

## TECHNICAL NOTE

*Carlotta C. Stackhouse,<sup>1</sup> M.S. and Connie I. Gray,<sup>2</sup> B.S.*

### Alternative Methods for Processing Arson Samples in Polyester Bags

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**ABSTRACT:** Various heating methods for arson samples packaged in polyethylene lined polyester pouches are evaluated. A comparison of the efficiency of accelerant recovery from pouches versus glass jars is also noted. Spiked samples of soil and fire debris are used to simulate actual conditions encountered in arson cases. Advantages and disadvantages of the pouches are discussed.

**KEYWORDS:** forensic science, arson, polyethylene, accelerants

The quest for a fast and simple sample preparation with desired sensitivity parameters has been ongoing for forensic scientists in the arson field. Ambient and heated headspace is of course rapid in sample analysis; however, the sensitivity is limited [1]. Solvent wash, a sensitive method with rapid sample analysis, has the drawback of nonselective inclusion of background material [2]. Steam distillation is a very cumbersome and time-consuming technique. One of the most sensitive methods of extraction, purge and trap, is still time-consuming. Making charcoal traps [3] and using conventional convection heating (ovens and heating mantles) leads to a total extraction time varying from 15 up to 60 min [4].

Recently, the use of microwave ovens to heat polyester bags in arson analysis has been presented [4]. Several papers have stated extraction time to be shortened to 2 min, a 60% decrease in preparation time [5]. The sensitivity also has been stated as comparable to that of the conventional purge and trap for light to heavy petroleum distillates [5].

For years our laboratory has processed arson debris in jars and cans for state and local agencies in South Carolina. In an effort to work with field investigators who wanted to start using polyester bags, we set up methods to extract accelerants from heat sealed bags. In this study we used only 8- by 12-in. (20- by 30 cm) polyethylene-lined polyester Kapak<sup>®</sup> pouches (4 1/2 mils thick). These will be referred to as "bags."

This laboratory has the capability to process six samples at the same time using the purge-

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<sup>1</sup>Chemist, South Carolina Law Enforcement Division, Columbia, SC.

<sup>2</sup>Formerly, laboratory supervisor, South Carolina Law Enforcement Division, Columbia, SC, now, president, Divex, Inc. and Gray Laboratories, Columbia, SC.

and-trap extraction method. To evaluate the efficiency of microwave processing, we recorded the extraction times of heat sealed bags in a microwave, heat-sealed bags in heating mantles, heat sealed bags under a heat lamp, and jars in a heating mantle. We also compared the percent recovery of five components for the different heating methods.

### Experimental Procedure

Four sets of triplicate samples were prepared using a mixture of soil and charred wood debris. Heat sealed bags were used for three sets and new quart mason jars for the fourth set. A piece of Whatman filter paper, spiked with 10  $\mu\text{L}$  of the test standard using a Hamilton syringe, was placed in each container. Fifty millilitres of deionized water were added to all samples and each was sealed. The test standard was a 50/50 mixture of "fresh" gasoline from our state pumps and kerosene obtained from the South Carolina Department of Agriculture. The samples were allowed to equilibrate 30 min before purging.

One corner was cut off all bags and an 8-in. (20-cm) piece of 1/8-in. (0.3-cm) diameter Tygon<sup>®</sup> tubing inserted 2 in. (5 cm) into the bag. Each bag was tied tightly around the tubing with twine.

Set 1 bags were heated in an Amanda/Radarange Model RRL-8XA at full power. The Tygon tubing was passed through a hole drilled in the top back inside corner of the microwave and exited through the vent for the charcoal tube connection. The bags were under constant vacuum. When the bag inflated, heating was stopped until the vacuum deflated the bag. This process was repeated a second time, and then the bag was removed and total extraction time recorded.

The bags in Set 2 were heated in heating mantles. The bags in Sets 2 and 4 had one bottom corner cut off to allow air flow through the bag. Set 2 bags were positioned in a gallon heating mantle with the largest surface area of the bag in contact with the bottom of the mantle. The open corner and the corner attached to the vacuum line were turned up the side of the mantle to prevent water from leaking from the bag.

The jars in Set 3 were heated to approximately 90°C in a quart heating mantle after an adapted lid [6] was placed on each jar. The bags in Set 4 were heated under a Techina Labs heat lamp set at intensity 5 for 20 min.

All purge times and temperatures are given in Table 1. All samples had vacuum being pulled from a central vacuum system set at 15 in. (38 cm) of mercury.

