

## ENGINEERING AND GINNING

### Laboratory Detection of Plastics in Seedcotton with Ion Mobility Spectrometry

P. A. Funk\*, G. A. Eiceman, C. R. White, and W. White

#### ABSTRACT

**The US cotton industry wants to increase its market share and value throughout the world by supplying pure cotton. Removing contamination requires developing a means to detect plastics in seedcotton. This study was conducted to determine if ion mobility spectrometry (IMS) could be used to find small amounts of plastic in a matrix of heated seedcotton. Commercial IMS analyzers equipped with membrane inlets sampled air displaced from heated flasks containing seedcotton and five common plastic contaminants: bale twine, new and weathered polypropylene tarp, polyethylene film, and plastic film shopping bags. In the first of two sets of experiments the temperature was varied from 85 to 115 °C to determine its influence on vapor emissions; in the second, air samples were taken from seedcotton containing various amounts of each plastic. Vapors from plastics associated with cotton contamination were detected with IMS in negative polarity; different plastics were distinctive. Vapor molecules and product ions from plastics and seedcotton both increased with temperature. Seedcotton vapors spectra coincided with plastics vapors spectra but had different slope characteristics at specific drift times. Digital signal processing may be used to detect plastic contamination, triggering an alarm or control action. Spectra from new and old polypropylene tarp were nearly identical suggesting detection will be possible after weathering. IMS detection of plastics vapors in seedcotton works in the laboratory. The next step is developing IMS systems for commercial cotton gins.**

**D**amage claims exceeding \$5,000,000 annually have been filed against US spinning mills when contaminants have spoiled the retail value of finished

goods (National Cotton Council, 1997). The majority of contamination objects that end up in yarn come from plastic in the field. For example: Dale Fite, vice president of fiber operations, Harriet & Henderson Yarns stated “The most frequent contaminant we’ve found in eastern cotton is Wal-Mart bags; those blue plastic bags.... Careless littering is putting a tremendous amount of contamination in cotton fields. This is creating a problem for farmers, for yarn spinners and for our customers (Hudson, 2000).

Cotton’s hollow fiber and cellulose chemistry facilitate wetting and staining by various dyes (reactive, sulfur, vat, and naphthol). By contrast, only disperse dyes adhere to solid plastic fibers (Smith and Cothren, 1999). Because plastic fiber contaminants either do not accept cotton dyes or already have a contrasting color, they cause blemishes in finished goods. Plastic contaminants persist through each step of textile production because it is difficult mechanically to separate similar plastic and cotton fibers and because human inspection is cost prohibitive and impractical in modern high-volume automated ginning and textile manufacturing operations. Thus, these defects are detected only at the end of the manufacturing process after all value-added operations are complete.

Despite considerable effort expended in policing cotton fields before harvest, some plastic still ends up in seedcotton. Detecting and removing these contaminants needs to be done at the point they enter the cotton gin, before they are reduced in size and dispersed through large amounts of fiber by gin machinery. Presently there is no system to detect plastic contamination in raw seedcotton at the gin.

This research was conducted to provide the US ginning industry with technology to assure globally competitive lint quality. The immediate challenge is detecting the presence of plastic contamination in raw seedcotton. Because plastic materials emit volatile organic chemicals (VOCs) when heated, the direction chosen was to adapt or develop vapor sensors to detect plastic in seedcotton. Vapor sensors have the advantage over optical sensors because they can sense plastic material beneath the surface of a volume of seedcotton.

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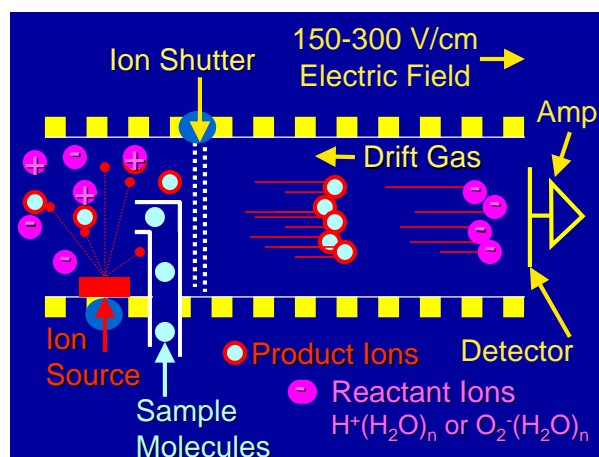
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The decomposition kinetics of polyethylene and polypropylene heated from 200 to 450 °C at ambient pressure were described; the activation energy was ~200 kJ mol<sup>-1</sup> (Westerhout et al., 1997). The VOCs from thermal decomposition of polypropylene were hydrocarbons with carbon numbers from 1 to 7, principally propylene (C<sub>3</sub>H<sub>6</sub>), pentanes (C<sub>5</sub>H<sub>12</sub>), and hexanes (C<sub>6</sub>H<sub>14</sub>) (Hedrick and Chuang, 1998).

