

TECHNICAL NOTE**CRIMINALISTICS**

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Performance Testing of the New AMPAC Fire Debris Bag Against Three Other Commercial Fire Debris Bags*

ABSTRACT: Fire debris evidence is collected and stored in a wide range of containers, including various polymer bags. Four different polymer bags have been investigated, including the NYLON, DUO, ALU, and AMPAC bags. The latter is the successor of the Kapak Fire DebrisPAK™. Microscopy and infrared spectroscopy were used to elucidate the composition of the bags. Gas chromatography/mass spectrometry was used to investigate performance parameters such as background volatiles, leak rate, cross-contamination, recovery, and sorption. The NYLON bag was susceptible for leakage and cross-contamination and showed decreased recoveries. The DUO and ALU bags showed some background volatiles, sorption, and poor recoveries. The AMPAC bag performed excellent: low background, no leakage or cross-contamination, good recoveries, and only traces of sorption. Heat sealing proved to be the best method of closure. Preliminary studies on AMPAC bags showed that polyethylene clamps are easy to use on-site and preserve ignitable liquids adequately for a limited period of time.

KEYWORDS: forensic science, fire debris, evidence bag, evidence container, ignitable liquid analysis, fire debris bag, packaging

Fire debris evidence collected for ignitable liquid analysis must be properly preserved prior to analysis to avoid diminution (loss) and contamination (1). The sample container, used for collection and storage of the fire debris evidence, should not release interfering background volatiles and should retain the volatiles from the fire debris material without leakage or sorption. Both leakage and sorption can result in decreased recovery, whereas leakage can also increase the potential risk of cross-contamination of volatiles between different sample containers. In practice, sample containers should also be resistant to punctures and cuts, should be available in different sizes for the collection of all sorts and sizes of fire debris evidence, and should be easy to use.

Various types of fire debris containers have been examined, for example, different types of nylon bags (2–4), nylon–polyethylene bags (4), polyester–polyolefin Kapak Fire DebrisPAK™ bags (3–6), polyethylene–polyvinylidene dichloride bags (7), and various cans and jars (3,5,6). The majority of the fire debris evidence collected in the Netherlands is secured in 2.5 L glass jars. When the evidence material is too voluminous or awkward in shape, nylon-11 bags are used as an alternative container, although it is known from own experience that leakage and cross-contamination can occur.

The objective of this study was to find an alternative to the nylon-11 bag. Three other types of bags were selected: a multilayer polyethylene/aluminum bag (ALU), a multilayer nylon/polyethylene bag (DUO), and a dual layer nylon/polyacrylonitril-co-methacrylate-co-butadiene bag (AMPAC). The ALU and DUO bags are, together with nylon bags, the most commonly used collection bags

in Europe today, based on a survey conducted in 2007 among 23 European forensic institutes by the Fire and Explosion Investigation working group of the European Network of Forensic Science Institutes. The AMPAC bag was introduced in 2010 as the successor of the Kapak Fire DebrisPAK™ bag, which was removed from the market several years ago, despite being a suitable container in all regards for fire debris evidence (2,3,5,6). The Kapak bag was not included in this study as it is no longer commercially available.

This study investigated the performance of the four fire debris bags including leak rate, cross-contamination, sorption, and recovery using various ignitable liquids. The majority of the experiments were performed with commercial gasoline. Additional experiments were conducted using methylated spirits and white spirits. The performance was monitored over a period of 8 weeks. Prior to these investigations, the elucidation of the composition of the fire debris bags was addressed. The study is finalized with some preliminary experiments on the on-site closure of the fire debris bags.

Materials and Methods

The four fire debris bags examined in this study were the following: nylon-11 bags (NYLON) obtained from De Ridder B.V. (Uitgeest, the Netherlands), polyethylene/aluminum bags (ALU) received from Gruber Folien (Straubing, Germany), nylon/polyethylene bags (DUO) received from Duotec (Glostrup, Denmark), and nylon/polyacrylonitril-co-methacrylate-co-butadiene bags (AMPAC) received as a test material from Ampac Flexibles (Cincinnati, OH).

The fire debris bags were subjected to both microscopic and Fourier transform infrared analyses to gain information on the composition of the bags. Microscopic analysis was performed with a Bio-Rad UMA-500 transmission microscope at 400× magnification (BIO-RAD Laboratories Europe, Hemel Hempstead, UK). The different layers were subsequently subjected to Fourier transform

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*Presented at the 63rd Annual Meeting of the American Academy of Forensic Sciences, February 21–26, 2011, in Chicago, IL.

Received 18 Feb. 2011; and in revised form 6 July 2011; accepted 19 July 2011.

infrared spectroscopy by means of a Bio-Rad FTS 6000e spectrometer.

The background volatiles of the different fire debris bags were analyzed at room temperature and after heating the particular bag in a glass jar at 70°C for 4 h. Fifty milliliters of headspace was sampled onto a Tenax TA thermal desorption tube (Markes, Llantrisant, UK) and analyzed according to the thermal desorption gas chromatography/mass spectrometry (GC/MS) procedure stated below.

The experimental setup for measurement of leak rate, cross-contamination, recovery, and sorption consisted of two heat-sealed fire debris bags of *c.* 15 × 15 cm of the same type of bag inside a 2.5-L glass jar as shown in Fig. 1. Sealing of the fire debris bags was performed using an Audion Elektro Sealboy 321 (Weesp, the Netherlands). In this study, the 2.5-L glass jar was considered to be a closed system.

One of the bags (a) contained a sample of filter paper that was infused with 10 μL of gasoline. Subsequently, the bag was filled with 100 mL of air. The other bag (b) was only filled with 100 mL of air. Infusion with gasoline and filling with air was carried out by means of a syringe. A puncture was made near one of the corners of the bag. The puncture was closed by an additional diagonal heat seal in the corner of the bag. The 2.5-L glass jars with lids were obtained from De Ridder B.V. The filter paper 589/1 with a diameter of 90 mm, which was used to infuse ignitable liquid onto, was obtained from Whatman (Kent, UK).