The charcoal traps were rinsed until a 1-mL eluate of carbon disulfide was obtained. The resulting eluates were run on a Perkin-Elmer Sigma 2000 gas chromatograph equipped with a AS2000 autosampler, a flame-ionization detector (FID) detector, and a 15-m SP-2100 column. One microlitre was injected splitless for 0.3 min and then opened with a 40-mL split flow. A temperature program was used with the following conditions:

Initial temperature = 45°C,  
Initial hold = 4 min,  
Rate = 10/min,  
PTV injector = 250°C,  
Total run time = 27.5 min,  
Final temperature = 230°C,  
Final hold = 5 min,  
Carrier gas = helium,  
DET = 250°C,  
Head pressure = 11 psi,  
Range = 10, and  
Attenuation = 4.

TABLE 1—Purge times and temperatures for samples.

Sample Identification	Purge Time, min	Temperature, °C	Observations
Set 1: Microwave			
Bag 110	2:45	82	two holes burned in Bag 110
Bag 111	2:21	90	all samples in set had hot charcoal tube and water in line
Bag 112	2:26	90	
Temperatures measured at end of second heating			
Set 2: Heating mantle			
Bag 210	12:38	80	all samples in set had hot charcoal tube and water in line
Bag 211	7:38	80	
Bag 212	9:18	80	
Thermometer inserted in bag, heating stopped at 80°C			
Set 3: Heating mantle			
Jar 310	14:30	82	all samples had warm charcoal trap and water in line
Jar 311	7:46	80	
Jar 312	9:46	88	
Thermometer in adapted lid			
Set 4: Heat lamp			
Bag 410	20:00	53	all samples had warm charcoal trap and water in line
Bag 411	20:00	60	
Bag 412	20:00	58	
Temperature measured at the end of 20 min			

Real-time plots were collected on the Perkin-Elmer LCI-100 printer plotter and data stored on the Perkin-Elmer 7500 computer hard disk for later manipulations.

## Results and Discussion

Figure 1 shows the real time plot of a 1- $\mu$ L injection of carbon disulfide containing the standard test mix in a concentration of 10- $\mu$ L/mL. If 100% recovery of the test standard had been obtained, all of the test sample chromatograms would appear as in Fig. 1.

Percent recovery numbers were calculated for each sample based upon five different peak areas. Peaks with retention times of 2.7, 5.2, 8.2, 10.7, and 12.3 min were chosen. These are given in Table 2. Averaged recovery numbers for each set are given in Table 3. The real-time plots for the samples are shown in Figs. 2 through 13.

As shown in Table 2, the microwave extraction method is the least efficient, especially for the higher boiling components. During microwave heating, two holes were burned in Sample Bag 1 in Set 1, creating airflow across the debris. Samples 2 and 3 did not have airflow from outside the bag. The recovery of accelerant in Fig. 2 is noticeably greater than in Figs. 3 and 4. The difference in airflow may account for the differences in efficiency. No area counts were available for Peak Number 5 in Samples 2 and 3 because the peak area was below the peak area reject number in the computer program. The percent recovery of Peak 5 in Set 1 Sample 1 was 16%. When averaged with Samples 2 and 3, the percent recovery for Set 1 is 5%. Further studies are planned to examine this point.

The highest efficiency of recovery was Set 2 (bags heated in heating mantles), closely followed by Set 3 (jars heated in heating mantles). Set 4 samples (bags heated under a heat lamp) showed good recovery for the first three peaks. However, the efficiency decreased for