In a first exploratory study (Eiceman et al., 2002), the specific VOCs emitted by polypropylene-based tarp and seedcotton at 100 and 150 °C in air and helium were determined using gas chromatography–mass spectrometry (5890A gas chromatograph, 5971A mass selective detector (Hewlett-Packard, Palo Alto, CA) and 30 m (0.25 mm diameter) capillary column, 50% phenyl, 50% methypolysiloxane d<sub>f</sub> 0.25 μm (Restek, Bellefonte, PA). The VOCs emitted during polypropylene heating were primarily alkanes and alkenes with carbon numbers from 14 to 19. Hydrocarbons positively identified were: tetradecane (C<sub>14</sub>H<sub>30</sub>), hexadecane (C<sub>16</sub>H<sub>34</sub>), hexadecane (C<sub>16</sub>H<sub>32</sub>), heptadecane (C<sub>17</sub>H<sub>36</sub>), octadecane (C<sub>18</sub>H<sub>38</sub>), octadecene (C<sub>18</sub>H<sub>36</sub>), nonadecane (C<sub>19</sub>H<sub>40</sub>), and two unidentified aromatic hydrocarbons, all emitting from 0.7 to 9.3 μg g<sup>-1</sup>. These hydrocarbons appeared to arise both from simple thermal desorption and from oxidative decomposition. The VOCs emitted during heating of seedcotton were eicosane (C<sub>20</sub>H<sub>42</sub>), heneicosane (C<sub>21</sub>H<sub>44</sub>), and four unidentified hydrocarbons, all emitting from 0.21 to 0.31 μg g<sup>-1</sup>. Thus, polypropylene VOCs differed from seedcotton VOCs and were formed in quantities typically an order of magnitude greater, suggesting that a vapor sensor approach is feasible. A vapor sensor meeting the requirements of speed, reliability, low limits of detection, and low cost is the ion mobility spectrometer (IMS).

Ion mobility spectrometry has been developed over the last 35 years to detect trace amounts of chemicals in air (Eiceman and Stone, 2004). IMS trace analytical detectors using established principles of ion mobility in differential electrical fields have proven themselves in life-critical applications such as screening passengers' baggage for explosives at airports and detecting warfare chemicals on battlefields (Eiceman and Karpas, 2005). IMS variants also have been used to detect specific insect pheromones (Eiceman et al., 2001) and to discriminate types of smoke from various combustibles found in a cotton warehouse (Eiceman et al., 2003).

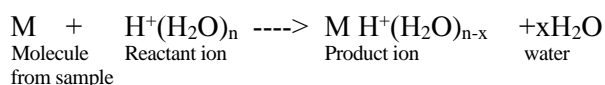
Several IMS configurations exist; a typical analyzer draws sample air into the instrument where it is mixed with gaseous ions (formed by exposing analyzer internal atmosphere to beta particles, photons, or corona discharge, Fig. 1). Reactions between vapors and ions result in product ions that are associated with the chemical composition of the sample (Equation 1).



**Figure 1.** Basic operating principles of a typical IMS:

**Ionization of water vapor forms reactant ions that react with sample molecules to form product ions. Ion clusters enter the drift tube through the shutter and move toward the detector plate under the influence of an electric field. Ions are neutralized when they exchange electrons with the detector and are then swept from the drift tube by clean dry gas. Detector plate current is amplified and plotted against drift time (elapsed from shutter's opening) resulting in a spectra characteristic of ion size and shape (drift time or x-axis) and ion abundance (voltage or y-axis).**

**Equation 1:** Ionization chemistry based upon attachment to protons

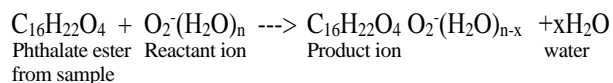


Product ions are introduced into an electric field when an electric shutter opens (typically for about 2 ms) and then characterized on the basis of their drift speed. Electrons are exchanged when reactant and product ions collide with a detector plate at the far end of the drift tube. An amplifier converts the resulting electric current into a voltage representing ion abundance. This is plotted against the time elapsed between the shutter's opening and detection to form a mobility spectrum. The mobility spectrum characterizes a substance based on the terminal velocity in a gas (a function of the size and shape) of its product ion. A

spectrum can be completed in 10 to 20 ms. Several spectra are accumulated for digital signal processing. The total time to make a measurement is 1 s or less. Comparison to a library of spectra from known substances means chemical “fingerprints” can be identified even when sample molecule concentrations are as low as 100 to 10 parts per billion (ppb) (Eiceman and Karpas, 2005). Both trial analyzers used in these tests used these basic operating principles.

