

TECHNICAL NOTE

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The Identification of Isopar H in Vinyl Flooring

ABSTRACT: Vinyl flooring manufacturers use plasticizers to decrease the viscosity and increase the pliability of vinyl. Several ignitable liquid plasticizers used in the manufacture of vinyl flooring were identified and investigated in this study. Twenty-nine collections from five major vinyl manufacturers, a total of 72 samples, were analyzed using passive headspace concentration in accordance with the American Society for Testing and Materials (ASTM E 1412-00) and gas chromatographic-mass spectrometric (GC-MS) analysis as described in ASTM E 1618-01 (1,2). Norpar products and TXIB (2,2,4-trimethyl-1,3-pentanediol diisobutyrate) are ignitable liquids common to the manufacture of vinyl flooring and were identified in all recently obtained samples. Isopar H is an ignitable liquid found in various products such as charcoal starters, copier toners, and some solvents (2). Of the 29 collections analyzed, Isopar H was only identified in Armstrong's Interflex-Traditions pattern.

KEYWORDS: forensic science, fire debris, gas chromatograph-mass spectrometry, vinyl flooring, Norpar, Isopar H, plasticizer, total ion chromatogram

Vinyl flooring, often mistaken as linoleum flooring, is one of the most popular types of flooring chosen by consumers. Vinyl flooring and linoleum flooring are two distinctive products distinguished by their manufacturing processes. Linoleum is composed of all natural products such as limestone, wood powder, and linseed oil from flax seed. The backing of linoleum is made from a natural grass called jute (3).

Vinyl flooring is composed of several different layers, including a wear layer, print layer, foam layer, and a felt-backing layer. The felt backing layer is composed of limestone and clay and held together with a plastic binder (3). Adhesives used by most manufacturers to install vinyl flooring are typically water-soluble with a hazardous materials flammability rating of zero or one (4,5).

Vinyl is composed of approximately 43% ethylene and 57% chlorine (6). In the vinyl manufacturing process, the vinyl resin is hard and brittle and is not considered a usable material (7). With the addition of plasticizers to decrease viscosity, the vinyl resin will become soft, flexible, and ready to use (8). Plasticizers are used in the core layer of vinyl construction. It must be noted that some plasticizers in liquid form are ignitable. However, not all plasticizers used in the manufacture of vinyl flooring are marketed to the general public as ignitable liquid products that may be identified by fire debris analysts as ignitable liquid patterns on GC-MSD chromatograms. Some of the most commonly identified plasticizers in vinyl flooring include TXIB (2,2,4-trimethyl-1,3-pentanediol diisobutyrate) and Norpar products (9,10). Norpars, normal alkane products consisting of straight chain alkanes, are marketed for several different purposes ranging from charcoal starters to industrial cleaning solvents (3,11). Vinyl flooring manufacturers use Norpar products in the range of Norpar 13 to Norpar 15 as plasticizers marketed under different trade names (12). For example, Norpar 14

is marketed by Exxon as Jayflex 214[®]. The purpose of this study is to sample vinyl flooring products and determine if there are any other plasticizers with recognizable ignitable liquid patterns being used in the manufacture of vinyl flooring other than normal alkane products.

Materials and Methods

Instrumental Conditions

All samples were analyzed using an Agilent 6890N Gas Chromatograph connected to an Agilent 5973 Mass Selective Detector. Injections of one microliter were made using an Agilent 7683 Series Autosampler. A 30 m J&W Scientific DB-1 column with an internal diameter of 0.25 mm and a film thickness of 0.25 μm was used. The carrier gas was helium set at a constant flow of 1 mL/min with a split ratio of 50:1 being obtained. The initial oven temperature was held at 50°C for 3 min. The temperature was then ramped to 260°C at a rate of 10°C/min. The final temperature was held at 5 min, resulting in a 28 min run time. The mass spectrometer was operated in the scan mode with a range from 10 to 330 m/z.

Sample Preparation

Vinyl samples were collected from several local flooring dealers. A total of 72 unburned samples from 29 different vinyl collections were tested from five of the major vinyl manufacturers: Domco, Congoleum, Tarkett, Mannington, and Armstrong. Twelve randomly chosen vinyl samples were burned using a propane torch for approximately 10 s. The vinyl samples were cut into approximately 3 by 3 in. pieces and placed in clean, one-quart lined paint cans. One third of a charcoal strip (Albrayco Lab, Inc., Cromwell, CT) was suspended on a wire inside each can. The cans were placed in an oven at 70°C for approximately 16 h in accordance with ASTM E 1412-00 (1). The cans were allowed to cool and the strips were removed and placed in individually labeled vials with Teflon

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coated screw caps. Approximately 250 μL of carbon disulfide were added to the vials and allowed to equilibrate for approximately 30 min. Ninety μL were removed from each vial and placed inside a glass insert confined to an autosampler vial. All samples were analyzed by Gas Chromatography with a Mass Selective Detector (GC-MSD) in accordance with ASTM E 1618-01 (2).