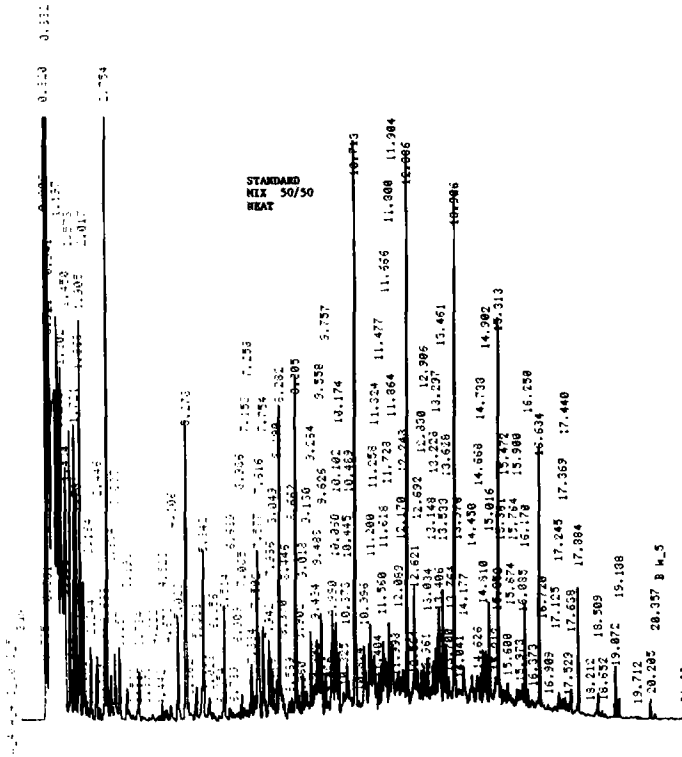


FIG. 1—Chromatogram of a 1-μL injection of the test standard (concentration is 10 μL per 1 mL of carbon disulfide).

TABLE 2—Percent recoveries.<sup>a</sup>

Sample Identification	Peak 1, %	Peak 2, %	Peak 3, %	Peak 4, %	Peak 5, %
Set 1					
Sample 1	35	27	22	21	16
Sample 2	27	28	14	6	0
Sample 3	38	39	20	26	0
Set 2					
Sample 1	28	36	27	33	29
Sample 2	48	62	40	21	18
Sample 3	42	59	48	30	21
Set 3					
Sample 1	42	53	44	32	32
Sample 2	20	27	23	18	18
Sample 3	36	42	38	33	29
Set 4					
Sample 1	28	43	40	24	15
Sample 2	37	51	35	15	8
Sample 3	25	39	35	16	15

<sup>a</sup>  $\frac{\text{Sample area count}}{\text{Standard area count}} \times 100 = \text{Percent recovery of peak.}$

Area counts from Fig. 1 were used for standard area counts.

TABLE 3—Averaged percent recoveries.

Peak No. (min)	Set 1, %	Set 2, %	Set 3, %	Set 4, %
Peak 1 (2.7)	33	39	33	30
Peak 2 (5.2)	31	52	41	44
Peak 3 (8.2)	19	38	35	37
Peak 4 (10.7)	11	28	28	18
Peak 5 (12.3)	5	23	26	13

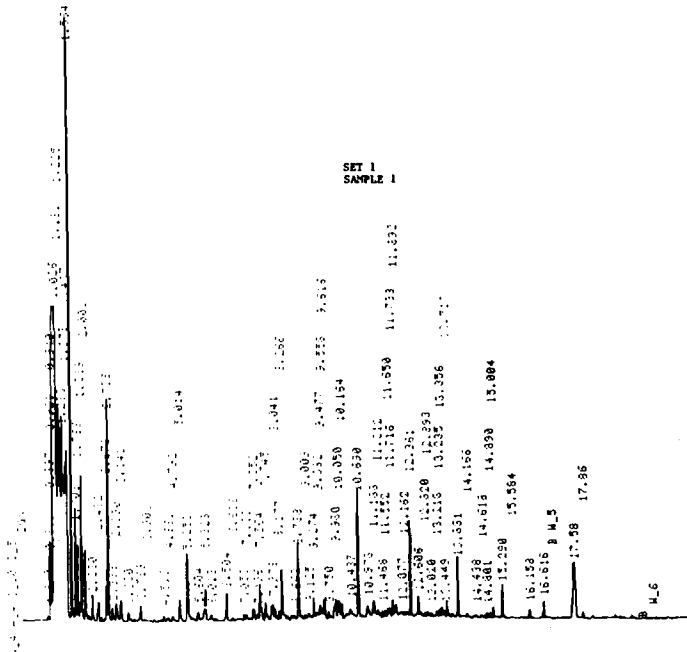


FIG. 2—Chromatogram of Sample 1 bag heated in microwave.

recovery of Peaks 4 and 5. This would be expected since the samples in Sets 2 and 3 were heated to 90°C, and the samples in Set 4 were heated to only 60°C.

