887	0.932	0.951	1.000

Table 9: Similarities between reference samples classified as heavy petroleum distillates at various stages of weathering.

	H156A	H157B	H158C	H159D
H156A	1.000	0.965	0.925	0.876
H157B	0.965	1.000	0.953	0.900
H158C	0.925	0.953	1.000	0.944
H159D	0.876	0.900	0.944	1.000

Table 10: Similarities between reference samples classified as isoparaffinic products at various stages of weathering.

	I168A	I168B	I168C	I168D	I87A	I87B	I87C	I87D
I168A	1.000	0.966	0.956	0.922	0.740	0.786	0.799	0.827
I168B	0.966	1.000	0.988	0.950	0.730	0.781	0.794	0.823
I168C	0.956	0.988	1.000	0.961	0.731	0.781	0.795	0.823
I168D	0.922	0.950	0.961	1.000	0.732	0.778	0.791	0.820
I87A	0.740	0.730	0.731	0.732	1.000	0.939	0.925	0.893
I87B	0.786	0.781	0.781	0.778	0.939	1.000	0.985	0.953
I87C	0.799	0.794	0.795	0.791	0.925	0.985	1.000	0.968
I87D	0.827	0.823	0.823	0.820	0.893	0.953	0.968	1.000

Table 11: Similarities between reference samples classified as light petroleum distillates at various stages of weathering.

	L126A	L126B	L126C	L126D	L8A	L8B	L8C	L8D
L126A	1.000	0.965	0.956	0.928	0.860	0.912	0.926	0.918
L126B	0.965	1.000	0.979	0.949	0.855	0.920	0.944	0.940
L126C	0.956	0.979	1.000	0.967	0.841	0.903	0.934	0.940
L126D	0.928	0.949	0.967	1.000	0.821	0.880	0.912	0.930
L8A	0.860	0.855	0.841	0.821	1.000	0.919	0.878	0.843
L8B	0.912	0.920	0.903	0.880	0.919	1.000	0.957	0.920
L8C	0.926	0.944	0.934	0.912	0.878	0.957	1.000	0.963
L8D	0.918	0.940	0.940	0.930	0.843	0.920	0.963	1.000

Table 12: Similarities between reference samples classified as medium petroleum distillates at various stages of weathering.

	M160A	M161B	M162C	M163D	M164A	M165B	M166C	M167D	M30A	M30B	M30C	M30D
M160A	1.000	0.961	0.942	0.913	0.865	0.886	0.857	0.837	0.872	0.887	0.882	0.867
M161B	0.961	1.000	0.966	0.917	0.847	0.869	0.846	0.833	0.861	0.879	0.881	0.872
M162C	0.942	0.966	1.000	0.918	0.839	0.861	0.840	0.831	0.854	0.873	0.876	0.873
M163D	0.913	0.917	0.918	1.000	0.885	0.907	0.900	0.895	0.898	0.921	0.931	0.929
M164A	0.865	0.847	0.839	0.885	1.000	0.962	0.947	0.918	0.958	0.952	0.932	0.910
M165B	0.886	0.869	0.861	0.907	0.962	1.000	0.959	0.931	0.965	0.961	0.948	0.924
M166C	0.857	0.846	0.840	0.900	0.947	0.959	1.000	0.968	0.961	0.947	0.952	0.943
M167D	0.837	0.833	0.831	0.895	0.918	0.931	0.968	1.000	0.934	0.922	0.935	0.949
M30A	0.872	0.861	0.854	0.898	0.958	0.965	0.961	0.934	1.000	0.960	0.944	0.925
M30B	0.887	0.879	0.873	0.921	0.952	0.961	0.947	0.922	0.960	1.000	0.977	0.949
M30C	0.882	0.881	0.876	0.931	0.932	0.948	0.952	0.935	0.944	0.977	1.000	0.971
M30D	0.867	0.872	0.873	0.929	0.910	0.924	0.943	0.949	0.925	0.949	0.971	1.000

Table 13: Similarities between reference samples classified as naphthenic paraffinic products at various stages of weathering.

	N140A	N141B	N142C	N143D	N243A	N243B	N243C	N243D
N140A	1.000	0.974	0.955	0.932	0.942	0.946	0.948	0.916
N141B	0.974	1.000	0.977	0.953	0.921	0.926	0.942	0.929
N142C	0.955	0.977	1.000	0.971	0.906	0.911	0.932	0.935
N143D	0.932	0.953	0.971	1.000	0.882	0.888	0.907	0.929
N243A	0.942	0.921	0.906	0.882	1.000	0.978	0.952	0.903
N243B	0.946	0.926	0.911	0.888	0.978	1.000	0.971	0.920
N243C	0.948	0.942	0.932	0.907	0.952	0.971	1.000	0.949
N243D	0.916	0.929	0.935	0.929	0.903	0.920	0.949	1.000

Decomposition products from substrate materials interfere in the data interpretation for classifying the ASTM E1618 class of any present ignitable liquid. A library of neat ignitable liquids may not be truly being representative of fire debris samples containing both an ignitable liquid and decomposition products from substrate materials. Investigation of methods to correct for interfering products in fire debris samples due to decomposition of substrate materials began with comparing interfering products from the substrate materials to ignitable liquids. Total ion spectra from an ignitable liquids library and a substrate library were compared to one another by calculating a similarity based on the NCFS distance equation. Hierarchical clustering using single linkage with a Euclidean distance clustered together those substrates with a high similarity to an ignitable liquid. Figure 13 is a graphical representation of the similarities between 50 substrates and 62 liquids clustering the most similar together. The highest similarities between substrate and liquid are shown in red specifying a similarity around 0.800. Substrates such as olefin carpets, foil insulation (polyethylene), shoes, Trex (composite wood), and asphalt roofing shingles are most similar to distillate (LPD, MPD, and HPD) and naphthenic paraffinic classes of ignitable liquids. These substrates are also most similar to one another compared to the other substrates as shown in figure 14. The largest cluster of similar substrates belongs to paper products, various species of wood and wood laminate flooring. A method was developed not to correct for interfering products from substrate materials, but to combine an ignitable liquid and a substrate from each of the respective libraries into reference spectrum.

