

ITEX Application Note # 03

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Arson Detection using ITEX Headspace Sampling

Abstract

The analysis of residues of fire accelerants in fire debris samples can be used for arson detection. The analysis can be performed by ITEX sampling, followed by GC-MS analysis. In comparison to static headspace, the sensitivity is increased by a factor of 10 using ITEX enrichment.

Introduction

The detection of residues of fuels (gasoline, naphtha, kerosene) or organic solvents such as paint thinner in fire debris samples is an important application in forensic analysis. The target compounds include C5-C12 hydrocarbons, aromatic hydrocarbons, ethers and alcohols (methanol). Different methods are used for this analysis including static and dynamic headspace, solid phase micro-extraction, etc. Using static headspace, the sensitivity of the method is often not high enough to detect traces of solvent residues. Higher sensitivity can be obtained using dynamic headspace with enrichment of the volatile organic compounds that are characteristic for fire accelerators. These solutes can be enriched on a Tenax trap. Consequently the solutes are desorbed from the trap and analyzed by GC-MS. Enrichment of VOCs from the headspace of solid or liquid samples can be done fully automated using the in-tube extraction (ITEX) option on the CTC Combipal sampler. Enrichment is done in a Tenax packed modified syringe.

Sample Preparation

Typically 1-5 g material is placed in a 20 mL headspace vial and the samples are analyzed as such. Materials introduced are not homogeneous and therefore often multiple samples are analyzed. For the example showed below, two fire debris samples were taken from a burned wooden floor. Sample A was taken at the place where the fire started and sample B was taken at an area away from the original fire location. From both samples similar amounts were introduced in a 20 mL headspace vial and the vial was sealed.

ITEX conditions

Sample Conditioning @ 80°C, 10 min
Extraction Strokes: 20 x 1 mL; 50 µL/s
Desorption @ 250°C with 1 mL headspace; 50 µL/s
Trap Material Tenax TA 80/100 mesh

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GC conditions

The analysis was performed on an Agilent 6890 GC – 5975 MSD combination.

Column: 20 m x 0.18 mm i.d. x 1 µm df DB-VRX (Agilent)

Carrier gas: helium, 170 kPa constant pressure at inlet (column outlet pressure: 28 kPa using AUX EPC and QuickSwap connector)

Inlet: split, 1/10 split ratio

Oven temperature program: 35°C, 2 min, 8°C/min to 190°C, 20°C/min to 250°C, 2 min.

Detection: MS in scan mode (33-300 amu)

Results

The total ion chromatogram obtained for sample A (suspected sample) is given in Figure 1. The most abundant peaks are identified as alpha-pinene (12.5 min), camphene (12.9 min), and limonene (14.8 min). These compounds are typical pyrolysis products from wood and are no indicators for fire accelerants.

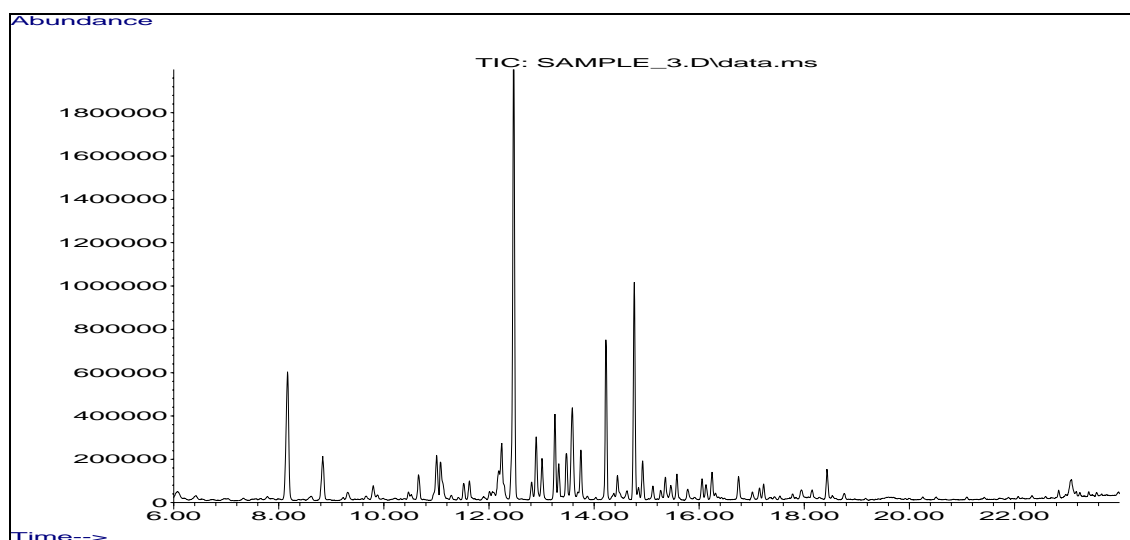


Figure 1

The total ion chromatogram obtained for sample B (believed to be blank) is given in Figure 2. The chromatogram is similar and the major peaks correspond also to the peaks detected in sample A (pyrolysis products of wood).