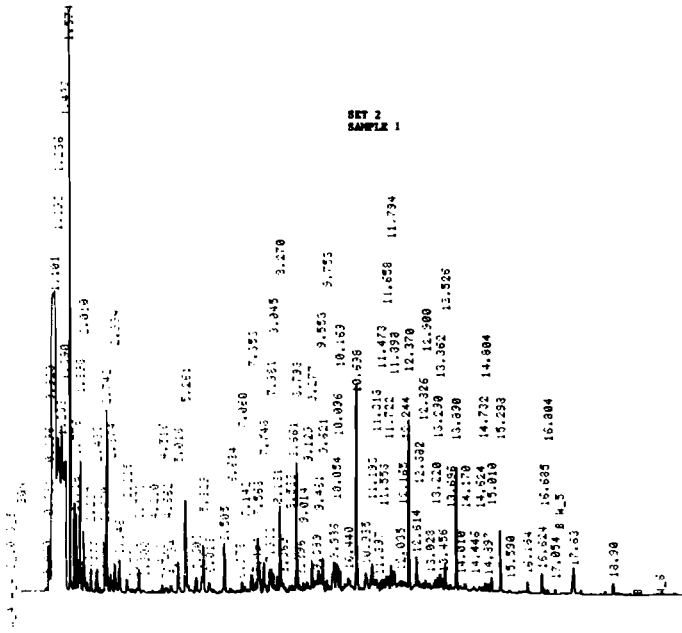
In developing a successful microwave extraction method, we have compiled the following information:

1. The bag must puff to be able to recover heavy petroleum distillates. This indicates a temperature approximately of 90°C.
2. Water in the Tygon tubing does not appear to have an adverse effect on recovery of accelerants.
3. Steam being pulled through the charcoal tube does not cause any appreciable loss of trapped accelerants.

**Conclusion**

Across the country there are different collection, storage, and extraction preferences for arson analysis. The majority of laboratories use only jars or cans or both for sample storage.





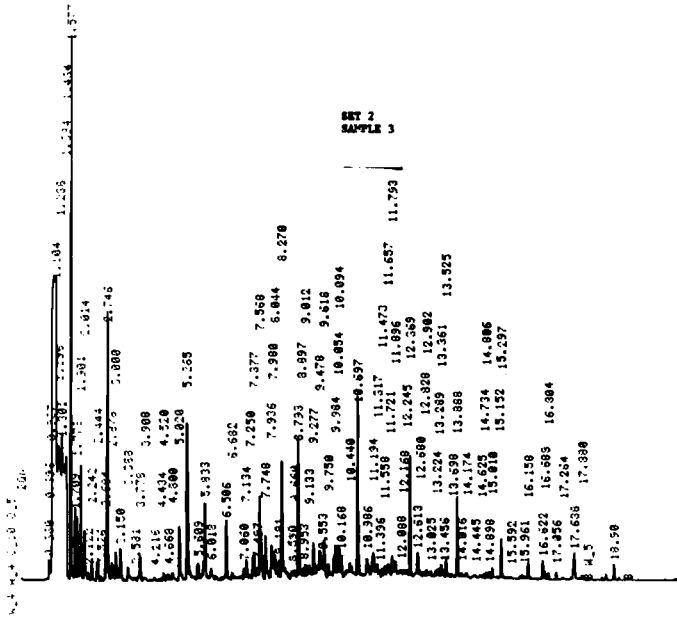


FIG. 7—Chromatogram of Sample 3 bag heated in heating mantle.

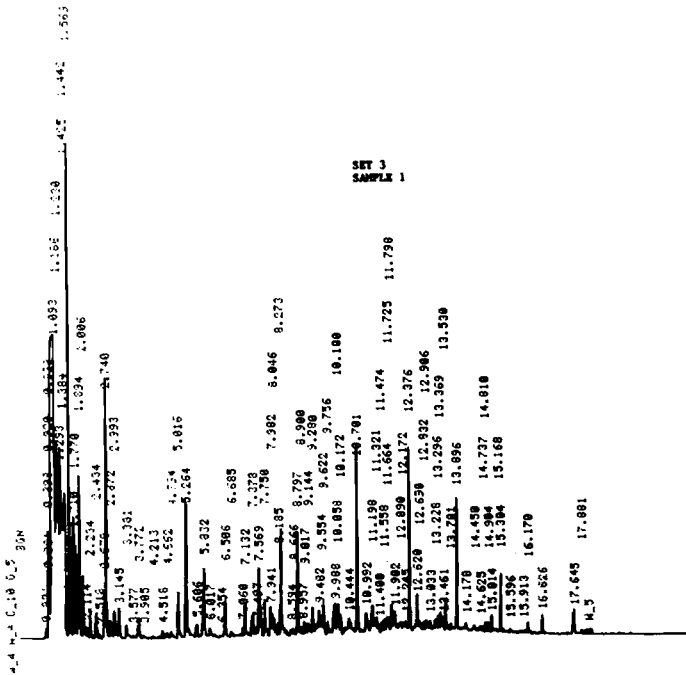


FIG. 8—Chromatogram of Sample 1 jar heated in heating mantle.