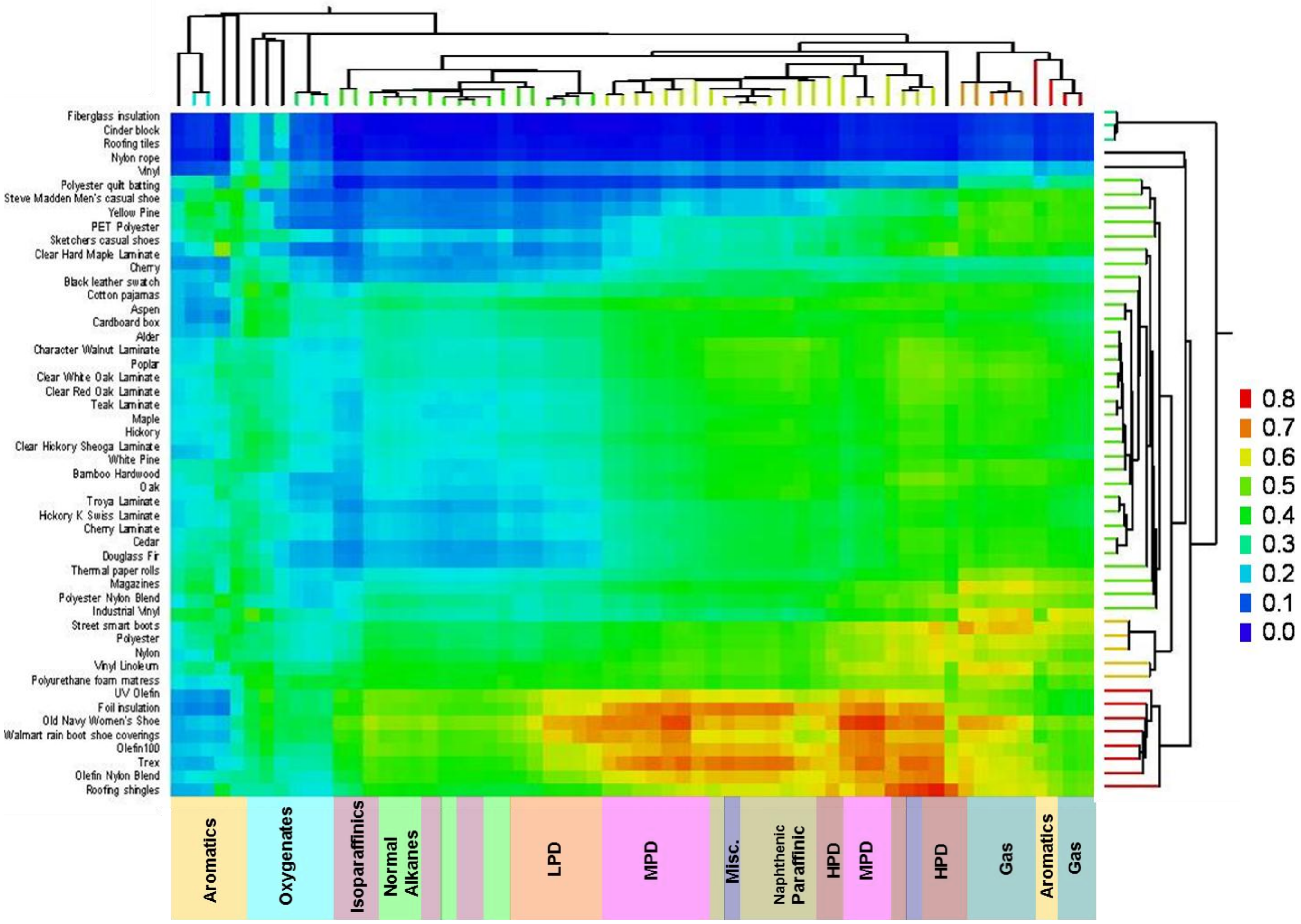


Figure 13: Graphical representation of the similarities between 50 substrate materials and 62 ignitable liquids using hierarchical clustering in the investigation to correct for decomposition products interfering in data analysis of fire debris.

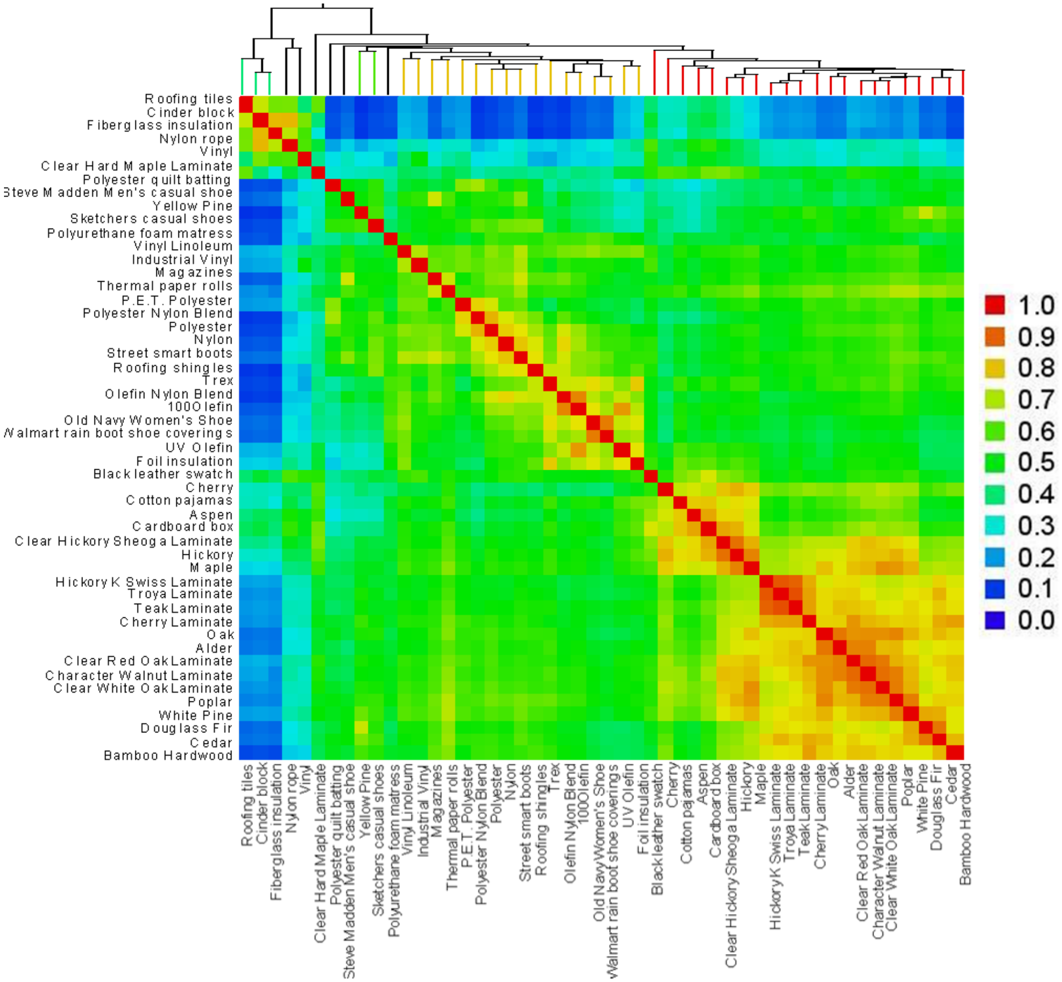


Figure 14: Graphical representation of the similarities between 50 substrate materials using hierarchical clustering in the investigation to correct for decomposition products interfering in data analysis of fire debris.

Similarities are calculated between reference spectra of each ignitable liquid plus a substrate of a certain (ignitable liquid to substrate) ratio that represents the most similar combination to the fire debris sample.

Software was provided to three crime laboratories for evaluation. Feedback from the users of the software consisted of an easier process for uploading the fire debris sample GC-MS data into the software and more information about the ignitable liquids in the search results other than a sample reference number. Initially, the GC-MS data files were exported to a comma separated data file format where the user had to choose a mass scan range and a mass/charge range. Currently, exportation of the GC-MS data is performed using an AIA or CDF file format that does require the user to perform multiple steps in the exportation process. The newer version of the software provides product name and classification of the ignitable liquid and connects the user to that particular ignitable liquid in the Ignitable Liquids Reference Collection Database for further information. When searching the substrate materials library, the substrate product description is provided and connects the user to that particular substrate material in the Substrate Database for further information. If the user selects a reference spectrum from the combination of the ignitable liquids and substrate libraries, the software will provide the product name and classification of the ignitable liquid, the product description of the substrate, and the ratio of the two. The newest version of the software now allows the reference spectrum TIC to be displayed. General user feedback on the software search results were that the classes of ignitable liquids returned by the software search as being most similar to fire debris samples were the same classes as those determined by the analyst using current procedures; however, the software did not perform as well with casework samples containing large contributions of substrate interfering components.

The software provides the fire debris analyst the ability to search multiple source libraries containing hundreds of ignitable liquid total ion spectra rapidly. Improvements to the software comprise of searching multiple libraries and combinations of total ion spectra within the libraries. Other tools included in the software were added based on current data analysis methods described by ASTM E1618. The chart in figure 15 illustrates how the software operates. The user exports the GC-MS data of a fire debris sample to an AIA (CDF) file format from the instrument computer, opens the preferred library or libraries in the software, and then opens the data file into the software, see figure 16. An equation from “parameters” that calculates the similarities between the fire debris sample and the library reference samples is selected, see figure 17. Comparison of the fire debris sample with the reference samples occurs once the user selects the library or combined libraries to search. A list of results from the search is ordered from the highest similarity to the lowest similarity with the associated sample reference number as shown in figure 18. The product name and classification of a selected ignitable liquid from the search results is displayed with a button that connects the software to the ILRC database, see figure 19. The product description of a selected substrate from the search results is displayed with a button that connects the software to the Substrate database, see figure 20. The display/sample library box allows the user to select the type of data to display as shown in figure 21. The types of display are total ion chromatograms, total ion spectra, extracted ion chromatograms of various functional groups, and a composite of the extracted ion intensities. The fire debris sample is displayed in the upper window and the chosen library reference sample is displayed in the lower window. The data displayed in the windows can be rescaled to align the fire debris sample with the library reference sample and to visualize any features of interest, see figure 22. The software is designed to provide the fire debris analyst tools to assist them in current data analysis methods and a process to search sizable databases for comparable reference liquids to classify an ignitable liquid residue.