Unfortunately, the vapors emitted from heated plastics, principally hydrocarbons such as alkanes and alkenes, exhibit only weak associations to gas phase protons. Thus further development of this method of contamination detection using positive polarity (reactant ion  $H^+(H_2O)_n$  accelerated in a field of decreasing voltage, +1400 to 0 VDC) did not appear feasible. However, plastics generally contain phthalate esters as plasticizers—substances known to be detectable in negative polarity (reactant ion  $O_2^-(H_2O)_n$  accelerated in a field of increasing voltage, -1400 to 0 VDC). Typical negative ion gas chemistry is shown for dibutylphthalate in Equation 2.

**Equation 2:** Ionization chemistry for dibutylphthalate



Dibutylphthalate and similar phthalate esters yielded distinctive mobility spectra with limits of detection in the picogram range (Poziomek and Eiceman, 1992). Because reactions in negative ion polarity in mobility spectrometers are seen as selective compared to the ionization chemistry in positive polarity, the importance of impurities in cotton or ambient atmospheres will be minimized, favoring the detection of trace levels of plastic in seedcotton. Mindful that phthalate esters are widely distributed in the environment through the widespread use of plastics during the past half century, and concerned that some level of contamination might be found in seedcotton from low-level widespread atmospheric pollution (Poziomek and Eiceman, 1992), care was taken to use typical seedcotton handled in a normal manner as a control.

There were three objectives for this study: determining if mobility spectra derived from vapors of plastic are sufficiently different from those of seedcotton in negative polarity; determining if trace levels of plastics can be detected with an IMS analyzer in the presence of a background of vapor emission from

heated seedcotton; and determining the quantitative limits of detection of plastics in seedcotton.

## MATERIALS AND METHODS

Two hand-held IMS analyzers were used in these trials: a Chemical Agent Monitor (CAM) (Graseby Dynamics, now Smiths Detection, Watford, UK) and a SABRE 2000 (Barringer Research, now Smiths Detection, Princeton, NJ). These instruments had common design features including polymethylsilicone membrane inlets, recirculated internal gas atmospheres, 10 mCi  $^{63}Ni$  foil ionization sources, electric ion shutters, and Faraday plate detectors (Eiceman and Karpas, 2005). Both instrument’s drift tubes were 12 mm in diameter by 100 mm long with an electric field strength of approximately  $200 \text{ V cm}^{-1}$ . Significant differences existed between them as well. The CAM drift tube functioned at ambient temperature, whereas the SABRE 2000 maintained a drift tube temperature of  $100 \text{ }^\circ\text{C}$ . The SABRE 2000 also came with an internal calibrant, the CAM used an external standard. The units were used as configured by their manufacturers except that the CAM reagent chemical reservoir was removed. As standard field analyzers, experimental parameters were preset at the factory. These instruments were designed for the detection of chemical warfare agents and explosives and could not readily be optimized for the detection of vapors from plastics. However, vapor yield through the membrane inlet, response stability over an extended period of time, and the quality of the spectral resolution were suitable for these trials. Limits of detection were in the ppb range for chemical standards in air. Quantitative precision was within 15% with replicated measurements. Spectral peaks could be separated for reactant ions, seedcotton, and plastics vapors’ product ions.

Studies on response to cotton and plastics were made using both hand-held IMS analyzers for two purposes. The sample temperature best suited to detect plastic was determined using the CAM with samples at 85, 100, or  $115 \text{ }^\circ\text{C}$ . The amount of plastic detectable in a cotton matrix was determined using the SABRE 2000 with samples at  $115 \text{ }^\circ\text{C}$ . Plastic samples weighing 0.02, 0.10, 0.20, and 0.30 g were added to 2.0 g of seedcotton corresponding to 0, 1, 5, 10, and 15% (by mass) of plastic in cotton.

In each experiment, sample handling was the same. Samples were placed in a 500 ml Pyrex round flask with a heating mantel (Glasscol, Terre Haute, IN) maintaining the desired temperature. A Fathom

Technologies (Round Rock, TX) GR116 mass flow controller metered 8.33 ml/s clean dry air from an Aadco 737 pure air generator (Cleves, OH) through a glass tube preheating arrangement (warming it to 85, 100, or 115 °C) and into the flask (Fig. 2a). Though the dewpoint of the air produced from a 737-series pure air generator will not exceed -51 °C (-60 °F), enough water remained to form reactant ions. Next, flow from the flask was directed into the inlet of the IMS with space between the flask and inlet to allow excess flow to vent to the atmosphere. Measurements of negative controls detected no interferences. Sampling method, hardware, and air quality were deemed suitable.

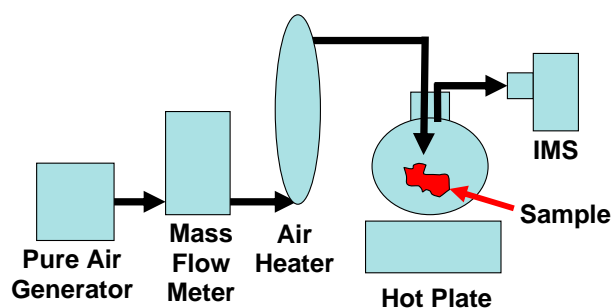


Figure 2a. Gas flow schematic.