Results

Figure 1 is an ignitable liquid plasticizer sold by both Exxon and Eastman as TXIB (2,2,4-trimethyl-1,3-pentanediol diisobutyrate). Figure 2 is an ignitable liquid product identified as Isopar H, marketed by Exxon as "Odorless Mineral Spirits." Figure 3 illustrates six selected ion profiles of Exxon's "Odorless Mineral Spirits."

Figure 4 shows the total ion chromatogram of Domco Customflor Legend 64273 vinyl flooring. In this sample, Norpar 14 and TXIB were identified. Early eluting non-aromatic compounds consisting of cycloalkanes, alkenes, and alcohols were identified before C12 and late eluting aromatic compounds consisting of alkylbenzenes were noted after C14; however, this study was only concerned with ignitable liquid patterns that may be misleading to a fire debris analyst. Figure 5 displays two total ion chromatograms of Domco Customflor Concourse 65311. The upper chromatogram is an unburned sample and the bottom chromatogram is a burned sample. Norpar 14 and TXIB were identified in both the unburned and the burned samples.

Figure 6 represents vinyl flooring manufactured by Mannington and marketed as Beautiflaire series 10009. TXIB was identified with other benzene compounds.

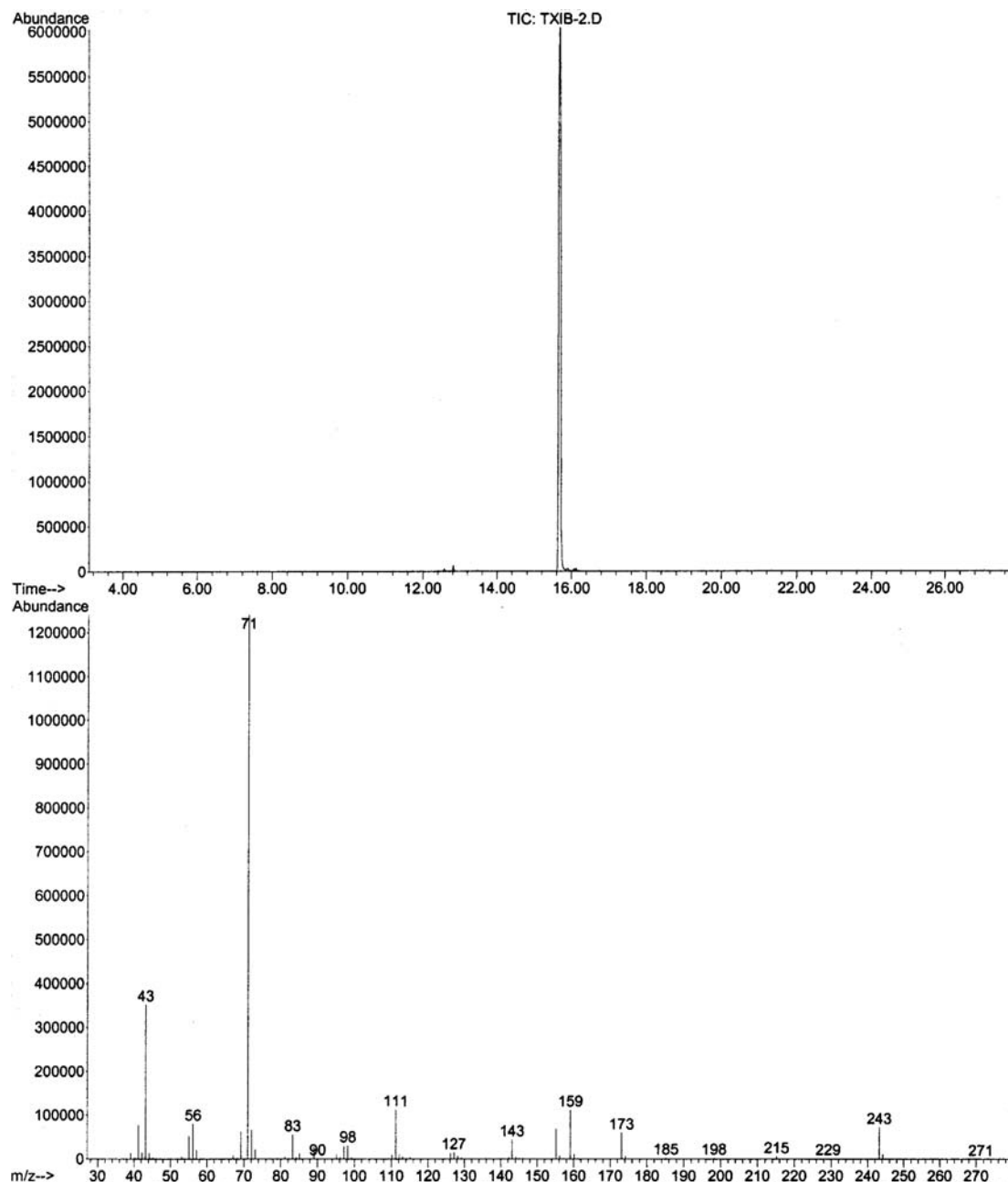


FIG. 1—Total ion chromatogram and mass spectrum of TXIB (2,2,4-trimethyl-1,3-pentanediol diisobutyrate).