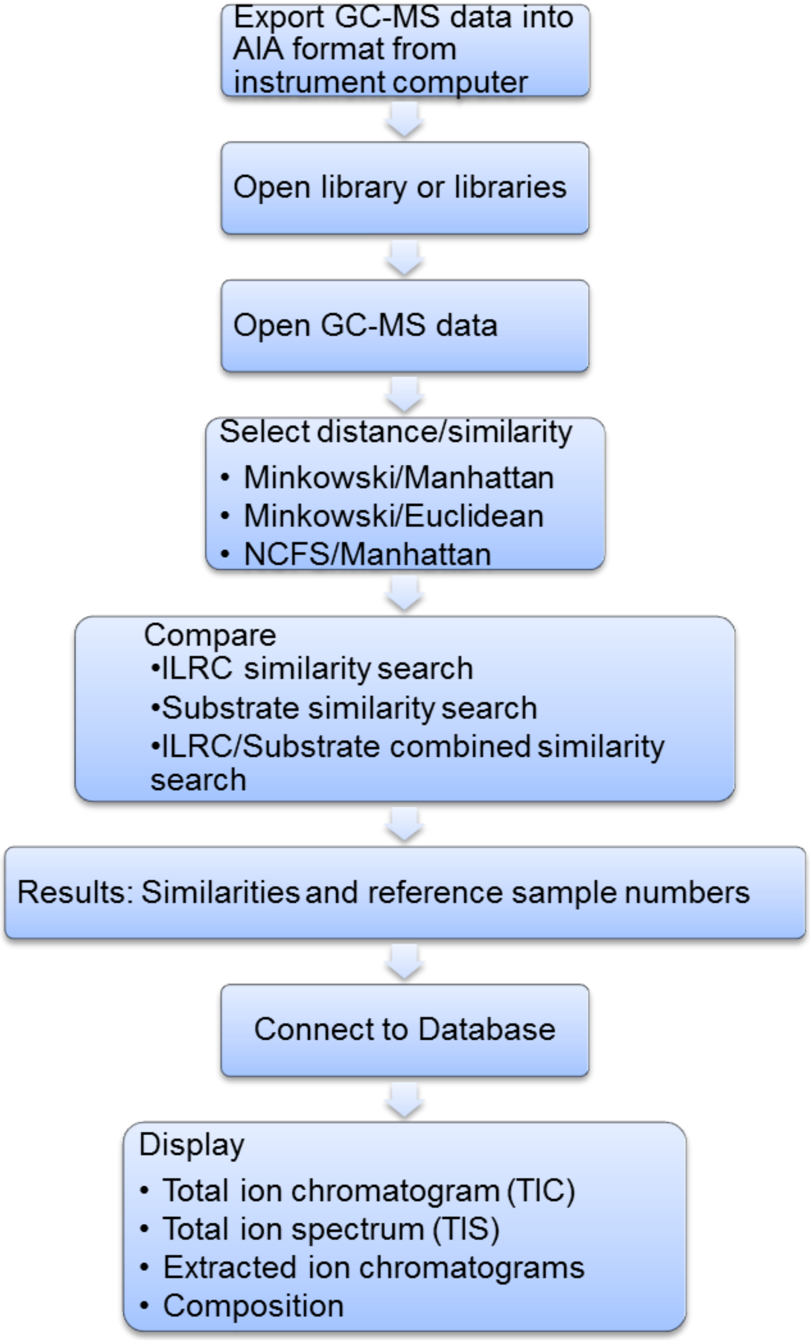
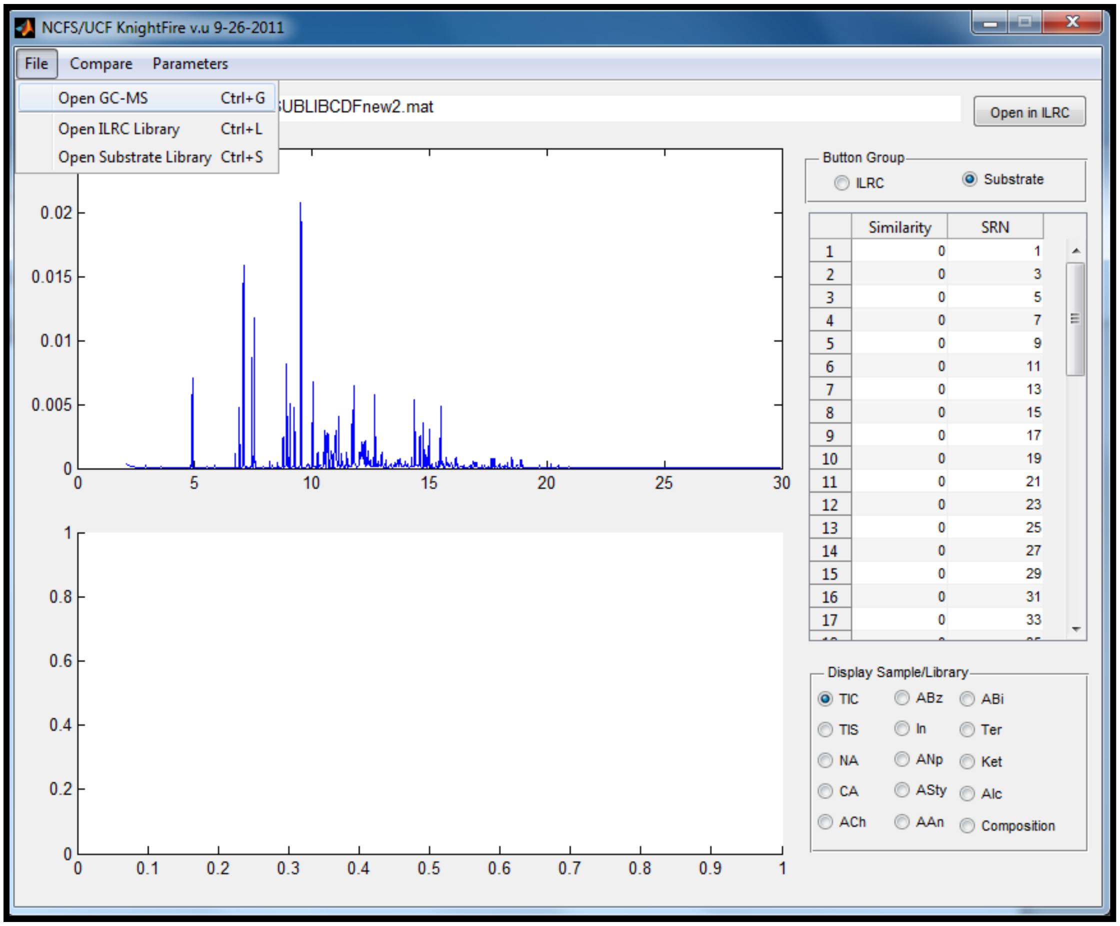