During measurements, mobility spectra were obtained continuously using a model 6024 National Instruments (Austin, TX) PCMCIA A/D card and laptop computer as shown in Fig. 2b. Parameters for digital signal averaging were: sampling rate, 33 kHz; points per spectrum, 1000; and number of scans per spectrum, 10. Spectra were collected every 1.5 s. Other amplifier and signal processing electronics were not under user control because instrument operating parameters were preset by the manufacturers.



Figure 2b. IMS sensing vapors from a heated flask containing plastic and cotton. Left to Right: gas mass flow meter, pure air preheater, 500 ml flask on hot plate, and IMS representative of analyzers used in these trials.

Each study was preceded by 20 min operation without sample in the flask to ensure stability in instrument performance and cleanliness of the sample container. After this period, the flask was loaded with seedcotton or with seedcotton and plastic and allowed to equilibrate for 60 s. Ten spectra were then recorded over ~15 s and these were averaged off-line into a single spectrum. Between experiments, the flask was washed with soap and water, baked at 120 °C for a minimum of 4 h, and examined with a negative control (above).

Five plastics were tested for vapor emissions as described above and included polypropylene orange bale twine (Wellington, The Lehigh Group, Macungie, PA), new polypropylene blue tarp (Orgill, Inc, Memphis, TN), old polypropylene tarp (weathered 1 yr, Las Cruces, NM), 6 mil black polyethylene film (Poly-America, Grand Praire, TX), and white plastic film shopping bags (Wal-Mart, Bentonville, AR).

## RESULTS AND DISCUSSION

### Effect of temperature on mobility spectra.

Increases in temperature were expected to affect the amount of background vapors emitted from seedcotton. Mobility spectra from analysis of vapors released by seedcotton at 85, 100, and 115 °C are shown in Fig. 3. The mobility spectrum (from the CAM) for vapors over seedcotton at 85 °C contained a single intense peak, the reactant ion  $(O_2^-(H_2O)_n)$ , at 4.3 ms, with peak height greater than 1.5 V. In nearly all spectragraphs in these trials the vertical scale was enlarged to show low intensity constituents. The reactant ion peak was always present, even if not shown in these plots.

Peaks for seedcotton vapors were observed at drift times between 5 to 8 ms in the mobility spectra. These product ions arose from reactions similar to those given in Equation 2. This minor level of contamination was not substantially increased with an increase in temperature to 100 °C. However, at sample temperature of 115 °C response to vapor emissions increased significantly. Low levels of product ions from 5 to 12 ms appear, presumably through increased vapor pressure of substances in seedcotton.

In an IMS analyzer a fixed amount of reactant ions are available, limited by the kinetics of ion formation in air by beta particles. Increasing product ion formation uses up reactant ions, decreasing the reactant ion peak in this case to below 1.5 V. These findings demonstrate that vapor emissions arise from raw seed-

cotton and IMS analyzers are sensitive to these vapors. However, intensities were low and spectral profiles were not well defined, portending the possibility of observing plastic vapors in this background.

In contrast to the mobility spectra from seedcotton, mobility spectra observed for emissions from plastics show distinctive patterns with well-defined peaks (Figs. 4 through 8). The intensity of product ions increased with temperature for each plastic as expected due to increased vapor pressure of semivolatile organic chemical constituents. The increase in vapor pressure of substances in plastic was associated with increased vapor levels in the headspace over the sample. Again, the reactant-ion peak intensity decreased with increasing temperature because charge was used to form product ions through reactions similar to Equation 2.

The mobility spectra for new polypropylene tarp are shown in Fig. 5. These spectra exhibited several product ion peaks that increased in intensity with temperature. For a sample temperature of 115 °C product ion peaks nearly equaled the reactant ion peak. The sum of the areas under each product ion peak together exceeded that of the reactant ion peak. There was little difference between that of new tarp and that of old tarp (Fig. 6), suggesting that the constituents associ-

ated with vapor emissions will persist in this type of plastic despite weathering and will allow its detection even after 12-m exposure in the field.

Other plastics may have different amounts of semivolatile constituents (assumed here to be plasticizers) and even different plasticizer formulations. These should be reflected as differences in vapor emissions and thus in mobility spectra. This is evident in Figs. 4, 7, and 8 for other plastics associated with cotton contamination. Polypropylene twine showed elevated response for product ions with complex spectral profiles and unresolved peaks between 5 and 10 ms (Fig. 4). Comparable responses were obtained for polyethylene film (Fig. 7) and film shopping bags (Fig. 8). Their profiles were distinctive from 5 to 8 ms.

Quantitative response differed slightly. Because sample amounts can be quantified through reactant ion peak intensity reductions these were compared among samples. Reactant ion peaks for polypropylene (twine and new tarp, Figs. 4 and 5) were below 1.0 V at 115 °C, whereas those for the same mass of polyethylene film and old tarp (Figs. 6 and 7) decreased only to 1.2 V at 115 °C. The film shopping bag (assumed to be polypropylene or polyethylene) showed a similar response but with less intensity (Fig. 8).