To measure leak rate, 50 mL of the headspace (c) was sampled onto a Tenax TA thermal desorption tube and analyzed according to the procedure as stated below. Six repeated measurements were performed after 1, 7, 14, 28, and 56 days. To measure cross-contamination, 50 mL of the headspace in bag (b) was sampled onto a Tenax TA thermal desorption tube and analyzed according to the procedure as stated below. Three repeated measurements were performed after 8, 15, 29, and 57 days. To measure the recovery of the particular ignitable liquid, 5 mL of the headspace in bag (a) was sampled onto a Tenax TA thermal desorption tube and

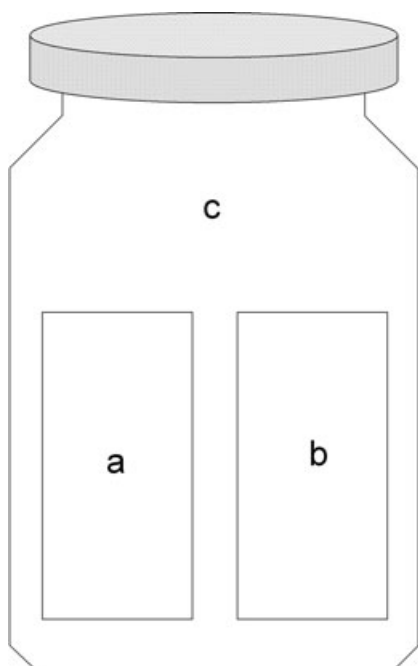


FIG. 1—Schematic representation of the experimental setup for the measurement of leak rate (c), cross-contamination (b), recovery (a), and sorption (a).

analyzed according to the procedure as stated below. Three repeated measurements were performed after 8, 15, 29, and 57 days. The analyses of the Tenax TA thermal desorption tubes were performed using a Markes Unity Ultra TD thermal desorption sampler coupled to an Agilent 7890A gas chromatograph equipped with a 5975C mass-selective detector (Agilent Technologies, Santa Clara, CA). Separation of the components was achieved by means of a 25-m Ultra 1 analytical column with a 0.2-mm inner diameter and a 0.33-μm film thickness. The samples were desorbed from the Tenax TA tube at 280°C and injected with a split ratio of 1:13.5. The oven temperature program started at 35°C for 3 min and increased to 250°C at a rate of 10°C/min where it was maintained for 6 min. The mass spectra were scanned over a range of 17–300 amu. To put the results of the leak rate experiments in the right perspective, a reference experiment was performed in which 10 μL of gasoline was deposited in a 2.5-L glass jar. Four repeated measurements of the reference experiment were performed. To quantify leak rates in a practical manner, eight distinctive components of gasoline were nominated as marker compounds. For each component, the average peak area obtained by GC/MS in the leak rate experiment was divided by the average peak area of the selected component in the reference experiment. This normalization expresses the leak rate of the particular gasoline component as a percentage of the maximum amount of gasoline present. The selected gasoline components comprised of four aliphatic hydrocarbons (coelution of 2-methylhexane and 2,3-dimethylpentane, iso-octane, and methylcyclohexane), five aromatic hydrocarbons (toluene, ethylbenzene, coelution of *meta*- and *para*-xylene, and 1,2,4-trimethylbenzene), and the oxygenate methyl *tert*-butylether (MTBE). In the case of the cross-contamination experiments, a similar approach was used. However, the volume of the empty bag (b) (100 mL) is considerably smaller than the glass jar (2650 mL). A volume-based correction factor was used to account for these differences.

Volatile components may penetrate different layers of the bags at different rates. The occurrence of migration depends on the type of volatiles and on the composition of the layers. Complete migration through all the layers results in leakage. Partial migration may result in adsorption and/or absorption by one or more of the layers of the bag. It is unclear whether adsorption or absorption or both occur, therefore both processes were explored under the experiments measuring sorption. After 57 days, bag (a) (Fig. 1) was opened and left in a fume hood for 24 h to evaporate the volatile fraction of the remaining ignitable liquid. Subsequently, the inside of these bags was rinsed with 2 mL of pentane. Two repeated measurements were performed. Pentane was purchased from Sigma-Aldrich (Steinheim, Germany). The pentane extracts were analyzed by GC/MS to detect sorption by the inner layer of the bag. The measurements were compared with pentane extracts of a reference experiment. In the reference experiment 10 μL of gasoline was added to a glass surface. After 24 h, the glass surface was rinsed with pentane and analyzed. The analyses of pentane extracts in the sorption experiments with gasoline were performed on an Agilent 6890N gas chromatograph equipped with a 5973 mass-selective detector. Separation of the components was achieved by means of a 25-m Ultra 1 analytical column with a 0.2-mm inner diameter and a 0.33-μm film thickness. The samples were injected at 250°C with a split ratio of 1:50. The oven temperature program started at 35°C for 2 min, increased to 200°C at a rate of 5°C/min, and then to 275°C at a rate of 15°C/min. Mass spectra were scanned over a range of 35–300 amu. The sorption experiments have been interpreted qualitatively. The magnitude of the observed changes in the chromatograms can be related to the degree of sorption.

Additional studies on potential leak rate and cross-contamination were performed for the AMPAC fire debris bag with 250 μL gasoline, 250 μL medium petroleum distillate (white spirit), and 10 μL oxygenated product (methylated spirit), respectively. The experiments with gasoline and the medium petroleum distillate were performed in six repetitions, using the same experimental setup (Fig. 1) and procedures as described above. In the experiments with methylated spirit, the headspace samplings were injected directly into a GC equipped with a flame ionizing detector, as the Tenax TA thermal desorption tubes are not well suited for the trapping of ethanol, the major component of methylated spirit. The analyses of the headspace samplings in the experiments with methylated spirit were performed using an Agilent 6890 gas chromatograph equipped with a flame ionizing detector. Separation of the components was achieved by means of a 30-m Agilent/J&W DB-624 methyl phenyl cyanopropyl polysiloxane analytical column with a 0.32-mm inner diameter and a 1.8 μm film thickness. The samples were injected at 250°C with a split ratio of 1:20. The oven temperature program started at 80°C and increased to 225°C at a rate of 40°C/min. The flame ionizing detector was held at 250°C.

