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TECHNICAL NOTE

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Preparation of Pyrolysis Reference Samples: Evaluation of a Standard Method Using a Tube Furnace

ABSTRACT: A new, simple method for the reproducible creation of pyrolysis products from different materials that may be found at a fire scene is described. A temperature programmable steady-state tube furnace was used to generate pyrolysis products from different substrates, including softwoods, paper, vinyl sheet flooring, and carpet. The temperature profile of the tube furnace was characterized, and the suitability of the method to reproducibly create pyrolysates similar to those found in real fire debris was assessed. The use of this method to create proficiency tests to realistically test an examiner's ability to interpret complex gas chromatograph–mass spectrometric fire debris data, and to create a library of pyrolsates generated from materials commonly found at a fire scene, is demonstrated.

KEYWORDS: forensic science, fire debris, arson, pyrolysis, proficiency test, tube furnace, GC-MS, volatile organic compound, VOC

Pyrolysis, or the thermal degradation of organic materials, has long been recognized by forensic scientists as the mechanism by which volatiles are generated from burning substrates (e.g., wood, flooring materials, and furnishings) at a fire scene. These volatiles are observed in the analysis of fire debris as a "background" profile in the resulting chromatogram and can confound the identification of an ignitable liquid that may have been intentionally added to the scene as an accelerant. However, the use of the word "pyrolysis" to describe the background products observed in fire debris, and the conditions under which it occurs, is open to debate. Strictly speaking, pyrolysis is the word used to describe the chemical degradation reaction(s) that are caused by the application of thermal energy in the absence of oxygen (1) and is usually applied to the thermal-chemical reaction of organic compounds. Occasionally, words with broader or more restrictive meanings have been used to describe this thermal degradation process. For example, it has been suggested that the word "thermolysis" could be used interchangeably with "pyrolysis" (2); however, thermolysis may be used to describe both organic reactions and inorganic dissociations (3), whereas pyrolysis is typically used to only describe organic reactions (4). Hurd has argued that "thermal decomposition" is a more restrictive term than pyrolysis because the formation of larger molecules from smaller ones can be caused by heat alone (i.e., pyrolysis), but cannot be considered a "decomposition" (4). "Oxidative pyrolysis" is defined as pyrolysis in the presence of oxygen (1). So what word or phrase should be used to describe the action of heat on substrates at a fire scene? Hurd points out that "pyrolysis" actually refers to the initial reaction of a single compound and went on to coin the term "pyrosynthesis" (4) to describe the simultaneous reaction of two or more components in a complex mixture

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(compounds either present in the original mixture or produced by subsequent thermal reaction). However, it is important to remember that Hurd was primarily interested in using pyrolysis as a pathway to synthesize new organic compounds (4). While Hurd did not specify the atmosphere under which he considered pyrolysis to occur, today the word pyrolysis usually refers to a reaction that occurs in an inert atmosphere, that is, in an atmosphere devoid of oxygen (1). Yet, in the field of renewable energy resource development, a broader definition of pyrolysis is generally accepted where pyrolysis has been defined as "[t]he thermal decomposition of materials in the absence of oxygen or when significantly less oxygen is present than required for complete combustion" (5, p. 850). In the typical structure fire, some oxygen will be present, and so for the sake of simplicity, this broader definition of pyrolysis could reasonably be applied to the field of forensic fire debris analysis as well.