Figure 15: Diagram illustrating functions of the software



UBLIBCDFnew2.mat

Button Group

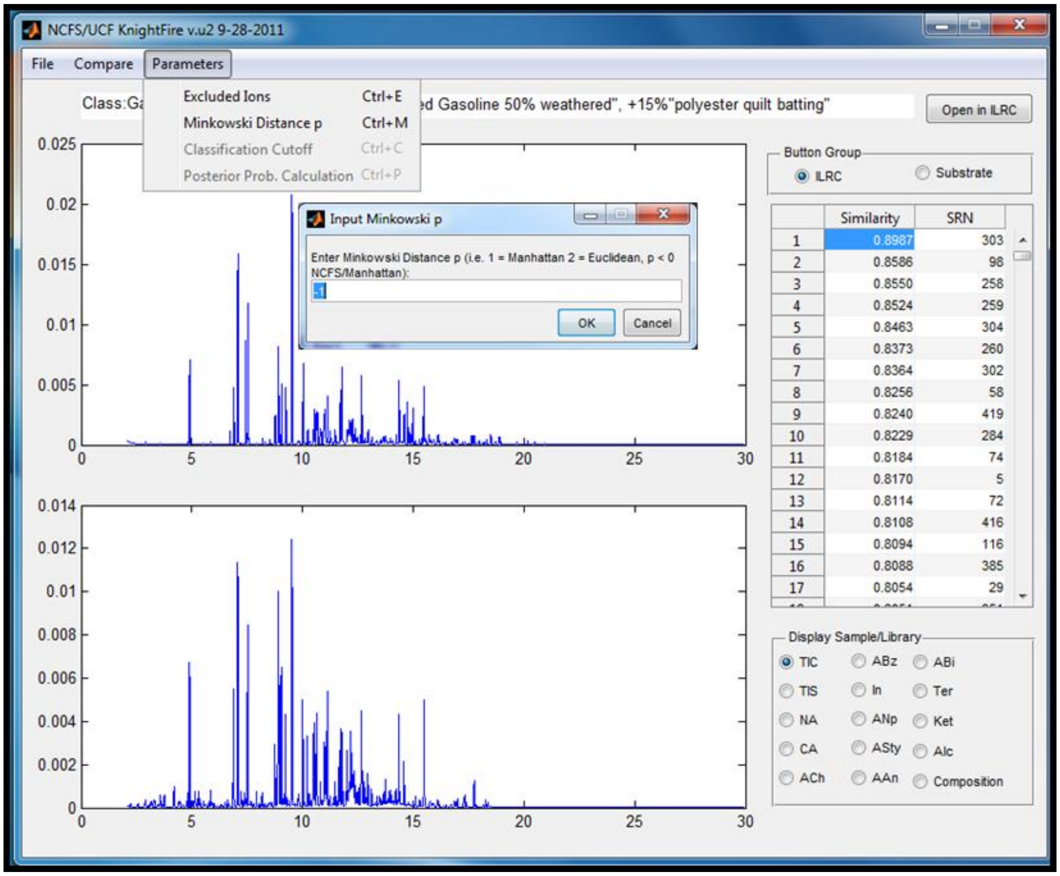
○ ILRC ● Substrate

	Similarity	SRN
1	0	1
2	0	3
3	0	5
4	0	7
5	0	9
6	0	11
7	0	13
8	0	15
9	0	17
10	0	19
11	0	21
12	0	23
13	0	25
14	0	27
15	0	29
16	0	31
17	0	33

Display Sample/Library

● TIC ○ ABz ○ ABi
○ TIS ○ In ○ Ter
○ NA ○ ANp ○ Ket
○ CA ○ ASty ○ Alc
○ ACh ○ AAn ○ Composition

Figure 16: Depicts opening of libraries and GC-MS data of fire debris sample.



Class:Ga… ed Gasoline 50% weathered", +15%"polyester quilt batting"

Input Minkowski p

Enter Minkowski Distance p (i.e. 1 = Manhattan 2 = Euclidean, p < 0 NCFS/Manhattan):

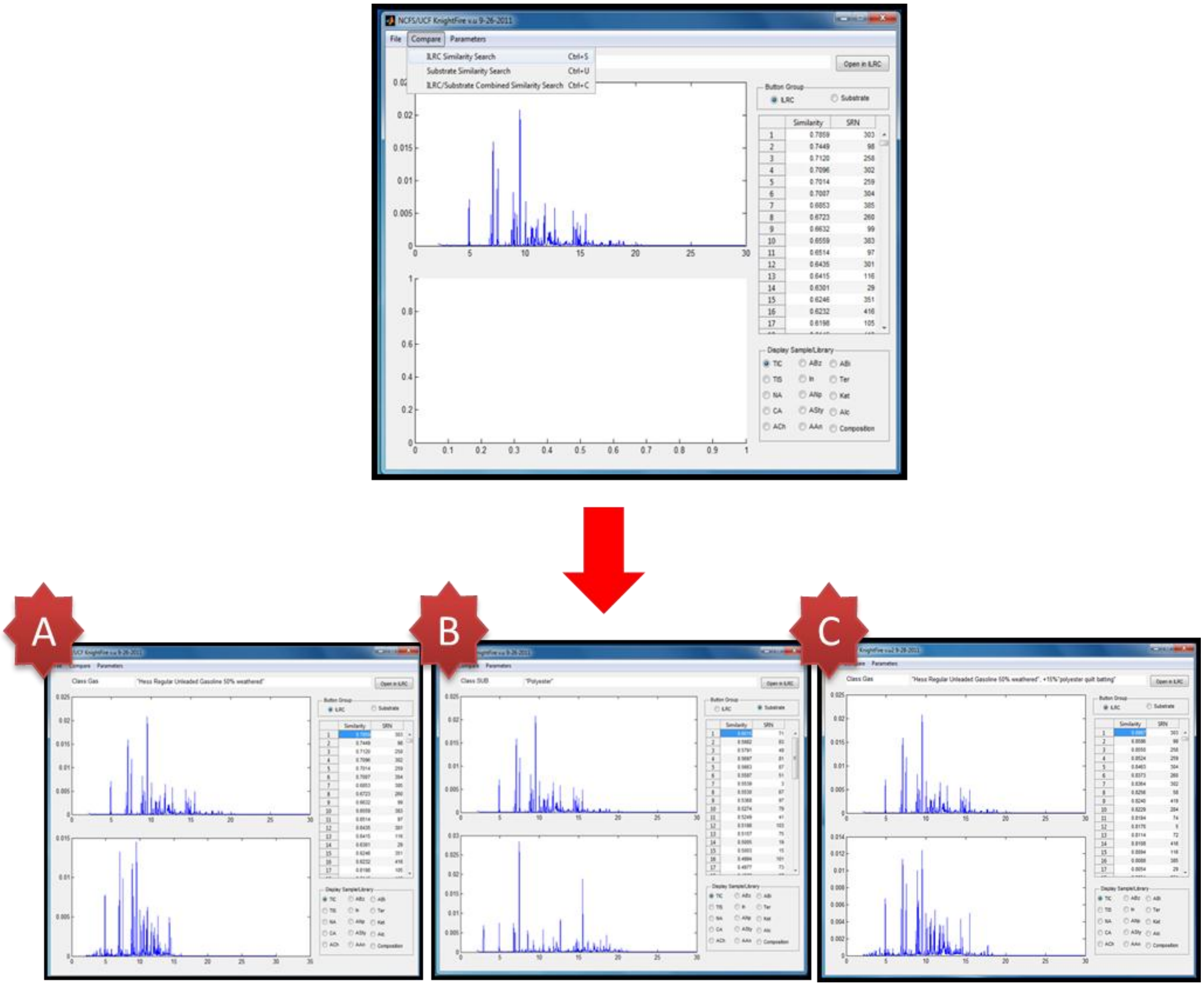
-1

Button Group: ● ILRC ○ Substrate

	Similarity	SRN
1	0.8987	303
2	0.8586	98
3	0.8550	258
4	0.8524	259
5	0.8463	304
6	0.8373	260
7	0.8364	302
8	0.8256	58
9	0.8240	419
10	0.8229	284
11	0.8184	74
12	0.8170	5
13	0.8114	72
14	0.8108	416
15	0.8094	116
16	0.8088	385
17	0.8054	29

Display Sample/Library: ● TIC

Figure 17: Depicts selection of distance/similarity equation.