Two alternative closing techniques have been addressed in this study, viz., closure by means of aluminum tape and closure by using a low-density polyethylene clamp and rod, the so-called C-Clamp. Aluminum tape was purchased at 3M (Zoeterwoude, the Netherlands), and C-Clamps were purchased at Jensen Inert Products (Coral Springs, FL). The C-Clamp consists of a high-density polyethylene rod and a C-shaped clamp of the same material. Sealing is carried out by placing the bag on the open side of the clamp. The rod can be pushed into the clamp resulting in an apparently airtight seal. The studies on the closure of the AMPAC bag were performed in a separate experiment with the same experimental setup as described above (Fig. 1). In this case, the two 15 \times 15 cm fire debris bags were closed on one side by means of either tape or C-Clamps. Leakage was measured after 2, 6, 24, 48, and 168 h, and cross-contamination was measured after 192 h. Additional studies to investigate long-term (up to 2 years) performance of the AMPAC bag are currently in progress.

Results and Discussion

Composition

Microscopic analyses revealed that number of layers of the fire debris bags varied from one to four. The NYLON bag consisted of only one layer, the AMPAC bag consisted of two layers, and the ALU and DUO bags consisted of four different layers. For each

bag, the observed layers were subjected to infrared spectroscopic analysis to determine the composition of each layer. Several polymers and copolymers have been identified ranging from various types of polyamide and polyethylene to copolymers such as polyacrylonitril-co-methacrylate-co-butadiene. The total thickness of the bags varied from 40 μm for the NYLON bag to 124 μm for the ALU bag. The results of the microscopic and spectroscopic analyses are summarized in Table 1.

Background Volatiles

The release of volatiles from the polymer of the fire debris bag could interfere with the interpretation of the results in casework. All four bags released negligible quantities of volatiles at room temperature. After heating, a common preparative step in fire debris analysis, some background volatiles were observed. The chromatograms resulting from the background analyses after heating are shown in Fig. 2.

After heating, the NYLON bag was still virtually free of background volatiles, the AMPAC bag released traces of 2-butanone and ethylacetate as well as some aldehydes and ketones that were observed near the detection limit. Although these compounds are not likely to interfere with ignitable liquid patterns, it is important for the fire debris investigator to be aware of these results. The ALU and DUO bags released linear and branched alkanes in the range of C_{10} – C_{16} , which could interfere with the pattern of petroleum distillates and thus could hamper the identification thereof.

Leak Rate

Leakage was monitored in time and analyzed in six repeated measurements. Typical chromatograms of the analyses of leakage after 14 days are shown in Fig. 3. Average leakage of eight selected gasoline components is shown in Figs 4–7 for the NYLON, DUO, ALU, and AMPAC fire debris bags, respectively.

The NYLON bag showed leakage of aromatic hydrocarbons within 7 days after the infusion with gasoline. Average leakage of toluene, ethylbenzene, and *m*-/*p*-xylene was calculated at 26%, 3%, and 4%, respectively, of the total amount of gasoline. The leakage of the aromatic hydrocarbons increased over time, while the aliphatic hydrocarbons and the oxygenate MTBE were well contained. The NYLON bag proved to be to the least gastight of the four bags, especially for the aromatic hydrocarbons. The decreased leakage of toluene, which was observed after 56 days (58%) compared to the leakage level after 28 days (71%), could be owing to the occurrence of cross-contamination and the accompanying

TABLE 1—Indication of the layer composition of the four fire debris bags as determined by Fourier transform infrared spectroscopy. The thickness of the particular layer (μm) is stated between brackets.

Fire Debris Bag	Layer			
	a (Outside)	b	c	d (Inside)
NYLON	Polyamide-11 (40)	—	—	—
DUO	Polyamide-6 (8)	Mix of polyamide-6 and polyethylene (5)	Mix of polyamide-6 and polyethylene (9)	Polyethylene with polyvinylacetate (55)
ALU	Polyethylene terephthalate (14)	(LD) polyethylene (15)	Aluminum (12)	(LD) polyethylene (83)
AMPAC	Polyacrylonitril-co-methacrylate-co-butadiene (26)	—	—	Polyamide 6.6 (47)

LD, low density.