Another difficulty in defining "pyrolysis" is the lack of agreement in the literature over what temperature range it occurs. Pyrolysis has been defined by Moldoveanu (6) as occurring between 300 and 350°C, while "thermal decomposition," not pyrolysis, occurs at lower temperatures (i.e., 175-250°C). In contrast, Evans and Milne (7) have studied the "pyrolysis" of woody materials at temperatures in excess of 800°C. These authors went on to state that "primary" pyrolysis products (the initial products formed by the application of heat at lower temperatures) can undergo "secondary cracking" at temperatures above 500°C, resulting in very complex mixtures (7). Guar and Reed (8) have used the terms "devolatilization" and "pyrolysis" interchangeably to describe heating substances in an inert atmosphere or under "oxygen lean" conditions in the temperature range of 300-700°C. The word "torrefaction" is used by some authors to describe the relatively low temperature (225-300°C) thermal degradation of natural materials such as wood (9,10). Given the wide range of temperatures (11) and oxygen levels that different materials in a structure fire may be exposed to, it becomes clear why forensic scientists have adopted the generic word "pyrolysis" to describe the thermal degradation of materials present at a fire scene without attempting to define the amount of oxygen that may be present or the exact temperature range over which it applies.

In the context of fire investigation, one of the first attempts to intentionally char different materials in the laboratory and analyze the resulting pyrolysis products by gas chromatography was described by Ettling and Adams (12). In this study, they burned pieces of wood, paper, or polyethylene in a muffle furnace at 600°C, controlling for the amount of material burned as well as the burn time. Howard and McKague (13) characterized the volatiles given off by a styrene/butadiene copolymer rubber by heating a sample in a test tube over an open flame and sampling the headspace. DeHaan and Bonarius placed samples of carpet and other floor coverings in a room and intentionally burned them in a simulated structure fire in order to study the volatiles evolved from the different substrates (14). Recognizing the need to create comparison samples of pyrolysis products for use in the interpretation of chromatograms from fire debris, two studies reported heating various building materials in clean, unused metal cans with an open flame (15,16). A similar method of burning a variety of substrates in a metal can to generate pyrolysis products was employed by Stauffer to elucidate the mechanisms that produce pyrolysis products (17). A major difficulty with heating substrates in a metal can with an open flame is that it is not possible to measure the exact temperature reached by the substrate, and so the process is inherently not very reproducible because an exact temperature for a specific period of time cannot be set for the experiment. Another drawback to this method is that a relatively large sample must be burned/pryolyzed. It was while collecting pyrolysis data from different substrates in our laboratory using a muffle furnace (18) that we found that accurately controlling the time and temperature for different samples could not only give reproducible results, but also that the amount and type of pyrolysis products generated varied with time and temperature. As a result of this observation, and the desire to scale-down the sample size, we turned our attention to the use of a steady-state tube furnace. The use of a tube furnace in which to generate and study toxic gases such as CO, CO2, and HCN from different materials has been documented in the literature (19,20), but to this author's knowledge has not been used to produce and characterize pyrolysis products in a low oxygen environment for use in fire debris examinations. The purpose of this paper is to describe a method that uses a tube furnace to reproducibly create pyrolysis products from a variety of substrates and to show how this data may be used in the fire debris analysis laboratory.

Materials and Methods

Formation of Pyrolysis Products

All pyrolysis products were generated by heating samples in Kimax culture tubes (Kimble Chase, Vineland, NJ) inserted into a Carbolite MTF 10/15 mini-tube furnace fitted with an Eurotherm 818 electronic temperature controller (Carbolite, Hope Valley, U.K.). The culture tubes measured 13×100 mm and were fitted with black phenolic screw caps modified as follows: a 0.25" (6.3 mm) diameter hole was bored through the center of the cap, the cap liner was removed, and replaced with a Tegrabond 10/90 mil PFTE/silicon septum (Chromatographic Specialties, Brockville, ON, Canada). A weighed sample was placed in the bottom of the culture tube and the tube capped. The tube was then inserted into the preheated tube furnace such that the end of the culture tube was in the middle of the furnace (a depth of 7.5 cm). Sample dwell time inside the tube furnace was timed using a stop watch. Once the desired time had elapsed, the tube was withdrawn from the furnace and quickly inserted into a cooling block, all the while keeping the tube in a horizontal orientation. The cooling block consisted of a water-jacketed copper tube, where the water bath was maintained at ambient temperature. Tests showed that the cooling block was capable of cooling a culture tube from 400°C to room temperature in <2 min. The pyrolysis products were recovered by injecting 0.5 mL CS₂ through the septum into the cooled tube using a 1 mL luer-slip disposable plastic syringe (National Scientific Company, Rockwood, TN).