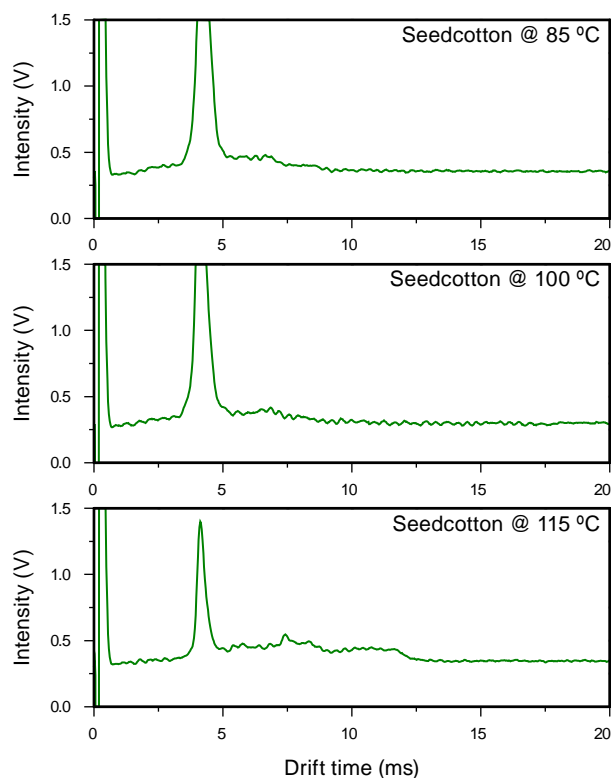


Figure 3. CAM plot for seedcotton at three temperatures.

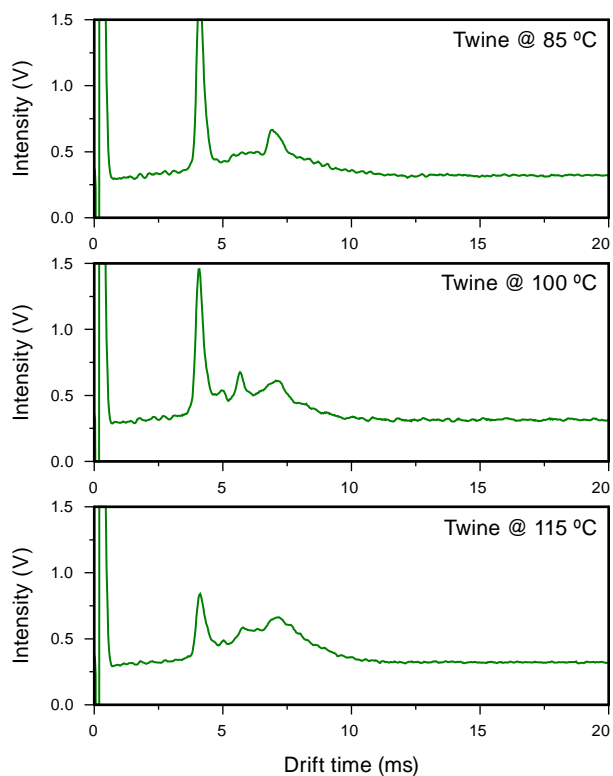


Figure 4. CAM plot for orange plastic bale twine at three temperatures.

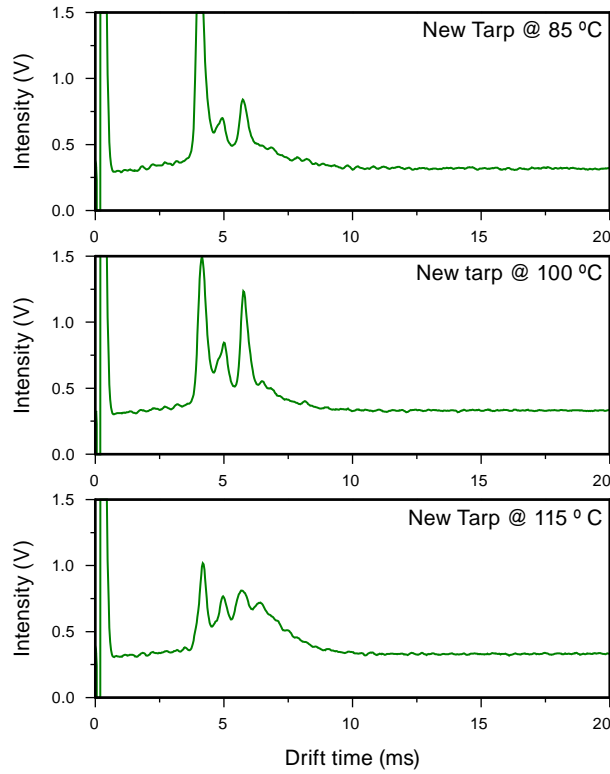


Figure 5. CAM plot for new blue polypropylene tarp at three temperatures.