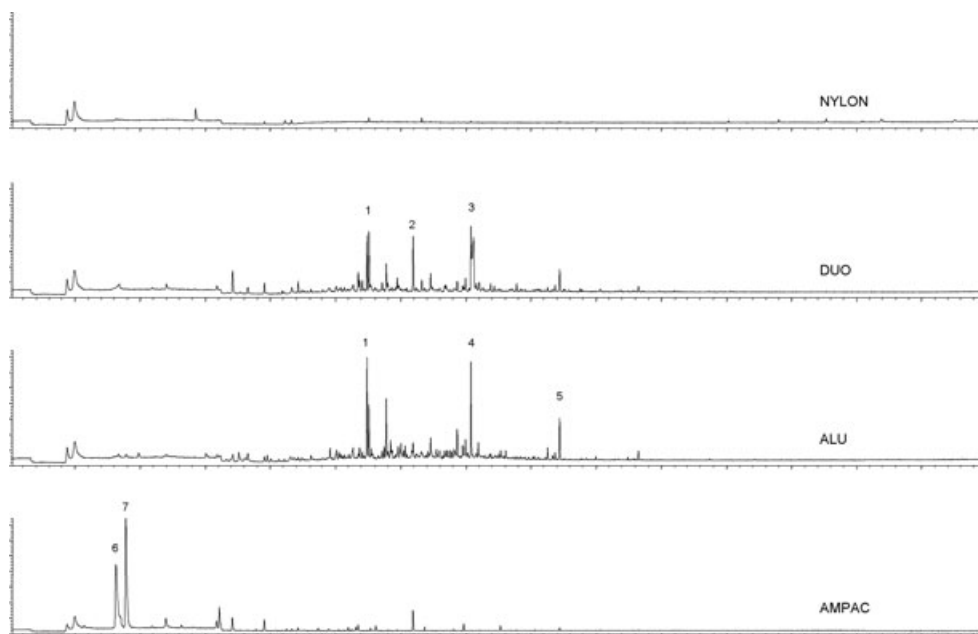


FIG. 2—Chromatograms of the background volatiles analyses of the different types of fire debris bags. The chromatograms are displayed with equal scales on the y-axis (1: pentamethylheptane and decane, 2: nonanal, 3: dodecane and caprolactam, 4: dodecane, 5: tetradecane, 6: 2-butanone, 7: ethylacetate).

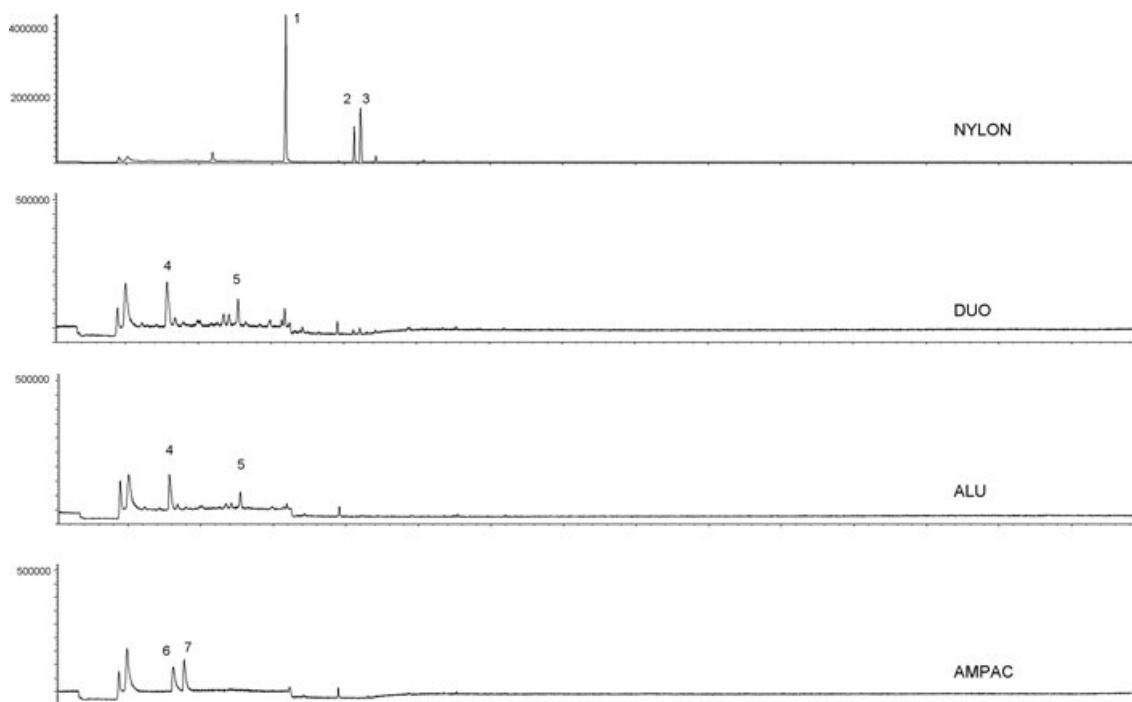


FIG. 3—Typical chromatograms of the leakage of gasoline from the different types of fire debris bags. Note the different scales on the y-axis (1: toluene, 2: ethylbenzene, 3: *m/p*-xylene, 4: MTBE, 5: iso-octane, 6: 2-butanone, 7: ethylacetate). MTBE, methyl tert-butylether.

equilibriums that require some time to be established. The other three bags were adequately gastight up to 56 days. The ALU and DUO bags showed minimal leakage of volatile aliphatic and aromatic hydrocarbons and MTBE. For most of the monitored components, leak rates were well under 2% even after a 56-day time interval. The AMPAC bag proved to be the least susceptible to leakage, with the rate of leakage measured in terms of the monitored gasoline components at or below limit of detection (LOD)

level. The 2-butanone and ethylacetate that were observed in the leak rate experiments of the AMPAC bag were elucidated as background volatiles from the AMPAC material.

Cross-Contamination

Cross-contamination occurs when volatiles in one fire debris bag escape and subsequently migrate into an adjacent fire debris bag.