Temperature Profile of the Tube Furnace

The air temperature inside the culture tube was measured using a Fluke 51 hand-held, digital thermocouple device (Fluke, Mississauga, ON, Canada) fitted with a K-type thermocouple. The end of the thermocouple was inserted directly into the culture tube through the septum in the cap, and the temperature taken at various distances from the center of the tube furnace. A wire guide was fastened to the outside neck of the culture tube to hold the thermocouple in line with the center axis of the tube, so that no part of the thermocouple would touch the glass walls of the culture tube. The temperature inside the tube was allowed to equilibrate for 5 min before a temperature reading was recorded.

The rate of temperature rise for a substrate inside the culture tube was measured by placing 0.8 g of dry, powdered alumina in the bottom of the culture tube, and holding it in place with a plug of glass wool. The end of the thermocouple was inserted into the middle of the alumina "slug" and the whole assembly heated in the tube furnace. As above, a wire guide was fastened to the outside neck of the culture tube to hold the thermocouple in line with the center axis of the tube, so that no part of the thermocouple would touch the glass walls of the culture tube.

Reproducibility of Pyrolysis Product Formation

Five samples of unbleached Kraft paper (Crownhill Packaging Ltd, Brampton, ON, Canada) were each pyrolyzed at 400°C for 30 min, and five samples of Armstrong vinyl sheet flooring (Armstrong World Industries, Montreal, QC, Canada) were each pyrolyzed at 240°C for 30 min. Disks of Kraft paper (10 mm diameter) were cut with a hole punch and a stack of paper disks weighed out to equal c. 0.1 g. Pieces of vinyl sheet flooring weighing c. 0.15 g $(c. 1 \text{ cm}^2)$ were cut from a larger sheet. The gas chromatographic data were aligned using ChemStation software (Agilent Technologies, Palo Alto, CA) and exported directly from the software as X:Y (scan time/amplitude) coordinates. Data were imported into an Excel spreadsheet (Microsoft, Redmond, WA) and mean centered. From the normalized data, the mean amplitude at each scan was calculated and these data were used to calculate the mean square error for the five samples at each scan.

Preparation of Proficiency Rests

Samples for internal proficiency tests were prepared from either Kraft paper or spruce wood. Samples of Kraft paper (0.1 g) were heated at 360°C for 30 min. One sample of pyrolyzed paper was solvent extracted with 0.5 mL CS₂ alone to give a background of pyrolysis products, while two others were solvent extracted with 0.5 mL CS₂ spiked with 50% evaporated regular gasoline (1:500 in CS₂), and unevaporated Zippo lighter fluid (1:1000 in CS₂), respectively. A fourth sample was prepared by heating a sample of Kraft

paper (0.1 g) spiked with 1 μ L Home Hardware turpentine (spotted onto the paper) at 360°C for 30 min, followed by solvent extraction with 0.5 mL CS₂. A sample of spruce wood (0.1 g) was heated at 360°C for 20 min, followed by extraction with 0.5 mL CS₂ spiked with a medium petroleum distillate (MPD) (Recochem, Nisku, AB, Canada) (1:800 in CS₂). Pyrolsates from the following materials were also generated at temperatures ranging from 280 to 440°C (sample weights are approximate): white pine (0.15 g), plywood (0.3 g), nylon carpet (0.3 g), and acrylonitrile/butadiene/styrene (ABS) plastic (0.16 g).