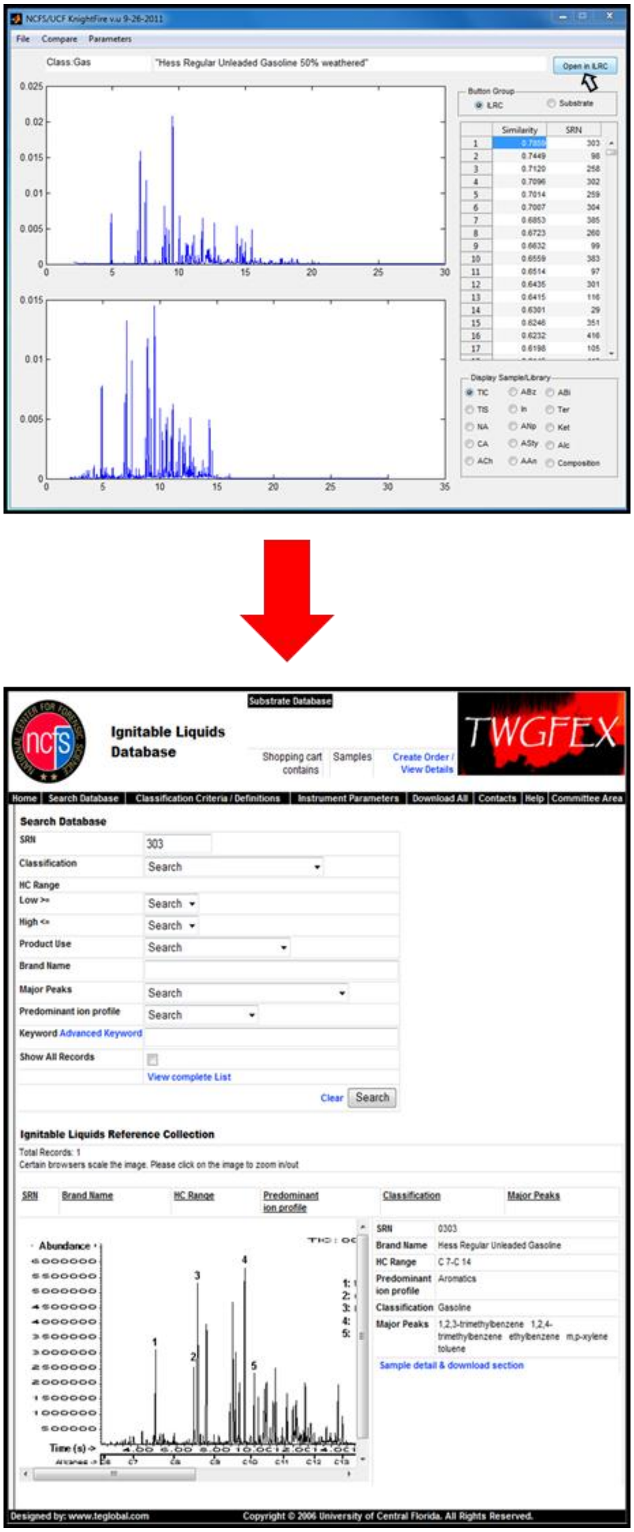
	Similarity	SRN
1	0.7859	303
2	0.7449	98
3	0.7120	258
4	0.7096	302
5	0.7014	259
6	0.7007	304
7	0.6853	385
8	0.6723	260
9	0.6632	99
10	0.6559	383
11	0.6514	97
12	0.6435	301
13	0.6415	116
14	0.6301	29
15	0.6246	351
16	0.6232	416
17	0.6198	105

Class Gas "Hess Regular Unleaded Gasoline 50% weathered"

Class SUB "Polyester"

Class Gas "Hess Regular Unleaded Gasoline 50% weathered" +15%"polyester quilt batting"

Figure 18: Depicts comparing library reference spectra to a fire debris sample and results from A) ignitable liquids library, B) substrate library, and C) reference spectra that are a combination from both the ignitable liquid and substrate libraries.



Class:Gas "Hess Regular Unleaded Gasoline 50% weathered"

	Similarity	SRN
1	0.7850	303
2	0.7449	98
3	0.7120	258
4	0.7096	302
5	0.7014	259
6	0.7007	304
7	0.6853	385
8	0.6723	260
9	0.6632	99
10	0.6559	383
11	0.6514	97
12	0.6435	301
13	0.6415	116
14	0.6301	29
15	0.6246	351
16	0.6232	416
17	0.6198	105

Search Database

SRN	303
Classification	Search
HC Range	
Low >=	Search
High <=	Search
Product Use	Search
Brand Name	
Major Peaks	Search
Predominant ion profile	Search
Keyword Advanced Keyword	
Show All Records	☐

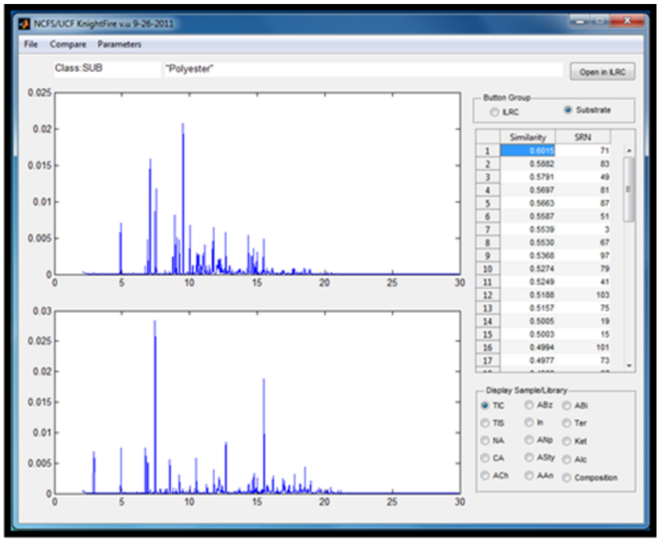
Ignitable Liquids Reference Collection

Total Records: 1
Certain browsers scale the image. Please click on the image to zoom in/out

SRN	Brand Name	HC Range	Predominant ion profile	Classification	Major Peaks

SRN	0303
Brand Name	Hess Regular Unleaded Gasoline
HC Range	C 7-C 14
Predominant ion profile	Aromatics
Classification	Gasoline
Major Peaks	1,2,3-trimethylbenzene 1,2,4-trimethylbenzene ethylbenzene m,p-xylene toluene

Figure 19: Shows product name and classification of selected ignitable liquid reference and connecting to the Ignitable Liquids Reference Collection Database.

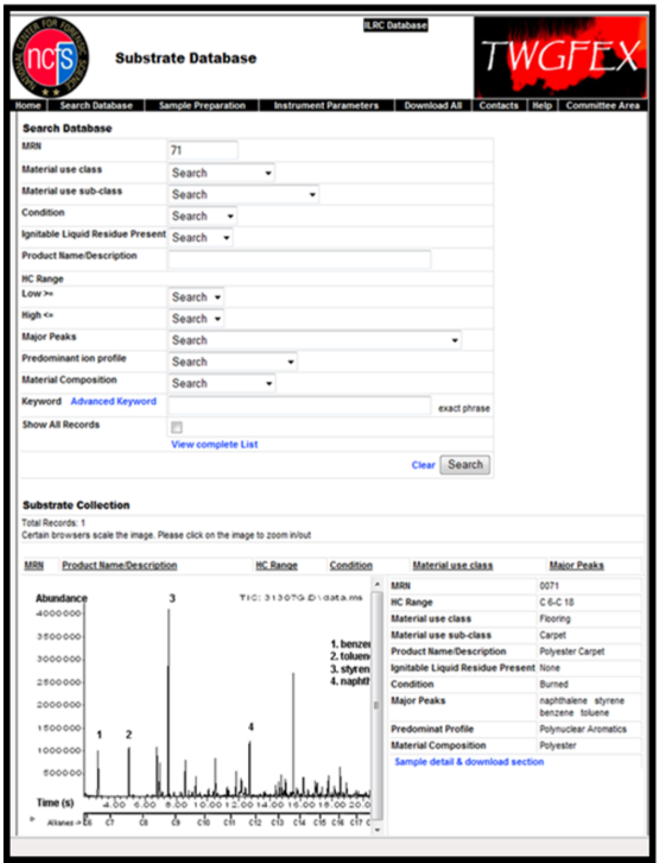


Class:SUB "Polyester"

○ ILRC ● Substrate

	Similarity	SRN
1	0.6015	71
2	0.5882	83
3	0.5791	49
4	0.5697	81
5	0.5663	87
6	0.5587	51
7	0.5539	3
8	0.5530	67
9	0.5368	97
10	0.5274	79
11	0.5249	41
12	0.5188	103
13	0.5157	75
14	0.5005	19
15	0.5003	15
16	0.4994	101
17	0.4977	73

● TIC ○ ABz ○ ABi
○ TIS ○ In ○ Ter
○ NA ○ ANp ○ Ket
○ CA ○ ASty ○ Alc
○ ACh ○ AAn ○ Composition



Substrate Database

Search Database

MRN	71
Material use class	Search
Material use sub-class	Search
Condition	Search
Ignitable Liquid Residue Present	Search
Product Name/Description	
HC Range	
Low >=	Search
High <=	Search
Major Peaks	Search
Predominant ion profile	Search
Material Composition	Search
Keyword	
Show All Records	☐