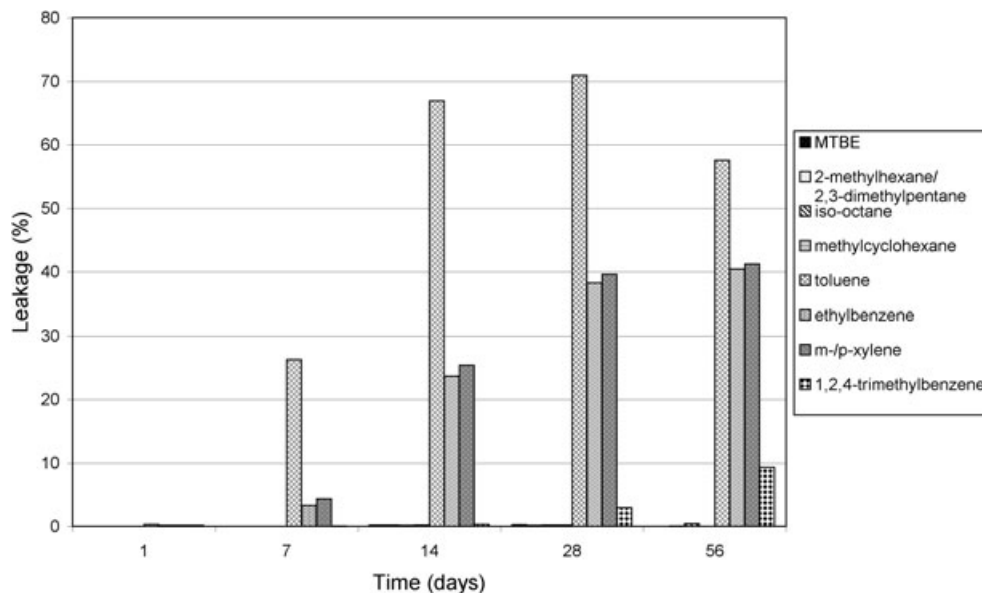


FIG. 4—Average leakage of selected gasoline components from the NYLON bag.

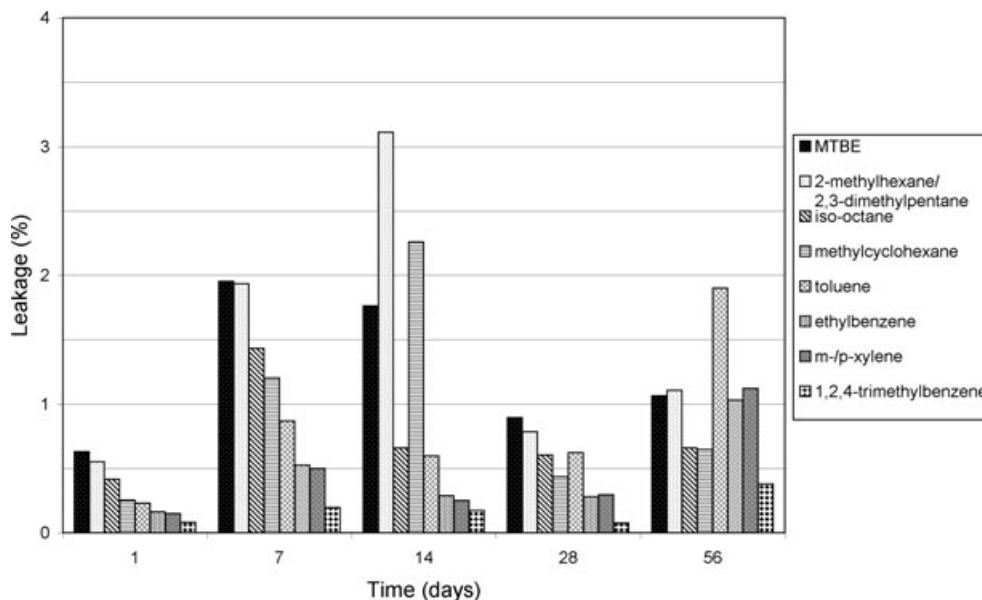


FIG. 5—Average leakage of selected gasoline components from the DUO bag.

Cross-contamination must be prevented as it may result in false-positive identifications. The analysis of a sealed fire debris bag filled with air and placed inside the test setup adjacent to a similar bag infused with gasoline provided information on the occurrence of cross-contamination of ignitable liquids. The results are in line with the results obtained from the experiments on leakage. Cross-contamination of mainly aromatic hydrocarbons of gasoline was observed for the NYLON bag. Users of these bags should be aware of these properties. NYLON bags are susceptible to cross-contamination of aromatic hydrocarbons. Average cross-contamination levels of selected components of gasoline in the NYLON bag after 59 days are shown in Fig. 8.

After 59 days, cross-contamination levels of up to 3% for toluene and 1% for ethylbenzene and *m-/p*-xylene were observed in the initially empty NYLON bag (b). The AMPAC, DUO, and

ALU bags showed good performance with respect to cross-contamination, with maximum levels far below 1% after 59 days.

Recovery

The results of the leakage and cross-contamination experiments demonstrate that some of the fire debris bags do not fully contain a collected ignitable liquid. To verify this, the recovery was determined for all bags at different time intervals. Adequate recoveries of gasoline were found within 7 days. A decrease in recovery of gasoline was observed after prolonged storage. Typical chromatograms of the gasoline recovered from bag (a) after 57 days are shown in Fig. 9.

The chromatograms obtained from the NYLON bags showed good recoveries of MTBE and the aliphatic hydrocarbons, but a

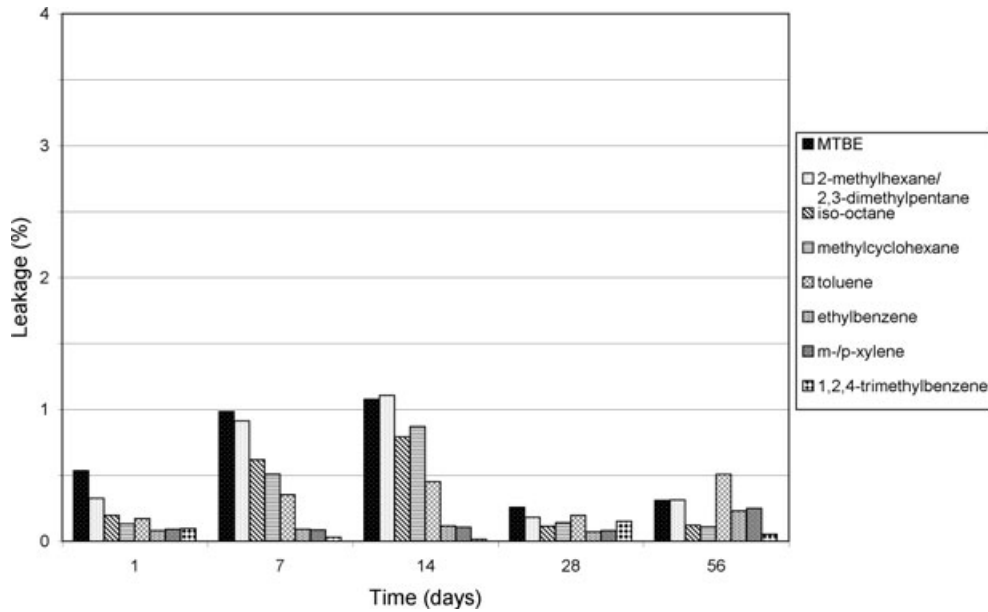


FIG. 6—Average leakage of selected gasoline components from the ALU bag.

