

# Best Practices for the Interpretation and Use of the Sample Data in the Substrate Database

*CAUTION: Care must be exercised in relating the contents of this database to extractions from debris collected in fire scenes. The samples in this database were prepared as single substrates under controlled conditions.*

## Scope

The substrate database is a collection of chromatographic mass spectrometric data for materials that could be found at fire scenes. For each unspiked material, unburned and burned data generated under various conditions are included. The purpose of the database is to increase the understanding of fire debris analysts as to the types of compounds generated from various materials burned/pyrolyzed materials.

## Cautions/considerations

The Substrate Database is a tool designed for **informational purposes only**. It does not replace the potential need for obtaining contemporaneous relevant comparison samples (as defined by ASTM E1732) to evaluate the matrix within questioned samples. As instrumental conditions and sensitivities may vary, comparison samples should be analyzed on the same instrument utilizing the same instrumental conditions as casework. Classification of any ignitable liquid residues identified within the substrate database parallels ASTM E1618-14 and is a consensus from a committee of practicing fire debris analysts.

Each substrate was burned individually under controlled conditions, which may not be indicative of fire scene conditions. Furthermore, there can be extensive variation within a given substrate type (e.g. plastic cups, types of plywood) based on manufacturer, geographic region of manufacture or intended use, production method, etc. Production methods and composition of substrates may also change over time. Real casework samples frequently contain multiple substrates exposed to a variety of conditions, and no database will be able to contain comparison samples for every conceivable condition and substrate combination.

## Methods

Substrate samples for records MRN 1 to 216 were prepared using a sample size of 6 cm<sup>2</sup>. There are two records for each substrate: an unburned sample and a burned sample. The burned sample was prepared following the modified destructive distillation method for 2 minutes (see method below).

Substrate samples for records beginning with MRN 220 are prepared using a sample size of approximately 16 cm<sup>2</sup> or 1.5 grams. Each substrate is burned by a modified destructive distillation method (MDDM), a direct method (DH), and an indirect method (IH). The substrate samples are burned at three different time intervals for each burning method. Including the unburned sample, each substrate consists of 10 samples/records.

### Modified Destructive Distillation Method



Substrates are placed into an unlined quart metal paint can with the substrate surface most commonly adjacent to the heat source facing downward. For example, a carpet sample is positioned so the tufted or woven textile side is in contact with the bottom of the can. Nine holes of approximately 1 cm in diameter are punched into the can lid and then placed on the can without sealing. Heat is applied with a propane torch so the tip of the torch is 4 cm from the bottom of the can and the flame is centered. Once smoke appears from the top of the can, the time interval begins. A sample is prepared for each of three time intervals: 1, 2, or 5 minutes. At the end of the time interval, the propane torch is removed. The punctured lid is replaced with an intact lid and the can is cooled to room temperature.

### Direct Method



Substrates are placed onto an upside down can lid with the substrate surface most commonly adjacent to the heat source facing upward. For example, a carpet sample is positioned so the tufted or woven textile side faces upward toward the propane torch. The torch flame is positioned at a 75-degree angle and 4 cm from the substrate sample. The time interval begins immediately. A sample is prepared for each of three time intervals: 1, 2, or 3 minutes. At the end of the time interval, the propane torch is removed. A can is placed over the sample, and then cooled to room temperature.

### Indirect Method



Substrates are placed onto an upside down can lid, which is positioned on a ring stand with the substrate surface most commonly adjacent to the heat source facing downward. For example, a carpet sample is positioned so the tufted or woven textile side is in contact with can lid. The torch flame is applied at a 75-degree angle underneath the lid. The time interval begins once smoke appears. At the end of the time interval, the propane torch is removed. A can is placed over the sample, and then cooled to room temperature.

Interfering products from the heated and unburned substrate samples are collected by passive headspace adsorption onto activated carbon following the ASTM E 1412 standard method. 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 66°C. Once cooled to room temperature, the activated carbon strip is removed from the can placed into a vial with 1/2 ml of carbon disulfide for analysis.