Gas Chromatography-Mass Spectrometry

Analysis was performed using an Agilent 6890 gas chromatograph fitted with a 30 m \times 0.25 mm i.d. \times 0.25 µm film HPMS-1 capillary column connected to an Agilent 5973 mass selective detector. The GC-MS conditions used were as follows: split injection (1:20) at 250°C; temperature programmed from 40°C (3 min isothermal) to 250°C at 8°C/min (postrun at 300°C for 5 min); and, hydrogen carrier gas held at a constant flow rate of 1.2 mL/min. The detector was operated in the full scan mode (30-300 amu) with a sampling rate of 9.4 scans/sec. Mass spectral searches of the Wiley 275 Mass Spectral Library (1998; Wiley, Hoboken, NJ) were conducted using Agilent ChemStation software. Peaks were identified by searching either the Wiley 275 mass spectral database using Agilent ChemStation software or by searching the NIST 2.0 mass spectral database with the NIST Mass Spectral Search Program (version 2.0 a; National Institute of Standards and Technology, Gaithersburg, MD) after deconvolution of the chromatographic data using AMDIS (version 2.1; National Institute of Standards and Technology).

Results and Discussion

Temperature Profile of the Tube Furnace

It was important to characterize the temperature profile of the tube furnace in order to relate the tube furnace settings to the actual temperature that the sample would be exposed to inside the culture tube. The placement of the centrifuge tube and sample inside the tube furnace is illustrated in Fig. 1. The temperature profiles inside an empty culture tube heated in the tube furnace are shown in Table 1 and are plotted in Fig. 2. The temperature measured by the thermocouple at the center of the furnace agreed well with the set point temperature of the furnace. The air temperature inside the culture tube dropped off toward the entrance of the furnace. When the furnace was set to a higher temperature, for example, 400°C,



FIG. 1—Cross-section showing placement of the centrifuge tube and sample inside the tube furnace.

 TABLE 1—Temperature profile of air inside culture tube heated in a tube furnace.

Set Point ([°] C)	Distance from Center of Furnace (cm)				
	0	1.5	3.5	5.5	7.5
200	197	191	164	114	57
250	248	240	207	148	68
300	299	288	251	178	84
350	345	337	292	217	94
400	396	385	331	253	103



FIG. 2—Temperature profile of air inside culture tube heated in a tube furnace.

the temperature inside the culture tube at the entrance of the furnace was c. 100°C, a temperature low enough to allow heavier volatile compounds to condense at the cooler end of the culture tube and thereby reduce the possibility of the primary pyrolysis products undergoing secondary pyrolysis/cracking.

The temperature rise of a "slug" of alumina was also measured to observe how rapidly the temperature of a sample may increase once it is introduced into the furnace (i.e., radiant heat flux). It was found that for all furnace set point temperatures, the temperature of the alumina would plateau within 5 min (Fig. 3). Clearly, the temperature rise for a sample is expected to depend on its structure and composition; however, the thermal mass for samples on the order of 0.1–0.2 g in weight will be small and so the initial heating rate for any sample (e.g., wood, carpet, or vinyl) is expected to be similar to that of the alumina slug. Similar tests conducted with the thermocouple in the same position, but in air, showed that the temperature inside the tube would plateau within 3 to 4 min after the culture tube was inserted into the furnace.



FIG. 3—Temperature profile of alumina "slug" inside culture tube heated in a tube furnace.

Reproducibility of Pyrolysis Product Formation

The ability to reproducibly generate pyrolysis products will not only allow pyrolysis chromatograms to be databased in a library of backgrounds for use in fire debris casework, but also will allow specific test samples to be created for inter and intra-laboratory proficiency tests, including creating standardized tests for accelerant detection canines. Plots of the mean square error as a function of scan (retention time) were made to assess the reproducibility of the pyrolysis method over five samples. Plots of the normalized Kraft paper data and the calculated mean square error are shown in Fig. 4. Visual comparison of the five normalized chromatograms clearly showed that good reproducibility was achieved in not only the products formed during pyrolysis, but also good agreement in the relative ratios between the products.