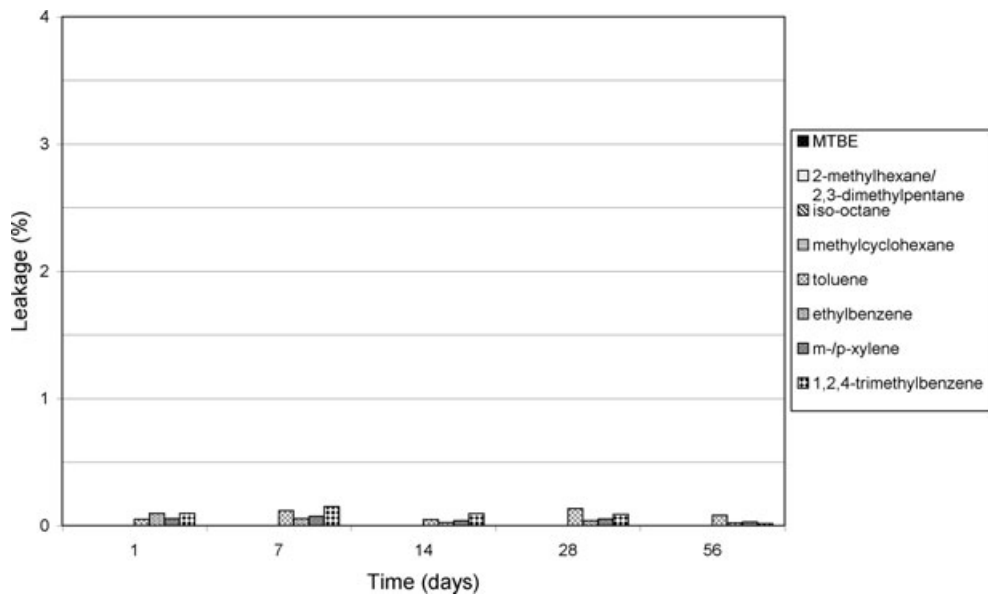


FIG. 7—Average leakage of selected gasoline components from the AMPAC bag.

drastic reduction in the recovery of the aromatic hydrocarbons. The latter observation was expected as most of the aromatic hydrocarbons were released by leakage. The ALU and DUO bags showed a good recovery of MTBE, a reduced recovery of the aliphatic hydrocarbons, and a severely reduced recovery of the aromatic hydrocarbons of gasoline. At the same time, the overall leak rates for these two bags were low. Other processes should account for these findings. The recovery measurements of the AMPAC bag yielded a clear gasoline pattern, even after 56 days.

Sorption

The pentane extracts of the NYLON bags contained low levels of aliphatic hydrocarbons. The pentane extracts of the ALU bag, however, contained substantial amounts of both aliphatic and aromatic hydrocarbons. The chromatographic pattern from the pentane

extract of this bag could be assigned to a partly evaporated gasoline. These results explained the reduced recovery of the gasoline stored in an ALU bag. The pentane extract of the DUO bag contained both aliphatic and aromatic hydrocarbons at considerably lower levels when compared with the pentane extract of the ALU bag. The sorption results cannot fully explain the reduced recovery in the DUO bag. The pentane extracts of the AMPAC bags as well as the pentane extract of the reference experiment showed a negligible amount of compounds that originated from gasoline.

Additional Studies with Higher Concentrations of Gasoline and Other Ignitable Liquids

From the performance test results, it was concluded that the AMPAC bag shows great potential for collection and storage of fire debris evidence. The performance tests of the AMPAC fire

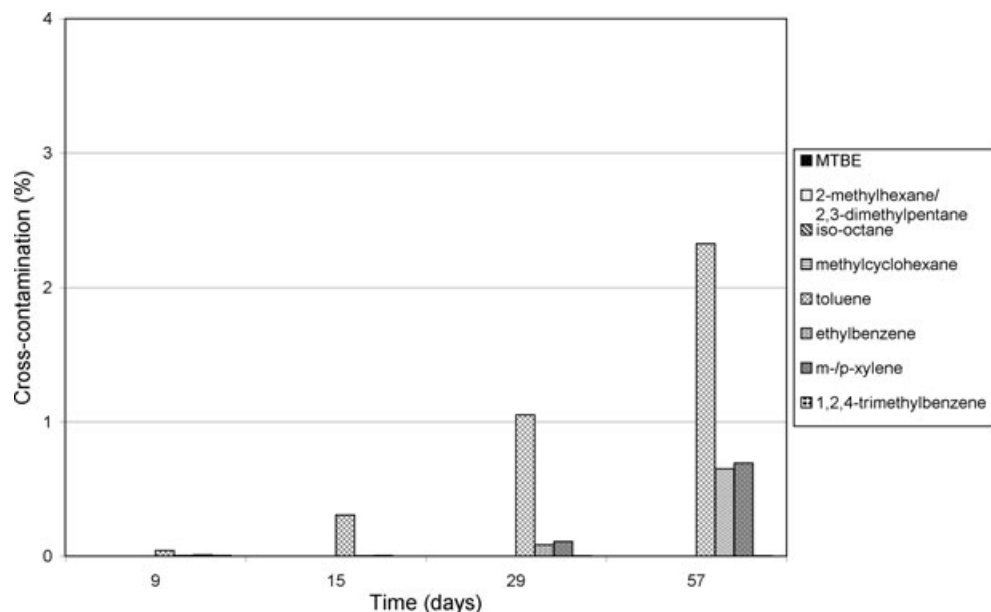


FIG. 8—Average cross-contamination of selected gasoline components for NYLON bags.