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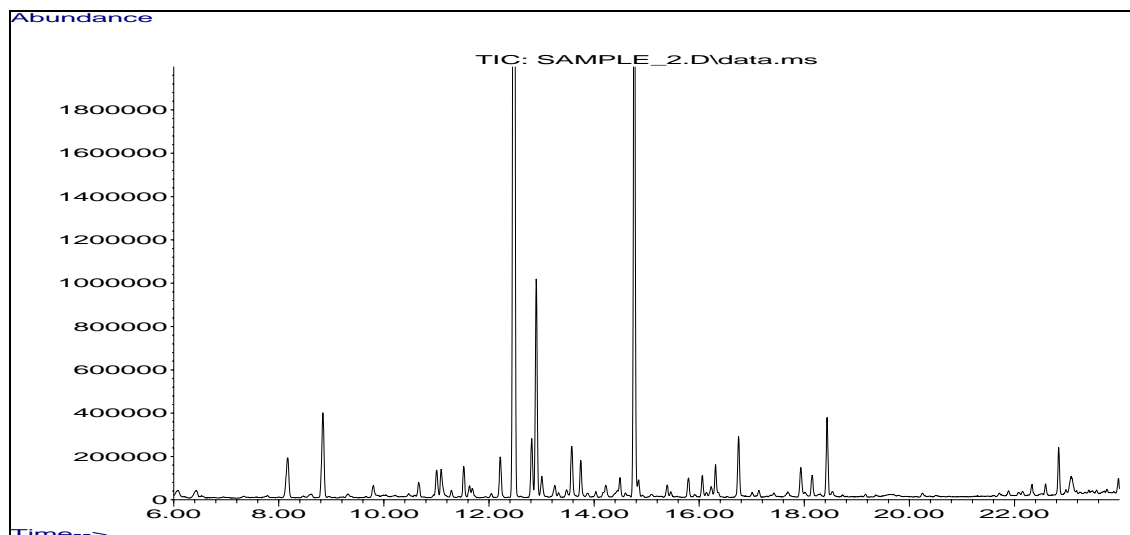


Figure 2

Using extracted ion chromatograms, it is however possible to differentiate both samples. The chromatograms below show the extracted ion chromatograms for m/e 120 (C3-aromatics) and m/e 134 (C4-aromatics) for respectively sample A (Figure 3) and sample B (Figure 4).

It is clear that in sample A, a typical profile of aromatic hydrocarbons is observed, while in sample B only one main peak is detected. This peak was identified as p.cymene and is also a pyrolysis product of wood. The profile of the aromatic hydrocarbons detected in sample A, on the other hand, corresponds to gasoline.

This could be confirmed by analyzing a blank sample spiked with a small amount of gasoline. In Figure 5, the profiles of the C3-aromatics in the spiked sample and in sample A are compared. It is clear that a good correspondence is obtained and that sample A contains traces of a fire accelerant, in this case gasoline.