These burning conditions were kept controlled throughout each substrates' sampling. However, the burning of material using these various methods and times only demonstrates some possible variations and results of the pyrolysis of a material. The amount of time, the temperature the environment achieves, the application and distance of the heating source and oxygen present, the orientation of the substrate, and the concentration of substrate(s) present can generate different variations of compounds.

## Observations and Discussions

The compounds observed for a given substrate, and the relative amounts of these compounds, can vary depending on whether the substrate is burned, and/or the burning method that is used. A wider range of combustion byproducts and a greater instrumental response were typically observed with the MDDM burning method. Volatile hydrocarbon compounds may not be observed for some unburned substrates. The direct and indirect heat methods also did not result in observable combustion byproducts for some substrates in the database.

The substrate database does not use an internal standard, therefore substrates that do not produce combustion byproducts appear as blanks with no visible peaks.

The contribution of each extracted ion profile (EIP) is reported as a percentage of the total integrated area of the TIC. The 'predominant ion profile' is generally the EIP with the largest area percentage. The percent contribution of each EIP can be found in the 'sample detail and download' section for each substrate (e.g. M00000519.pdf). In samples where there was low abundance of peaks no predominant profile was entered.

## Laboratory Recommendations/Considerations

The presence of small quantities of some components common to a particular class of ignitable liquid product does not necessarily indicate the presence of that liquid in the debris at the time of the fire. For example, the pyrolyzates of some polymers may include toluene and C<sub>2</sub>-benzenes, but that does not indicate gasoline or a light aromatic product is present. The components need to be present in the correct ratios, and comparable to a reference standard for identification of an ignitable liquid. When single compounds are to be identified and reported in a sample, they should be significantly greater

than the response of matrix compounds present in the data. For example, ASTM E1618-14 recommends that oxygenates be at least one order of magnitude above the matrix peaks in the chromatogram before the analyst should consider the finding of an oxygenated product significant.

As a by-product of the manufacturing process, certain products have been reported to contain ignitable liquids. These have included shoes, vinyl flooring, adhesives, clothing, as well as print media. Likewise, some substrates tested were found to contain low levels of ignitable liquids. Many materials are manufactured under conditions using a wide variety of feedstocks (i.e., for various plastics used in the manufacture of shoes see <http://www.gem-chem.net/artpubsoles.html>), processes, or additives that could contain a variety of ignitable liquids. The finding of an ignitable liquid on a given substrate does not necessarily imply that an ignitable liquid was intentionally used to start/speed up a fire. In the database, one sample of each substrate was analyzed without heating/pyrolysis/thermal degradation to show whether an ignitable liquid was inherent on the substrate tested. Due to differences in the processing and equipment used by various manufacturers, analyzing a single item is not representative of all items of the same type, so the contents of the substrate database should not be considered all-encompassing for any of the types of materials tested..

When you would want comparison samples –

- Literature or your laboratory's substrate collection indicates an ignitable liquid residue may be present in the matrix.
- Data indicates matrix interference may be masking a potential ignitable liquid residue.

When you don't need comparison samples –

- No ignitable liquid was identified/indicated in the unknown.
- The response for the ignitable liquid was overwhelming, and matrix response is minute in comparison (ref: US v Aman, 748 F. Supp.2d 531 (E.D. Va., 2010)).
- Literature does not indicate the ignitable liquid's presence in the particular matrix, and no matrix comparison is available.

It is recommended that laboratories generate their own library of burned and unburned matrices obtained from their region. Recommendations on how to make your own comparison samples –

- Based upon the research to populate this substrate database, burning the matrix for 2 minutes was the most consistent burning time that produced usable pyrolysis products.
- The three burn methods generally produced data with different pyrolysis products and/or distributions for the same matrix.
- Overall, the modified destruction distillation method (MDDM) produced a greater number of pyrolysis products at a greater concentration than the other two methods. Typically, the MDDM method would contain all the products found in the other two burn methods, but not necessarily at the same relative ratios.