When the mean square error is calculated from multiple chromatograms, it will show not only differences between the peak heights, but also differences in chromatography such as peak shape and shifts in retention time. Slight shifts in retention over the 30 min chromatographic run and differences in some peak widths contributed to most of the error calculated from the chromatograms obtained from five samples of the same vinyl flooring pyrolyzed under the same conditions (240°C for 30 min). In order to demonstrate that the pyrolysis method was capable of repeatedly generating pyrolysis products in the same relative ratios, a smaller region of the chromatogram, where the peaks were not tailing, was examined. Prior to aligning the chromatographic data, the mean square error was relatively large for a series of phthalate esters eluting in the region between 26.0 and 27.4 min. However, when normalized data from this region of each chromatogram were manually aligned, and the mean square error recalculated, it was found that the formation of this complex series of pyrolysis products was very reproducible (Fig. 5).

Proficiency Tests

The creation of proficiency tests using the tube furnace was also evaluated. For these proficiency tests, a small amount of ignitable liquid was spiked into the solvent used to extract the pyrolysis



FIG. 4—Plots of mean centered chromatographic data from five samples of Kraft paper each pyrolyzed at 400° C for 30 min and the mean square error calculated from the five chromatograms.



FIG. 5—Plots of mean centered, manually aligned chromatographic data from five samples of vinyl flooring each pyrolyzed at 240°C for 30 min and the mean square error calculated from the five chromatograms.

products. In the case of the Kraft paper pyrolsates spiked with 50% evaporated gasoline, the gasoline pattern is not easily seen in the total ion chromatogram (TIC), but can be clearly seen in the extracted ion profile (EIP) for the alkylbenzenes (Fig. 6). Likewise, a lighter fluid cannot be discerned in the TIC dominated by Kraft paper pyrolysates, but the pattern for the lighter fluid is visible in the aliphatic EIP (Fig. 7). In a similar way, volatile compounds from the pyrolysis of cellulose and lignin in spruce wood will obscure the pattern of an MPD in the TIC, but the alkane pattern of the MPD is visible in the alkane EIP (Fig. 8). Kraft paper and softwood were used in these tests because they gave a large number of compounds over a wide range of boiling points when pyrolyzed; however, other materials such as carpet or vinyl sheet flooring could have been used instead.

Pyrolysis of alpha-pinene is known to generate significant amounts of dipentene (1-methyl-4-[1-methylethenyl]-cyclohexene, or limonene) (21,22), and this was observed in one of the proficiency test samples where commercial turpentine was pyrolyzed in the presence of Kraft paper (Fig. 9). This is an interesting result and shows that softwood, which may contain large amounts of alpha-pinene, can give rise to large amounts of dipentene in fire debris.

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FIG. 6—Total ion chromatogram (TIC) (top) and alkylbenzene extracted ion profile (EIP) (middle) of a proficiency test sample created from the pyrolysis of Kraft paper (360° C for 30 min) spiked with 50% evaporated gasoline. The alkylbenzene EIP of the 50% evaporated gasoline is shown for comparison (bottom). Peak assignments: (1) toluene; (2) ethylbenzene; (3) p- and m-xylene; (4) o-xylene; and (5) 1,2,4-trimethylbenzene.



FIG. 7—Total ion chromatogram (TIC) (top) and alkane extracted ion profile (EIP) (middle) of a proficiency test sample created from the pyrolysis of Kraft paper ($360^{\circ}C$ for 30 min) spiked with lighter fluid. The alkane EIP of the lighter fluid is shown for comparison (bottom).

Profiles of pyrolysis compounds similar to those seen in chromatograms from casework can be generated by pyrolyzing samples in a tube furnace. For example, pieces of burnt softwood from a structure fire are often submitted for analysis to determine whether an ignitable liquid was present. Figure 10 compares the volatiles generated when the softwood cribbing underneath a mobile home was intentionally ignited with the volatiles produced when 0.15 g of pine wood was pyrolyzed in a tube furnace (320°C for 30 min). The pyrolysis of cellulose at a moderate temperature (i.e., 400°C) has been shown to produce 2-furancarboxaldehyde (furfural) and



FIG. 8—Total ion chromatogram (TIC) (top) and alkane extracted ion profile (EIP) (middle) of a proficiency test sample created from the pyrolysis of spruce wood ($360^{\circ}C$ for 20 min) spiked with medium petroleum distillate (MPD). The alkane EIP of the MPD fluid is shown for comparison (bottom).