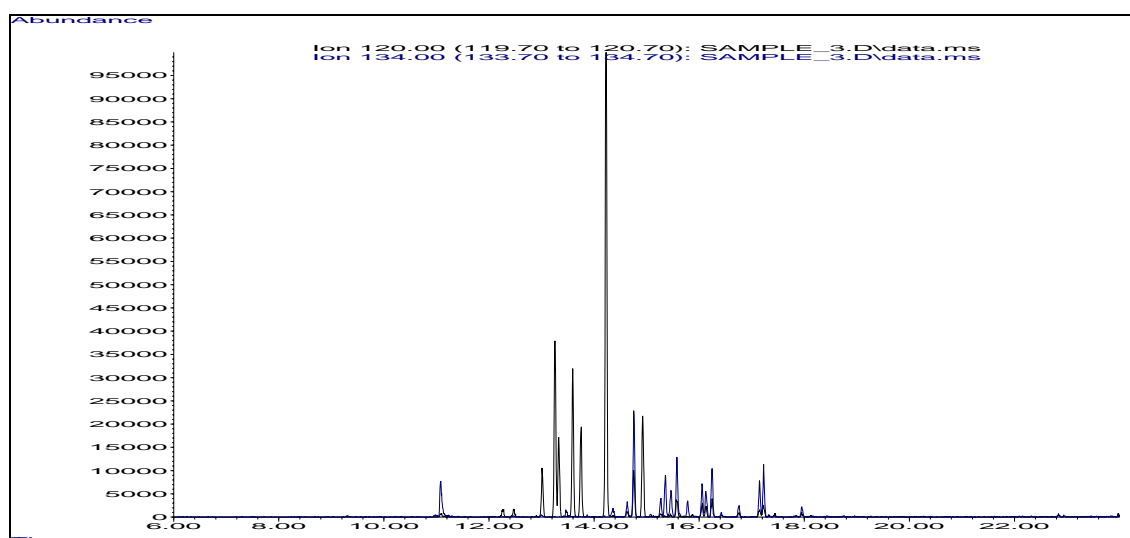


Figure 3

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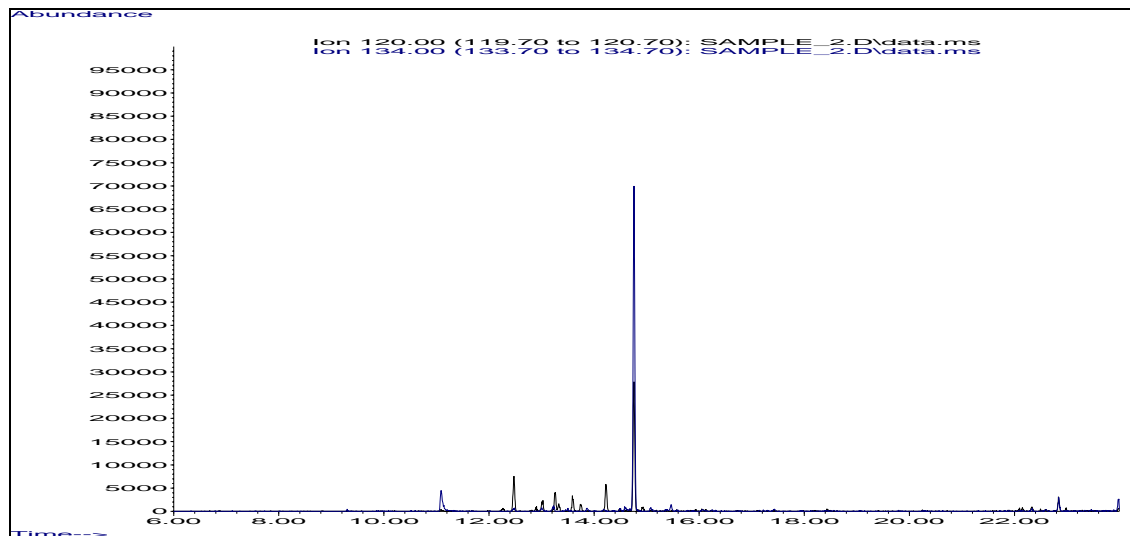


Figure 4

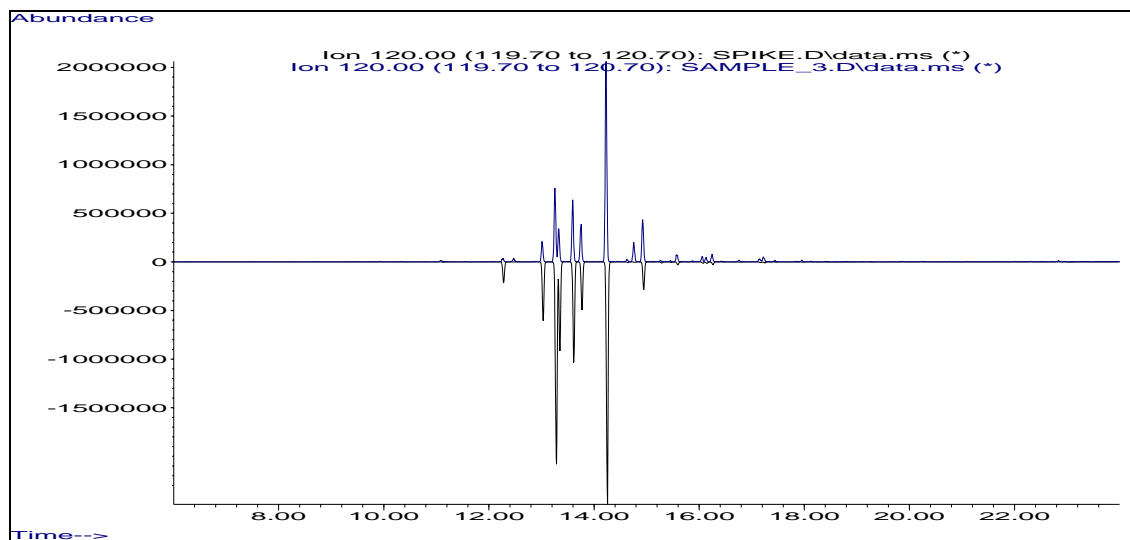


Figure 5: top: sample A (suspected sample); bottom: blank sample spiked with gasoline (reference)

Conclusion

Headspace sampling with enrichment of VOCs using the ITEX option was used for the detection of fire accelerants in fire debris samples. Excellent sensitivities are obtained, allowing detailed profiling of samples. In comparison to static headspace, the sensitivity of the ITEX-GC-MS was increased by a factor of 10, while no discrimination was observed in function of the boiling point of the solutes in the range from C5 to C15.