Substrate Collection

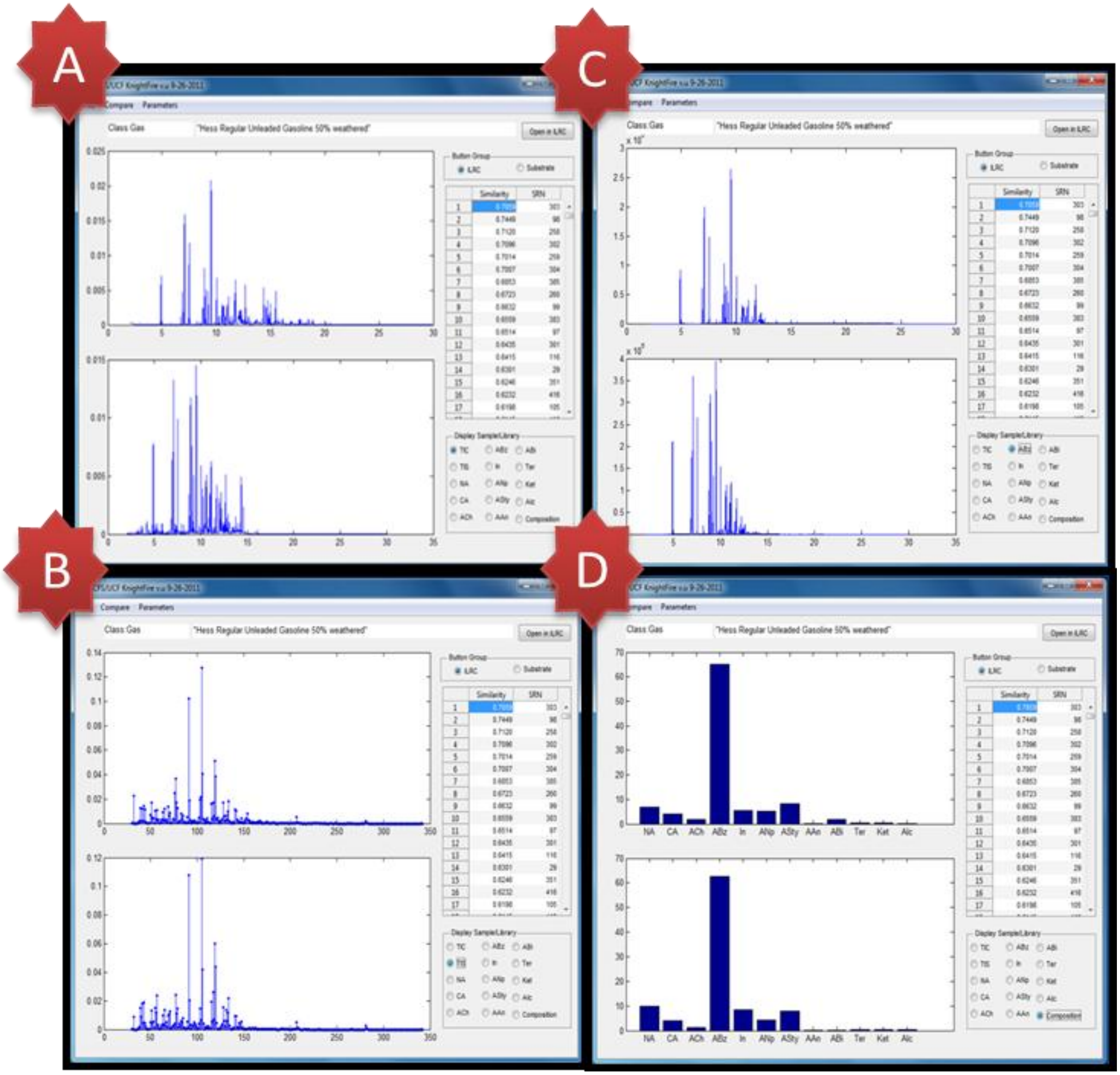
Total Records: 1

Certain browsers scale the image. Please click on the image to zoom in/out

MRN | Product Name/Description | HC Range | Condition | Material use class | Major Peaks

MRN	0071
HC Range	C 6-C 18
Material use class	Flooring
Material use sub-class	Carpet
Product Name/Description	Polyester Carpet
Ignitable Liquid Residue Present	None
Condition	Burned
Major Peaks	naphthalene styrene benzene toluene
Predominat Profile	Polynuclear Aromatics
Material Composition	Polyester

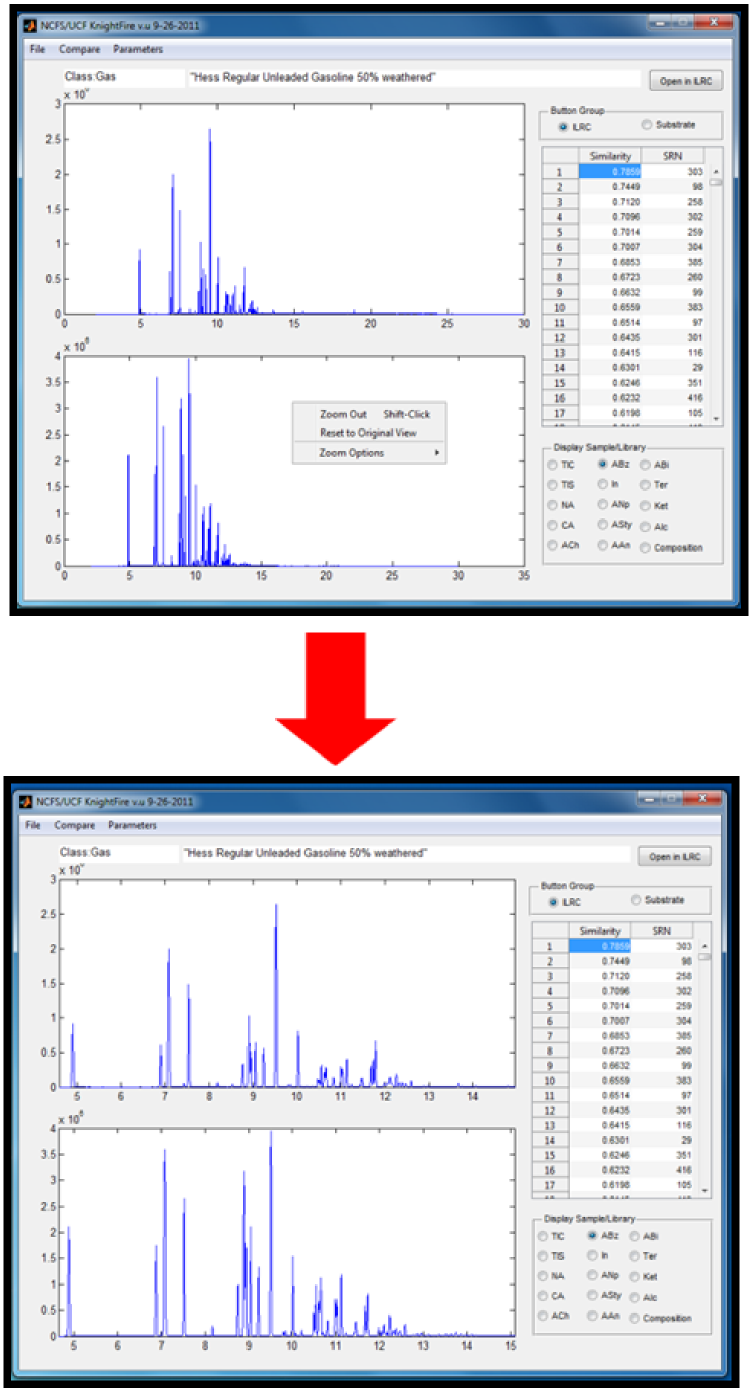
Figure 20: Shows product description of selected substrate material reference and connecting to the Substrate Database.



Class Gas "Hess Regular Unleaded Gasoline 50% weathered"

	Similarity	SRN
1	[illegible]	303
2	0.7449	98
3	0.7120	258
4	0.7096	302
5	0.7014	259
6	0.7007	304
7	0.6853	385
8	0.6723	260
9	0.6632	99
10	0.6559	383
11	0.6514	97
12	0.6435	301
13	0.6415	116
14	0.6301	29
15	0.6246	351
16	0.6232	416
17	0.6198	105

Figure 21: Depicts the various window displays of the fire debris sample and reference sample; A) TIC, B) TIS, C) one of the EIC, D) composition of extracted ion intensities.