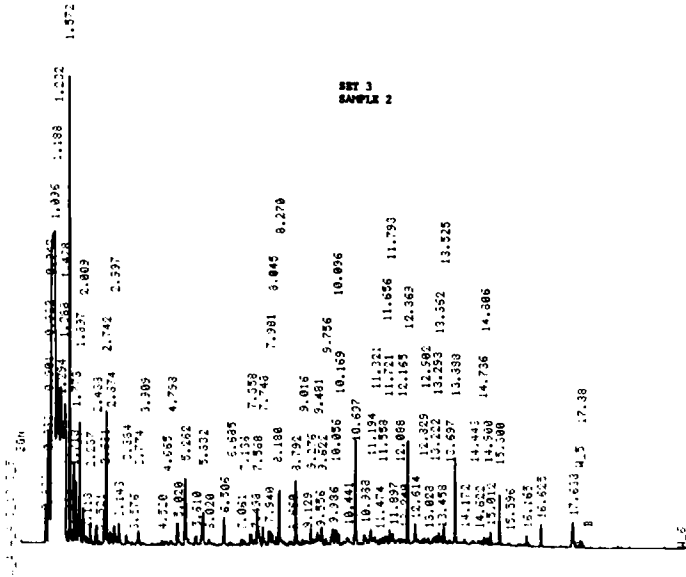


FIG. 9—Chromatogram of Sample 2 jar heated in heating mantle.

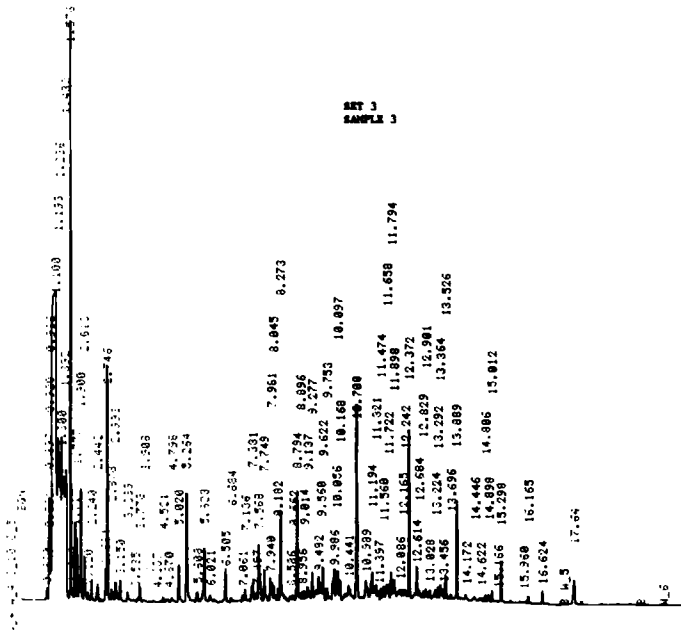


FIG. 10—Chromatogram of Sample 3 jar heated in heating mantle.