FIG. 9—Chromatogram of 0.1 g Kraft paper pyrolyzed with 1 µL turpentine at 360°C for 30 min. Peak assignments: (1) alpha-pinene; and (2) limonene.



FIG. 10—Comparison of chromatographic profiles obtained from burnt softwood cribbing collected from an arson scene with pyrolysis of 0.15 g of softwood (white pine) in a tube furnace. Tentative peak assignments: (1) 2-furancarboxaldehyde (furfural); (2) 5-methyl-2-furancarboxaldehyde; (3) 2-methoxyphenol (guaiacol); (4) 2-methoxy-4-methylphenol (4-methyl guaiacol); and (5) 4-ethyl-2- methoxyphenol (4-ethyl guaiacol).

5-methyl-2-furancarboxaldehyde (5-methyl-furfural) (23), while pyrolysis of lignin at temperatures between 400 and 600°C will produce significant amounts of 2-methoxyphenol (guaiacol) and its



FIG. 11—Comparison of the alkylbenzene extracted ion profiles $(m/z^+$ 78, 91, 92, 105, 106, 119, 120, 134, 135) obtained from fire debris containing burnt acrylonitrile-butadiene-styrene (ABS) plastic from an arson scene with pyrolysis of 0.16 g of ABS plastic in a tube furnace. Tentative peak assignments: (1) toluene; (2) ethylbenzene; (3) styrene; (4) 1-methylethylbenzene; (5) alpha-methylstyrene; (6) benzenebutanenitrile; (7) 1-(phenylmethyl)-1H-pyrrole; and (8) 1-benzylpyrrole.

alkylated analogs (24). Another example of the creation of pyrolysis compounds similar to those observed in casework is illustrated in the alkylbenzene EIPs shown in Fig. 11. In this case, fire debris containing burnt ABS pipe had a number of compounds reported previously in the literature (25,26), including styrene, alpha-methyl-styrene, and benzenebutanenitrile. The generation of these compounds, and others, by pyrolyzing a piece of black ABS plastic plumbing pipe in the tube furnace confirmed the source of these compounds. The simulation of pyrolysis conditions may be further explored by changing the temperature with time to simulate the temperature rise experienced by substrates as a fire grows (11). The tube furnace used here would lend itself well to this type of study because the temperature of the furnace is fully programmable.

Conclusion

A simple, reproducible method for the generation of pyrolysis products from a variety of substrates using a mini-tube furnace has been demonstrated. This method is currently being used in our laboratory to generate a new library of pyrolysis products to help identify compounds in chromatograms of fire debris extracts. This method has been successfully used to create chromatographic data for several internal proficiency tests and has also been used to generate nearly identical replicate backgrounds for testing accelerant detection canines. This method may also be used to help characterize those compounds that contribute to fire toxicity and so pose an occupational health risk to fire fighters and the general public.

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References

 International Union of Pure Applied Chemistry (IUPAC). Nomenclature and terminology for analytical pyrolysis. Pure Appl Chem 1993;65: 2405–9.