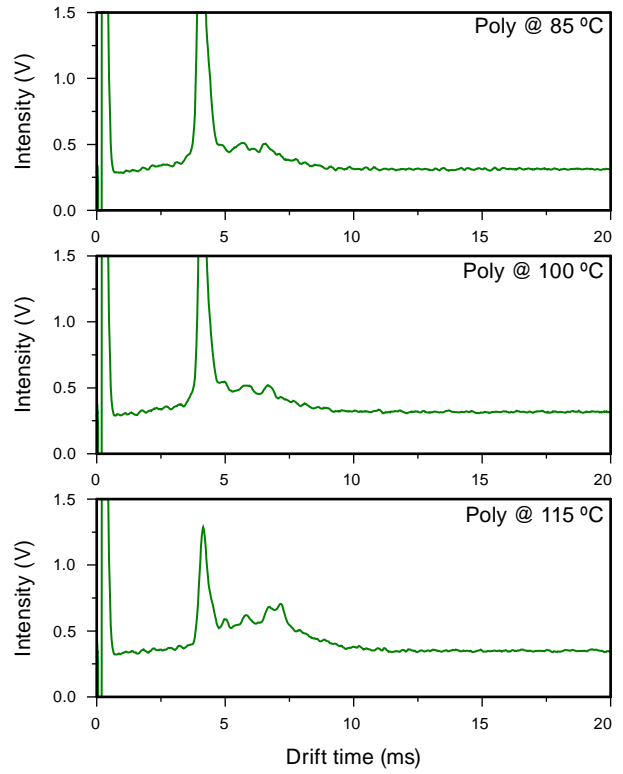


Figure 7. CAM plot for 6 mil black polyethylene film at three temperatures.

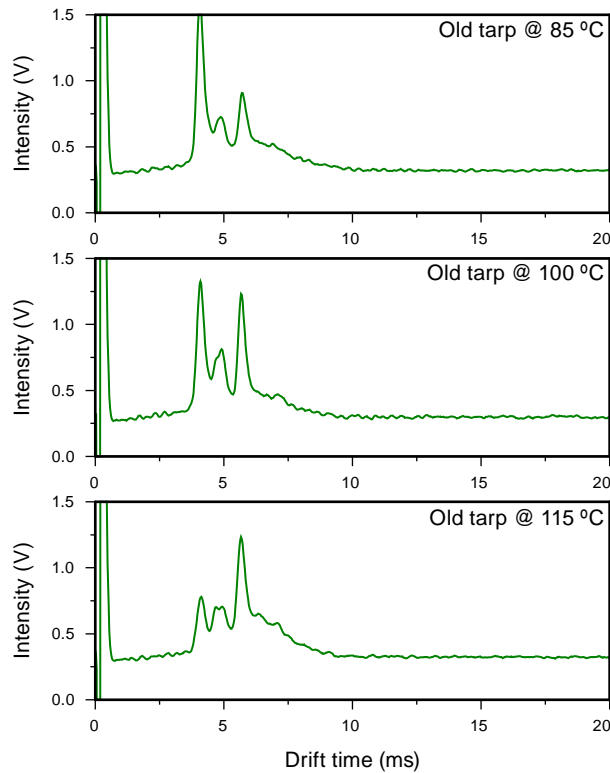


Figure 6. CAM plot for weathered blue polypropylene tarp at three temperatures.

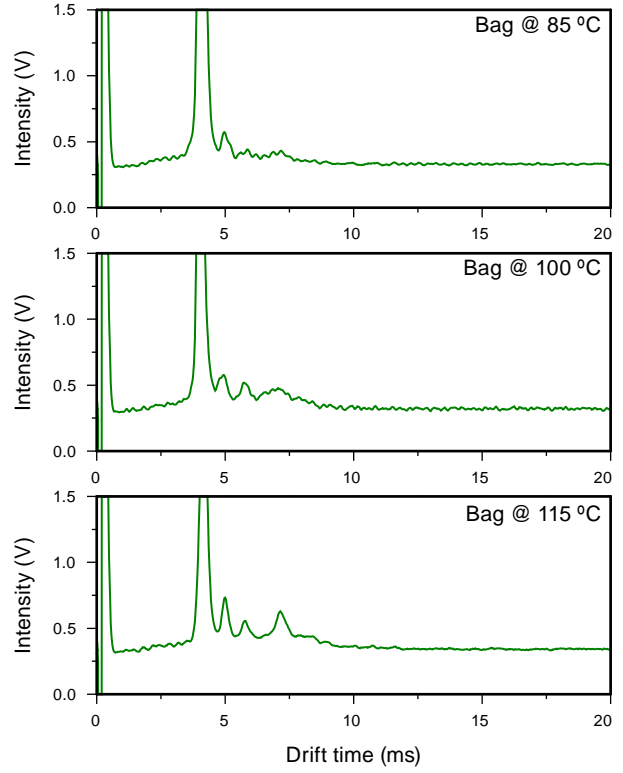


Figure 8. CAM plot for white plastic film shopping bag material at three temperatures.