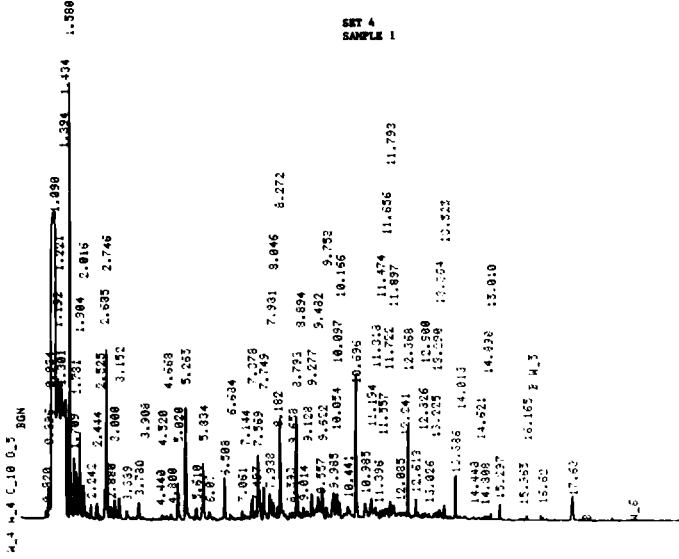
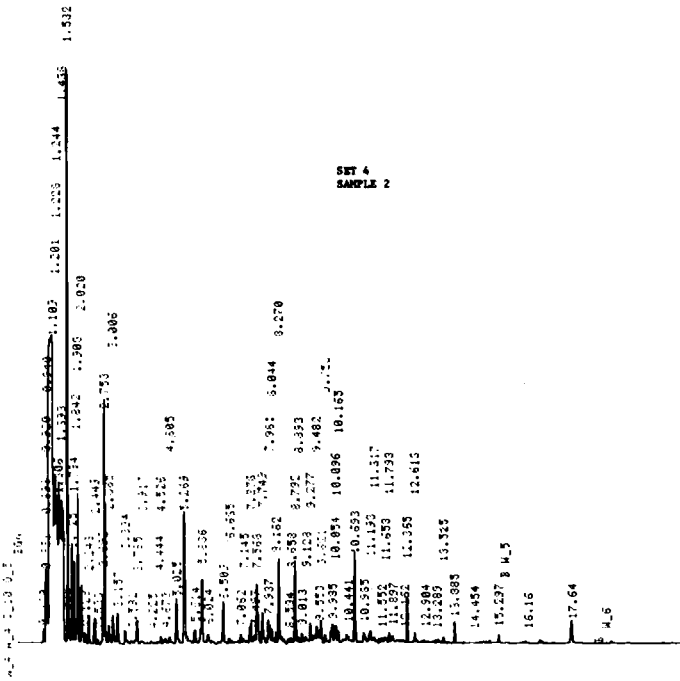


FIG. 11—Chromatogram of Sample 1 bag heated under heat lamp.



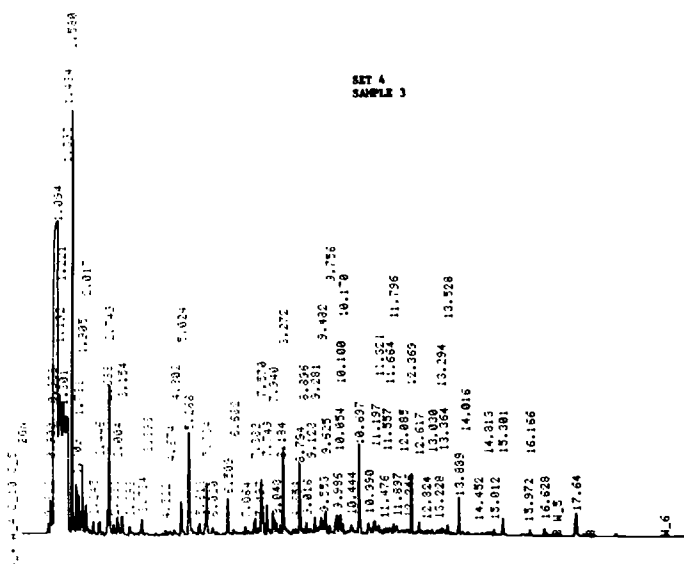


FIG. 13—Chromatogram of Sample 3 bag heated under heat lamp.

With the introduction of polyethylene lined polyester bags, some laboratories are now using bags on a routine basis in their arson cases. In our experience working with cans, jars, and bags, we have found that the bags are more of a convenience for field investigators than laboratory personnel.

Since we can process cans and jars in sets of six as quickly as we can run six microwave samples, there is no time saved. Some of the advantages of the bags are that

- there are no lids to adapt and clean up after use,
- field investigators can carry large quantities in their automobiles in a small amount of space,
- the bags are less bulky for long-term storage, and
- bags may be heated by a variety of methods; therefore purchase of a microwave oven is not mandatory.

Some of the disadvantages of heat seal bags are that

- bags must be screened for metal if heated in a microwave,
- bags must be checked for proper sealing to ensure the integrity of the sample,
- bags may be punctured by sharp objects in debris,
- holes may be burned in the bag if debris ignites when heated in a microwave, and
- holes may be melted through the bag if the heat lamp intensity is too high.

This laboratory will accept evidence properly packaged in polyester bags, jars, or cans. In our opinion, the heat sealed bags have as many disadvantages as advantages and are not more desirable than cans or jars. Although we realize that laboratories have different packaging preferences, by using one of the heating methods described a forensic arson laboratory should be able to successfully process polyester bags.

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Address requests for reprints or additional information to  
Connie I. Gray  
Gray Laboratories  
P.O. Box 1195  
Irmo, SC 29063