- Cooley JH, Williams RV. The pyrolysis of polystyrene. Chem Educ 2003;8(5):309–11.
- IUPAC. Compendium of chemical terminology, 2nd edn. Compiled by McNaught AD, Wilkinson A. Oxford, UK: Blackwell Scientific Publications, 1997.
- Hurd CD. The pyrolysis of carbon compounds—American Chemical Society Monograph Series No. 50. New York, NY: American Chemical Society, 1929.
- Mohan D, Pittman CU Jr, Steele PH. Pyrolysis of wood/biomass for bio-oil: a critical review. Energy Fuels 2006;20(3):848–89.
- Moldoveanu SC. Pyrolysis of organic molecules with applications to health and environmental issues. Techniques and instrumentation in analytical chemistry, Vol. 28. Amsterdam, The Netherlands: Elsevier, 2009.
- Evans RJ, Milne TA. Molecular characterization of the pyrolysis of biomass. 1 Fundamentals. Energy Fuels 1987;1(2):123–37.
- Guar S, Reed TB. Pyrolysis of the components of biomass. In: Thermal data for natural and synthetic fuels. New York, NY: Marcel Dekker, 1998);55–101.
- Prins MJ, Ptasinski KJ, Janssen FJJG. Torrefaction of wood. Part 1. Weight loss kinetics. J Anal Appl Pyrolysis 2006;77(1):28–34.
- Prins MJ, Ptasinski KJ, Janssen FJJG. Torrefaction of wood. Part 2. Analysis of products. J Anal Appl Pyrolysis 2006;77(1):35–40.
- Lennon T, Bullock MJ, Enjily V. The fire resistance of medium-rise timber frame buildings. Proceedings of the World Conference on Timber Engineering; 2000 July 31–Aug 3; British Columbia, Canada, 2000; Paper 4-5-4, http://timber.ce.wsu.edu/Resources/ (accessed June 8, 2009).
- Ettling B, Adams M. The study of accelerant residues in fire remains. J Forensic Sci 1968;13(1):76–89.
- Howard H, McKague A. A fire investigation involving combustion of carpet material. J Forensic Sci 1984;29(3):919–22.
- DeHaan J, Bonarius K. Pyrolysis products of structure fires. J Forensic Sci Soc 1988;28(5-6):299–309.
- Bertsch W. Volatiles from carpet: a source of frequent misinterpretation in arson analysis. J Chromatogr A 1994;674(1-2):329–33.
- 16. Chasteen C, Hurchins R, Render M. Preparation of pyrolysis standards to approximate pyrolysis products observed in fire scenes. In: Proceedings of the International Symposium on the Forensic Aspects of Arson Investigations; 1995 July 31–Aug 4; Fairfax, VA. Washington, DC: U.S. Dept. of Justice, Federal Bureau of Investigation, 1995;331–4.
- 17. Stauffer E. Identification and characterization of interfering products in fire debris analysis [dissertation]. Miami (FL): Florida International University, 2001.
- Locke A, Basara G, Sandercock P. Evaluation of internal standards for the analysis of ignitable liquids in fire debris. J Forensic Sci 2009;54(2):320–7.
- Hull TR, Paul KT. Bench-scale assessment of combustion toxicity—a critical analysis of current protocols. Fire Safety J 2007;42(5):340–65.
- Stec AA, Hull TR, Lebek K. Characterisation of the steady state tube furnace (ISO TS 19700) for fire toxicity assessment. Polym Degrad Stab 2008;93(11):2058–65.
- 21. Goldblatt LA, Palkin S. Vapor phase thermal isomerization of α and β -pinene. J Am Chem Soc 1941;63(12):3517–22.
- 22. Stolle A, Ondruschka B, Hopf H. Thermal rearrangements of monoterpenes and monoterpenoids. Helv Chim Acta 2009;92(9):1673–719.
- Sakuma H, Munakata S, Sugawara S. Volatile products of cellulose pyrolysis. Agricultural Biological Chem 1981;45(2):443–51.
- Jiang G, Nowakowski DJ, Bridgwater AV. Effect of the temperature on the composition of lignin pyrolysis products. Energy Fuels 2010;24(8): 4470–5.
- Rutkowski JV, Levin BC. Acrylonitrile-butadiene-styrene copolymers (ABS): pyrolysis and combustion products and their toxicity—a review of the literature. Fire Mater 1986;10(3-4):93–105.
- Jun HC, Oh SC, Lee HP, Kim HT, Yoo KO. Pyrolysis characteristics of acrylonitrile-butadiene-styrene resin. J Ind Eng Chem 1999;5(2):143–9.

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