Class:Gas

"Hess Regular Unleaded Gasoline 50% weathered"

	Similarity	SRN
1	0.7859	303
2	0.7449	98
3	0.7120	258
4	0.7096	302
5	0.7014	259
6	0.7007	304
7	0.6853	385
8	0.6723	260
9	0.6632	99
10	0.6559	383
11	0.6514	97
12	0.6435	301
13	0.6415	116
14	0.6301	29
15	0.6246	351
16	0.6232	416
17	0.6198	105

Figure 22: Depicts the ability of the software to adjust the displayed data in the windows.

Searchable Ignitable Liquids Database Tool Conclusions

A searchable ignitable liquids database tool has been developed based on the use of the total ion spectrum (TIS) for finding the best match between an ignitable liquid residue from fire debris and ignitable liquids of known classes in the Ignitable Liquids Reference Collection (ILRC). The match criterion is based on the use of several distance and similarity metrics. A search of the entire ILRC is completed in a fraction of a second. Similarly, a sample can be searched against the Substrates Library. A sample may also be compared to the best-fit combination of an ILRC liquid and a single substrate. These searches can be completed in a fraction of a second on standard laboratory computers.

The searchable ignitable liquids database tool was developed in collaboration with several forensic laboratories. The software developed under this project has been tested in three forensic laboratories (South Carolina Department of Law Enforcement, Columbia SC; Tucson Police Department, Tucson AZ; Dept of Public Saftey,Burlington, VT) during the development phase. The original version of the software was personally delivered to the laboratories by NCFS staff, installed on forensic laboratory computers and training was provided to laboratory personnel. Subsequent versions of the software were delivered over the internet and installed by laboratory personnel. Internet delivery of the software will be used for future delivery to laboratories wishing to use the software.

The searchable ignitable liquids database tool was originally developed as a compiled program to run under Microsoft Windows. The software was upgraded by reprogramming in MatLab, to run under the MatLab runtime compiler, which is distributed free of charge with the software. The user does not need to purchase MatLab to use the software developed under this program.

The accuracy of the algorithms has been tested on laboratory burn and large – scale burn samples obtained in collaboration with the Florida Fire College and leveraging other funding from the National Institute of Justice (Application of Chemometrics for the Identification of Ignitable Liquids in Fire Debris Samples, Grant Number 2008-DN-BX-K069). One US patent ("Systems and Methods for Identifying Substances Contained in a Material", application 12/244,281) covering the search method has been approved and the patent should issue in 2011.

Current fire debris analysis practice may benefit significantly from the use of the software provided under this project. The software will be further introduced to the fire debris analysis community through T/SWGFEX and future AAFS conferences. In addition to the immediate use of the software, we will also use the software as a platform for introducing research results into practicing fire debris analysis laboratories following their publication under peer – review.

Searchable Ignitable Liquids Database Tool References

[1] M. E. Sigman,* M. Williams "Covariance Mapping in the Analysis of Ignitable Liquids by Gas Chromatography/Mass Spectrometry," Anal. Chem., 2006, 78, 1713 - 1718.

[2] M. E. Sigman,* M. R.Williams, and R. G. Ivy "Comparative Analysis of Gasolines by Covariance Mapping and Gas Chromatography – Mass Spectrometry," Anal. Chem. 2007, 79(9), 3462-3468.

[3] M. E. Sigman*, M. R. Williams, J. A. Castelbuono., J. G. Colca, C. Douglas Clark "Ignitable Liquid Classification and Identification Using the Summed-Ion Mass Spectrum" Instrumentation Sci. Technol. 2008, 36, 375 - 393.

[4] Lennard CJ, Rochaix VT, Margot P, Huber K, Science & Justice 1995,35(1), 19.

[5] Bertsch, Wolfgang; Holzer, Gunther. Analysis of accelerants in fire debris by gas chromatography/mass spectrometry. Forensic Appl. Mass Spectrom. (1995), 129-69.

[6] Bertsch W. ; Sellers C. S. ; Babin K. ; Holzer G. "Automation in the chemical analysis of suspect arson samples by GC/MS. A systematic approach" Journal of High Resolution Chromatography & Chromatography Communications (1988) 11, 815.

Dissemination of Searchable Ignitable Liquids Database Tool Research Findings

US patent "Systems and Methods for Identifying Substances Contained in a Material", application 12/244,281, has been approved and will issue in 2011.

A presentation, "Ignitable Liquids Reference Collection Panel: Present Research" was made at the 2011 National Institute of Justice Conference, Washington D.C., June 2011.

The software developed under this project has been tested in three forensic laboratories; (1) South Carolina Department of Law Enforcement, Columbia SC; (2) Tucson Police Department, Tucson AZ; (3) Dept of Public Saftey,Burlington, VT.

Electronic software dissemination is available through the National Center for Forensic Sciences website. Information on the software is currently available on the NCFS website and new site dedicated to the software is under development. The UCF Office of Technology Transfer is developing the user agreement forms that must be signed by appropriate representatives from those laboratories that wish to use the software.

Appendix 1:

Operational Guideline for the Analysis, Identification, and Classification of Smokeless Reloading Powders

April 1, 2010

Prepared by

Dana-Marie Dennis and Jessica Frisch

Scope and Application

1.1 This document describes the methods employed in the analysis, identification and classification of smokeless reloading powders.

1.2 The method is suitable for distinguishing between classes of smokeless powders, namely, single-based and double-based powders.

1.3 The method utilizes micrometry, extraction techniques, Gas Chromatography-Mass Spectrometry (GC-MS) analysis, and Attenuated Total Reflectance-Fourier Transform Infrared Spectrometry (ATR-FTIR).

2. Summary of Methods

2.1 Micrometry

2.1.1 Smokeless powder kernels are photographed in batches, and measurements of each kernel per batch are taken. The averages and standard deviations of the measurements are calculated. The maximum and minimum kernel dimensions are also noted.

2.1.2 Micrographs are taken of fifty different kernels.

2.2 Extraction

2.2.1 The kernels are cut and then weighed and placed into small vials. Methylene chloride with 10 ppm undecane is added and extraction is allowed to take place for three hours.

2.3 Gas Chromatography-Mass Spectrometry

2.3.1 The extract supernatant is removed and placed into GC vials (with vial insert) for analysis. A 1:10 dilution is also analyzed.

2.4 Attenuated Total Reflectance

2.4.1 A few kernels are dissolved in acetone, and the solution is spotted onto a microscope slide resulting in a film after the acetone evaporates. A spectrum of the film is collected using ATR-FTIR.

3. Hazards

3.1 Nitroglycerin is flammable and poisonous. Wear gloves when handling it and keep away from open flames.

3.2 Camphor is highly flammable and should be handled away from open flames.

3.3 Ethyl Centralite is toxic. Wear gloves when handling.

3.4 2,4-DNT is listed as a possible carcinogen. Wear gloves when handling.

3.5 Dioctyl phthalate is listed as a possible carcinogen. Wear gloves when handling.

3.6 Diphenylamine is highly toxic and has a target organ effect. Wear gloves when handling.

3.7 4-nitrosodiphenylamine is a possible carcinogen. Wear gloves when handling.

3.8 Smokeless powders contain organics which are hazardous to health. Wear gloves when handling.

3.9 Lab coat, insulated gloves, and proper eye and face protection should be worn when handling liquid nitrogen.

4. Receiving and Storage

4.1 Receiving

4.1.1 Each sample should be packaged in a one dram vial with screw-cap lid.

4.1.2 Vials should be labeled with smokeless powder sample reference number (Source SRN), manufacturer name, and product name.

4.1.3 Received vials should be added to the inventory list.

4.2 Storage

4.2.1 One dram vials of smokeless powders are stored in a SciMatCo nonmetallic flammables cabinet labeled with “Flammable Solid 4” hazard placard.

4.2.2 The cabinet is housed in the Hazmat building.

4.2.3 Flammable cabinet should remain locked.

5. Methods, Materials and Instrumentation

5.1 Micrometry

5.1.1 A stage micrometer is used to calibrate the Leica Stereomicroscope micron bar in order to make measurements of the smokeless powder kernels. Measurements are made in millimeters to one decimal place. Calibration of the micron bar is done when the micrograph image is at a zoom of 2:1.

5.1.2 The smokeless powder kernels are placed on a white background, and a total of fifty kernels are photographed. The micrographs are taken at x10.1 magnification.