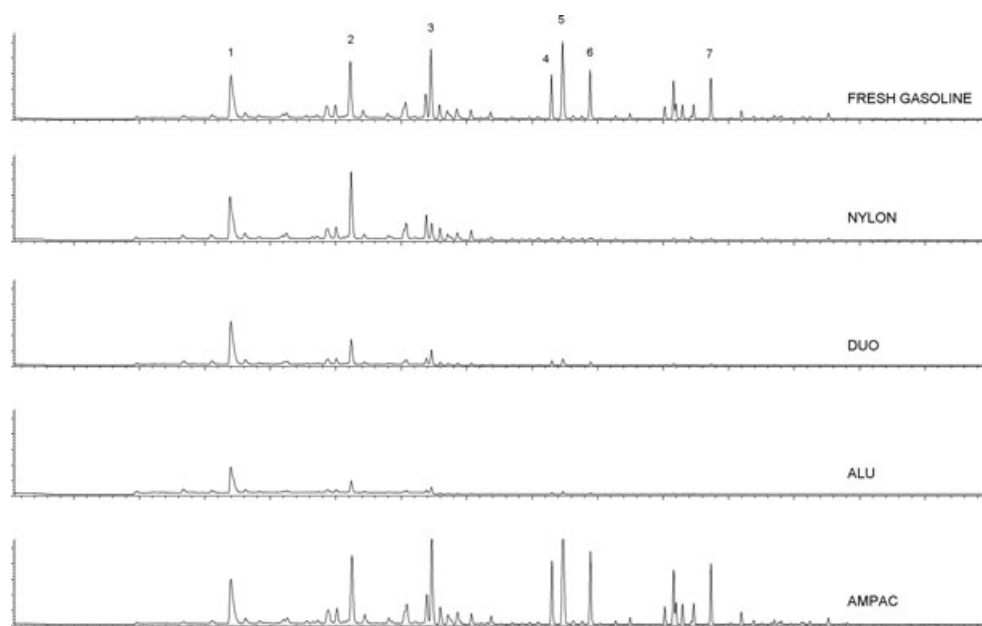


FIG. 9—Average chromatograms of the recovery of gasoline in the four different fire debris bags after 57 days at room temperature. The top chromatogram represents fresh gasoline. The chromatograms are displayed with equal scales on the y-axis (1: MTBE, 2: iso-octane, 3: toluene, 4: ethylbenzene, 5: m-/p-xylene, 6: o-xylene, 7: 1,2,4-trimethylbenzene). MTBE, methyl tert-butylether.

debris bag were therefore extended with three additional tests, viz., a high concentration of gasoline, a high concentration of medium petroleum distillate (white spirit), and an oxygenated product (methylated spirit). In the experiments with 250 μ L of gasoline, leakage was found to occur at levels near the LOD within 14 days. As of day 15, toluene was observed just above the LOD. No cross-contamination was observed. The experiments with methylated spirit, consisting of *c.* 85% ethanol, 10% water, 3% methanol, and traces of several other oxygenates including acetone, 1-propanol, 2-butanone, ethylacetate, 2-methyl-1-propanol, 1,1-diethoxyethane, 3-methyl-1-butanol, and 2-methyl-1-butanol, showed that none of the oxygenates leaked out of the sealed AMPAC bag within the 56-day trial period. This implies that cross-contamination could not

occur, which was verified by actual cross-contamination experiments. Finally, a sealed AMPAC bag was infused with white spirit (C_8 – C_{12} petroleum distillate fraction) and subjected to the same set of experiments. In this case, neither leakage nor cross-contamination was observed during the 56-day trial period. Thus, the additional tests with high concentrations of gasoline, methylated spirit, and a medium petroleum distillate on leakage and cross-contamination confirmed that the AMPAC bags are gastight.

Closing of the Fire Debris Bag

Heat seal closure of the fire debris bags is the best closing technique (1) and therefore employed in the performance testing and

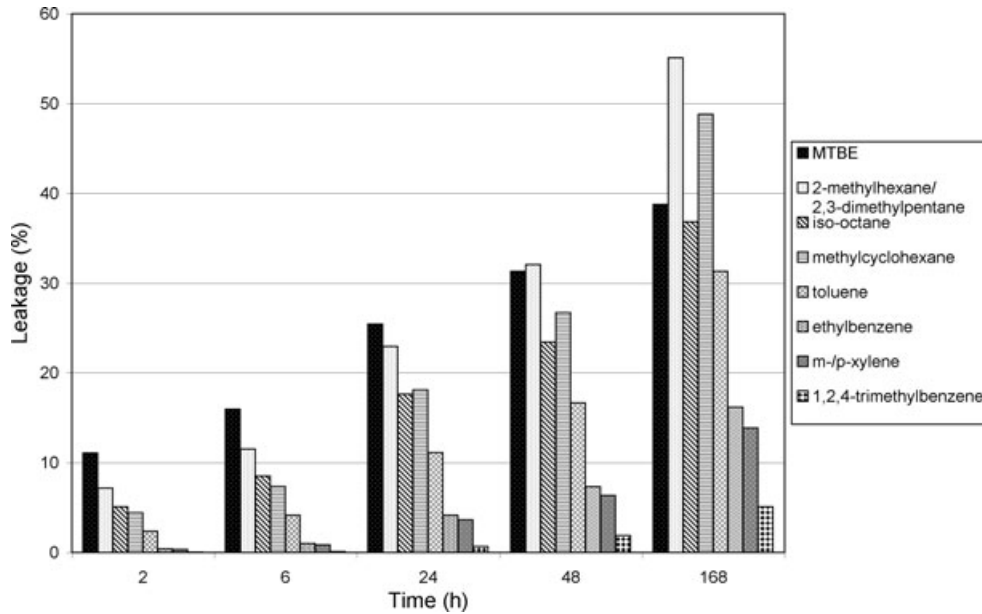


FIG. 10—Average leakage of selected gasoline components monitored in time for the AMPAC bag closed with aluminum tape.