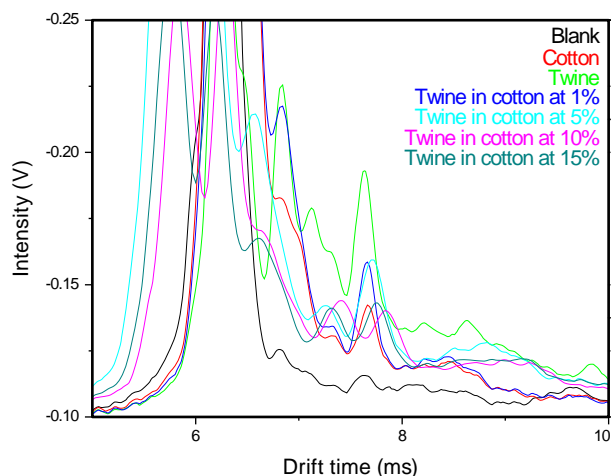
These findings demonstrate that vapors from plastics associated with cotton contamination can be detected with a mobility spectrometer in negative polarity. As expected, vapor levels from different plastics increased with temperature due to increases in VOC vapor pressure. Mobility spectra for different plastics were distinctive. A mobility spectrometer monitoring seedcotton purity should be able not only to detect a plastic but possibly also to identify it.

A complication is that seedcotton released vapors that showed response in the same region of the mobility spectrum as that of plastics. Though the response was lower on a mass basis than that for plastic, it is a potential source of background interference. It is also a potential source of decreased sensitivity as reactant ions consumed in the formation of product ions with seedcotton VOCs are not available to form product ions from plastics VOCs. Finally, VOCs from plastics might be absorbed by cotton fibers, further suppressing plastics response. These concerns can best be addressed through empirical studies where the level of plastic detected in an excessive background of seedcotton is determined in a real-world setting.

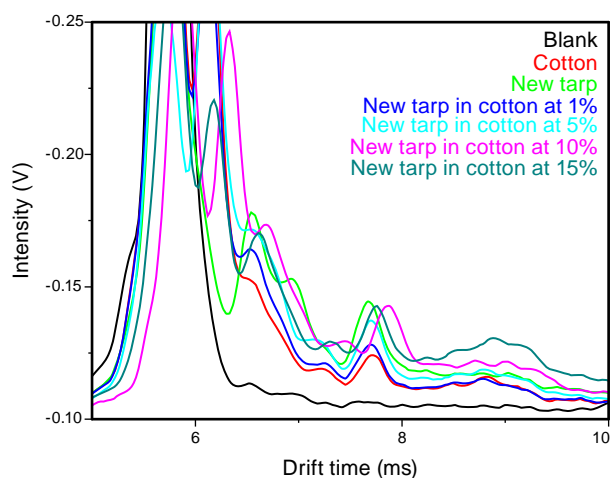
**Mobility spectra from plastic contamination in seedcotton.** Figures 9 through 13 show mobility spectra for plastics at several levels (0, 1, 5, 10, and 15% by mass) in a background of seedcotton. These measurements were made with the SABRE 2000, which used a heated drift tube to minimize condensation of VOC vapors on the inlet, membrane and drift tube surfaces, and later persistence of their product ions. An instrument and method control (Blank) is shown in each figure; these were uniformly free of detectable levels of contamination. When small amounts of plastics were placed in seedcotton, product ions from plastic emissions were observed in the mobility spectra.

Seedcotton alone resulted in emission profiles that appeared in the same region as those from plastic vapors (in Figs. 9 through 13 the red curve, Cotton, is from seedcotton). The appearance of spectral peaks (vapor product ions) at 6.6, 7.7, and 8.9 ms from seedcotton matched those from the polyethylene film (Fig. 12) at this level of scale expansion. The presence of peaks from plastics was attributed to plasticizers (phthalate esters) that could be present in seedcotton due to plasticizers' widespread presence in the environment. Though differences in spectra were seen at levels as low as 5% by mass, the differences were primarily quantitative and not,

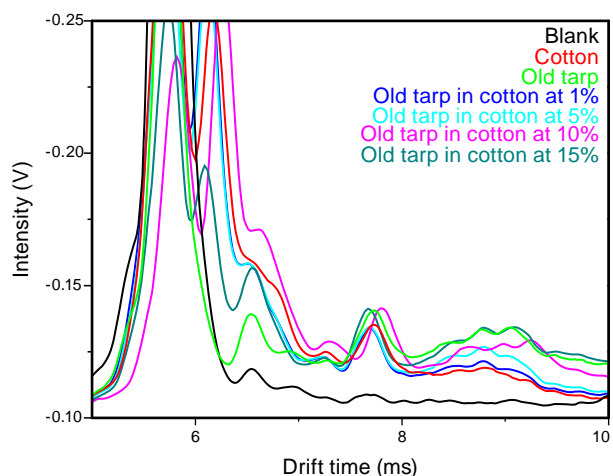
as hoped, distinctive from product ions exhibiting unique characteristic drift times. This was observed for each spectra in Fig. 9.