5.1.3 Double-sided tape is used to stand kernels upright so that micrographs may be taken for measurement of thickness.

5.1.4 ImageTool software is calibrated using the micron bar as described in section **Error! Reference source not found.**

5.1.5 ImageTool software is used to make measurements of each of the kernels in the micrographs. Measurements are taken when the micrograph image is at a zoom of 1:1. (The software may be downloaded from http://ddsdx.uthscsa.edu/dig/itdesc.html.)

5.1.6 Length and thickness measurements are taken from the images of the kernels in the micrographs. Thickness measurements of short tube kernels are obtained from the micrographs taken using the double-sided tape.

5.1.7 Thickness measurements for discs and flattened ball powder kernels are not recorded.

5.1.8 The averages and standard deviations of the measurements are calculated and the maximum and minimum measurements noted for the 50 kernels measured.

5.2 Extraction

5.2.1 The smokeless powder kernels not including markers are cut, and approximately 10 mg of the cut kernels are placed into a previously weighed small vial. Research indicates that cutting the kernels is necessary for the optimal extraction of the analytes.

5.2.2 300 µL of methylene chloride are added to the contents of the tube in order to facilitate the extraction. The tube is allowed to sit for three hours.

5.2.3 The supernatant is removed and place into a GC vial for analysis.

5.3 GC-MS

5.3.1 The supernatant is injected into the GC at its extracted concentration in an attempt to detect all organics which may be present.

5.3.2 A 1:10 dilution of the original extract may also be injected to prevent overloading. This will result in very broad peaks which will lead to incorrect retention times, and incorrect peak areas for nitroglycerin or other highly concentrated analytes which may be present. As a result, the quantity of the analyte will be difficult to calculate.

5.3.3 Positive identification of an analyte is determined by a 3:1 signal-to-noise ratio, the presence of at least three major ions, and its retention time as compared to a standard.

5.4 ATR-FTIR

5.4.1 Approximately 2.5 mg of smokeless powder kernels not including markers are added to a small vial with 80 µL of acetone and allowed to sit for thirty minutes.

5.4.2 The supernatant is spotted on a microscope slide, the solvent is allowed to evaporate, and the film is analyzed using ATR-FTIR.

6. Preparation of the Standards

6.1 A smokeless powder standard stock solution containing 150 ppm each of all organics listed in Table 1 (except 4-nitrosodiphenylamine, 4-nitrodiphenylamine and nitroglycerin) is prepared in methylene chloride. Undecane functions as the internal standard. A standard stock solution is prepared which contains 180 ppm each of 4-nitrosodiphenylamine and 4-nitrodiphenylamine as well as 100 ppm undecane. The solvent is methylene chloride. Undecane functions as the internal standard.

6.1.1 From the stock solutions, a single standard is prepared each day of analysis. This is prepared by combining 200 µL of methylene chloride along with 50 µL of the nitroamines standard stock solution, 30 µL of nitroglycerin, and 20 µL of the smokeless powder standard stock solution. It should be noted who made the standard for each day.

7. Quality Control and Quality Assurance

7.1 Micrometry

7.1.1 Fifty kernels of each sample are arranged and photographed as described in **Error! Reference source not found.**.

7.1.2 To ensure consistency in the measurements, two analysts take the measurements independently, and the results are compared.

7.2 Extraction

7.2.1 Extractions are performed in duplicate for each of the smokeless powder samples.

7.2.2 The extractions are performed by two analysts (a total of two extractions per powder sample) as described in section 5.2 to ensure consistency in the method.

7.2.3 Following the extraction, a second vial for each analyst is prepared with a 1:10 dilution (in methylene chloride) of the extract.

7.3 GC-MS

7.3.1 The smokeless standards are analyzed each morning to ensure that the instrument is functioning properly.

7.3.2 Each extract supernatant (from extractions as described 5.2.3) is analyzed in duplicate to ensure that results are consistent.

7.4 ATR-FTIR

7.4.1 Liquid nitrogen is added to the Dewar of the instrument to allow cooling.

7.4.2 A polystyrene standard is analyzed prior to analysis of the smokeless powder film.

7.4.3 Each film (as described in 2.4.1) is placed on a clean microscope slide, and four spectra are collected from four different places of the film for each slide. The spectrum with the flattest baseline and best resolution is chosen for incorporation into the database.

8. Instrument Parameters

8.1 GC-MS

8.2 Method name: SPDB1.M

8.2.1 Analyses of the samples are conducted using a Hewlett-Packard 6890 gas chromatograph interfaced to a 5973 mass spectrometer.

8.2.2 The 30m capillary column is an HP-5MS 5% phenyl methyl siloxane with a nominal diameter of 250.00 μm and a film thickness of 0.25 μm.

8.2.3 Helium carrier gas is maintained at a flow rate of 1.2 mL/min on the column with an average velocity of 40 cm/sec.

8.2.4 One micro liter injections of the sample are introduced using an Agilent ALS autosampler G2614A splitless injector.

8.2.5 The mass spectrometer transfer line is maintained at 250°C, with a source temperature of 230°C and a quadrupole temperature of 150°C.

8.2.6 Mass spectra are scanned between 43 and 400 m/z. The solvent delay is 4.00 minutes.

8.2.7 The initial oven temperature of 40°C is held for 1 minute, followed by a temperature ramp of 25°C/min to a final temperature of 280°C, which is held for three minutes.

8.3 ATR-FTIR

8.3.1 Method name: Smokeless Powders.EXP

8.3.2 Analyses of the samples are conducted on an ATI Mattson Infinity Series FTIR and Spectra Tech IR Plan Advantage IR microscope.

8.3.3 The internal reflectance element used to analyze the sample is silicon.

8.3.4 The spectrum is collected in absorbance mode using OMNIC software with the number of scans totaling 32 and a resolution of 4. An ATR correction was applied using the OMNIC software which corrects for the variance in the penetration depth. The data image is reported in percent transmittance.

9. Quantification of The Target Analyte – future

9.1 Determination of the response factor

9.1.1 The standards containing all analytes of known concentration are analyzed using the GC-MS as described in section 8.1.

9.1.2 The response factor (RF_A) for each analyte is determined as follows:

$RF_A = [(Area_{IS})/(Mass_{IS})]/[(Area_A)/(Mass_A)]$,

where “IS” refers to the internal standard, and “A” refers to the analyte.

9.1.3 The areas under the peaks corresponding to the internal standard, and the target analyte, as well as the mass injected for the analysis are used to calculate the response factor for each analyte.

9.2 Determination of mass of the target analyte

9.2.1 The extract supernatants of each sample are analyzed using the GC-MS as described in section 8.1.

9.2.2 The mass of the target analyte is calculated as follows:

$Mass_A = [(RF_A)(Area_A)(Mass_{IS}]/(Area_{IS})$

9.2.3 The areas under the identified peaks (corresponding to each target analyte and the internal standard), as well as the previously calculated response factors, are used to calculate the mass of the analyte in the sample.

Table 1. List of Analytes, CAS Numbers, and Three Major Ions (m/z) with Relative Intensities of the Target Analyte

Analyte	CAS Number	Mass-to-Charge	% Abundance*
Nitroglycerin	55-63-0	46	100
		76	15
		43	13
Camphor	21368-68-3	95	100
		81	64
		108	41
Dibutyl Phthalate	84-74-2	149	100
		223	6
		205	5
Diethyl Phthalate	84-66-2	149	100
		177	26
		105	7
Dipentyl Phthalate	131-18-0	149	100
		150	9
		237	8
Diphenylamine	122-39-4	169	100
		84	10
		51	7
Dioctyl Phthalate	117-81-7	149	100
		167	35
		57	17
Ethyl Centralite	85-98-3	120	100
		148	81
		268	46
Methyl Centralite	611-92-7	134	100
		240	62
		106	45

2,4-Dinitrotoluene	121-14-2	165	100
		89	50
		63	27
4-nitrodiphenylamine	836-30-6	214	100
		167	83
		184	26
2-nitrodiphenylamine	119-75-5	214	100
		167	82
		180	37
4-nitrosodiphenylamine	156-10-5	167	100
		198	72
		184	12
Undecane **	1120-21-4	57	100
		43	72
		71	55

* % Abundance determined using standards and in-house GC-MS method

**denotes internal standard