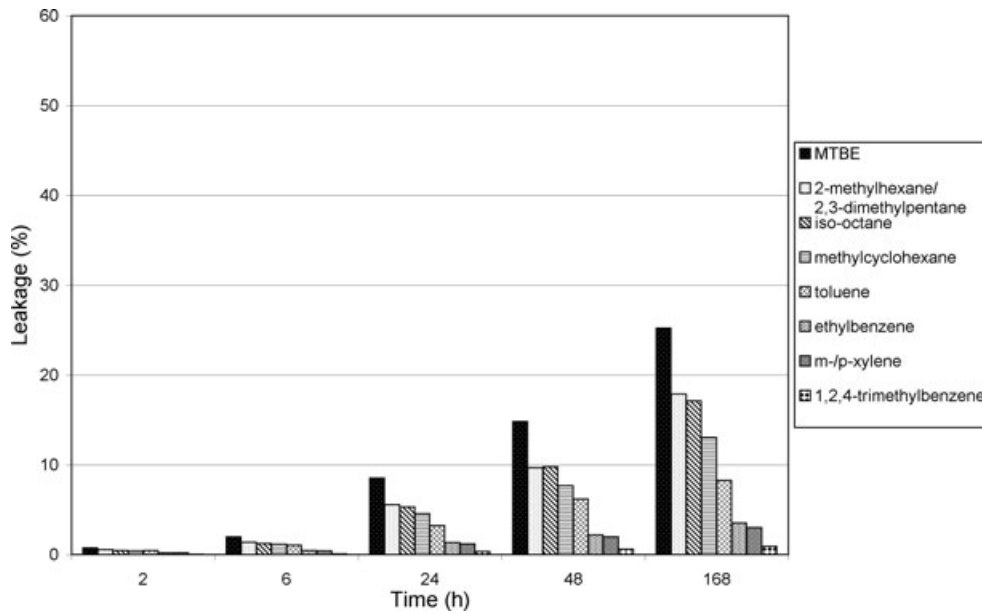


FIG. 11—Average leakage of selected gasoline components monitored in time for the AMPAC bag closed with a C-Clamp.

comparison of the four different fire debris bags described in the previous sections. As heat sealing may not be practical to perform on-site, alternative closing techniques have been examined in this study. At present, the NYLON bags used by the Dutch fire investigators are closed by means of a knot or a swan neck in combination with a cable tie. However, this procedure cannot be applied for the AMPAC bag as the AMPAC material is too thick and stiff. Therefore, an alternative closing procedure is required for the AMPAC bag to be a practical alternative for on-site fire debris collection. Preliminary tests with both aluminum tape and C-Clamps have been performed. The small bags were closed by either the aluminum tape or the C-Clamp. Leakage of the eight selected components of gasoline was monitored over time. Results comparing these two closure methods are shown in Fig. 10 (tape) and Fig. 11 (C-Clamp).

As expected, closing the AMPAC bag by means of tape or C-Clamp proved to be less efficient than closure by heat sealing. Aluminum tape was shown to have a significant effect on leak rates. In 24 h, an average leakage of up to 26% for MTBE, 18% for iso-octane, and 11% for toluene was observed. In 7 days, these percentages were significantly increased up to, for example, 39% for MTBE, 37% for iso-octane, and 31% for toluene. The C-Clamp turned out to be a more efficient closing aid. Leakage of the selected gasoline components stayed below 10% within 24 h. After 7 days, leakages of, for example, 25% for MTBE, 17% for iso-octane, and 8% for toluene were observed. In these experiments, cross-contamination was addressed as well. Cross-contamination levels remained below 1% for both tape- and C-Clamp-closed AMPAC bags throughout

a period of 8 days. This makes the C-Clamp the most promising closing technique on-site as it is easy in use and is able to preserve minimum 92% of the gasoline volatiles for at least 24 h. This time frame is considered to be sufficient to transport the evidence bags from the fire scene to the police station or the laboratory.

Conclusion

The NYLON bag was shown to have excellent properties when it comes to background volatiles, while the levels of leakage and cross-contamination proved to be relatively high. The relatively high leak rate accounted for the reduced recovery of the aromatic hydrocarbons of gasoline. The DUO and the ALU bags performed well in leak rate and cross-contamination experiments, whereas the levels of background volatiles, sorption, and recovery were unsatisfactory. The AMPAC bag demonstrated excellent performance in leakage, cross-contamination, sorption, and recovery experiments. The low leakage and cross-contamination levels were confirmed by additional experiments with the oxygenated product methylated spirit, the medium petroleum distillate white spirit, and high concentrations of gasoline. A minor disadvantage was the release of traces of 2-butanone and ethylacetate upon heating. The performance tests were all conducted with heat-sealed bags. However, heat sealing may not be practical to perform at the fire scene. Preliminary experiments showed that C-Clamps are an easy and reliable alternative technique to close the AMPAC bag at the fire scene, prior to heat sealing at the police station or the laboratory. However, leakage will always occur when the fire debris bag is not closed by means of heat sealing. In conclusion, the AMPAC fire debris evidence bag proved to be an excellent successor of the Kapak Fire DebrisPAK™.

Acknowledgments

The authors would like to thank Gruber Folien for providing ALU bags, Duotec for providing DUO bags, and Ampac Flexibles for providing the AMPAC test material. Fire debris expert L.J.C. Peschier is kindly acknowledged for critical review of the manuscript.

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