**Figure 9. SABRE 2000 plots showing increasing amounts of orange plastic bale twine.**



**Figure 10. SABRE 2000 plots showing increasing amounts of new blue polypropylene tarp.**



**Figure 11. SABRE 2000 plots showing increasing amounts of weathered blue polypropylene tarp.**

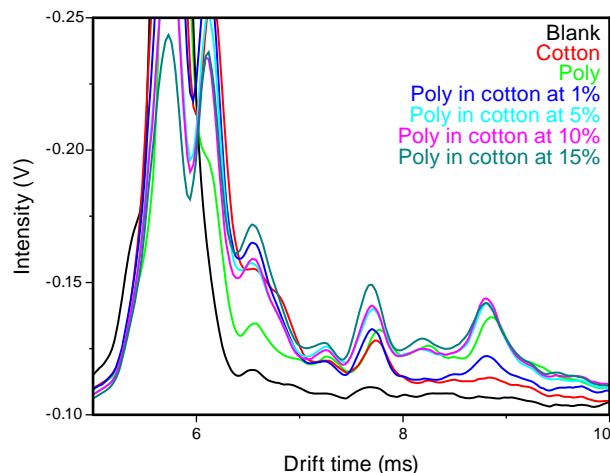


Figure 12. SABRE 2000 (plots showing increasing amounts of 6 mil black polyethylene film.

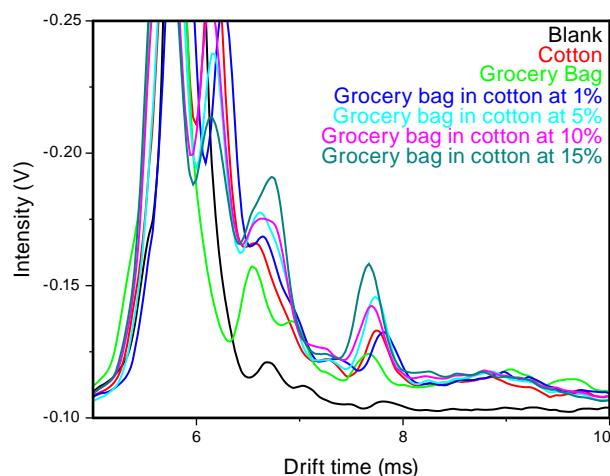


Figure 13. SABRE 2000 (plots showing increasing amounts of white plastic film shopping bag material.

Similar results with minor variations in absolute and relative peak heights were observed for the other plastics (Figs. 10 through 13). These illustrate that with a heated drift tube response to plastics at trace levels were stronger than suggested earlier at higher vapor levels with an unheated analyzer. Although the seedcotton background will present a significant challenge for on-line monitoring, close examination of Figs. 9 through 13 reveal a subtle difference between seedcotton and plastics. The slope of the seedcotton spectra between 6.2 and 6.7 ms is near zero or negative. The slope of all of the plastic spectra between 6.2 and 6.7 ms is positive for a brief span. This unique aspect of the spectra may be exploited by digital signal processing software to detect plastics, thereby triggering an alarm or initiating a control action.

## CONCLUSIONS

Vapor emissions of plastics in air at elevated temperature were detected with hand-held IMS analyzers using response for product ions in the negative polarity. The spectra were distinctive for each type of plastic and were attributed to formulations of plasticizers. Response seen in spectral intensity increased with sample temperatures between 85 to 115 °C, as expected. Increasing temperature resulted in increased vapor pressure of semivolatile constituents in the plastic. Little difference between analyzers was observed apart from improved (reduced memory effects) results with the heated drift tube instrument. Under comparable conditions, the levels of emissions from seedcotton were negligible. However, a goal of this work is the detection of trace amounts of plastic in huge volumes of seedcotton entering a gin. These studies with mixtures of seedcotton and plastics at various mass ratios demonstrated that vapors from plastics could be quantitatively distinguishable to levels of 1% by mass, though absolute values will be governed by sample temperature and the design of the interface between the seedcotton flow and the analyzer. Though plastics were detected at such low levels, efforts to computationally remove interfering responses to seedcotton must be developed. For the moment, sufficient selectivity was observed to justify further instrument development followed by field trials in a commercial cotton gin.

## ACKNOWLEDGMENT

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## DISCLAIMER

Mention of trade names or commercial products in this article is solely for the purpose of providing specific information and does not imply recommendation or endorsement by the U. S. Department of Agriculture.

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