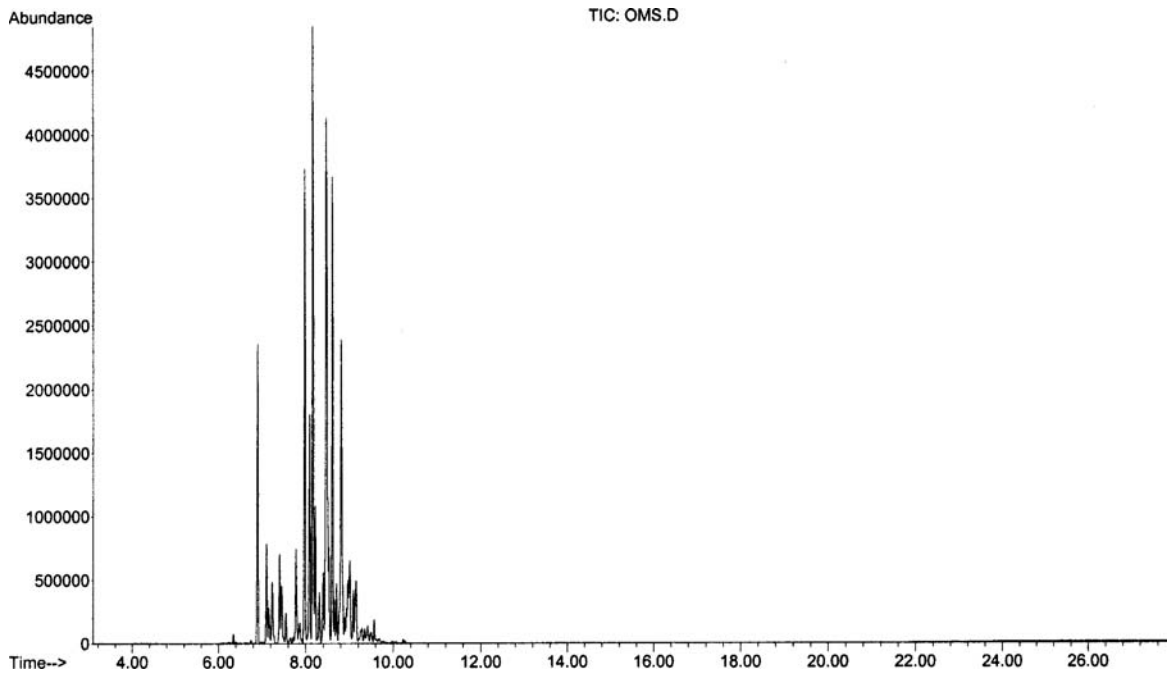


FIG. 2—Total ion chromatogram of Exxon's Odorless Mineral Spirits also marketed as an Isopar H product.

TABLE 1—Summary of the ignitable liquid patterns identified in the five major vinyl flooring manufacturers.

Manufacturer	Norpar	TXIB	Isopar H
Domco ¹	X	X	
Congoleum ²	X	X	
Tarkett ³	X	X	
Mannington ⁴	X ⁶	X	
Armstrong ⁵	X	X	X ⁷

X = Ignitable liquid present in samples.

Collections analyzed:

¹ Customflor, Rustic.

² Concept, Highlight, Ultima.

³ Ambiance, Contours, Preference Plus, Signature, Style Brite, 11000 Series, 73000 Series.

⁴ Aurora Bronze Series, Beautiflaire, Duracon, Duracon II, Marquis, Naturals, Style & Pace, Vega II Bronze Series.

⁵ Ashton, Canyon Creek, Imperial Accotone, Initiator, Rhythms, Signia, Solarian, Themes, Traditions.

⁶ Norpar not identified in Beautiflaire, Duracon, Duracon II, Marquis collections.

⁷ Isopar H only identified in Armstrong's Interflex-Traditions collection.

Figure 7 represents vinyl flooring manufactured by Armstrong and marketed as an Ashton series 46702. Norpar 14 and TXIB were identified in the total ion chromatogram.

Figure 8 represents vinyl flooring manufactured by Armstrong and marketed as Interflex-Traditions series 60221. Isopar H, Norpar 13, and TXIB were all identified in the total ion chromatogram. Figure 9 displays six selected ion profiles of Armstrong's Interflex-Traditions Collection series 60221.

Figure 10 displays two total ion chromatograms of Armstrong's Interflex-Traditions Marble Dot 60103. The upper chromatogram is an unburned sample and the bottom chromatogram is a burned sample.

Table 1 represents a summary of the ignitable liquid patterns identified in the five major vinyl flooring manufacturers' products.

All collections contained TXIB, and all but four collections contained a Norpar product in significant concentrations in the total ion chromatogram. In the vinyl collections tested, Isopar H was only detected in Armstrong's vinyl flooring. Furthermore, Armstrong's Interflex-Traditions collection was the only series among nine other Armstrong collections tested that contained Isopar H. After Isopar H was identified in the Interflex-Traditions flooring, nineteen other samples of Interflex-Traditions flooring were obtained. All nineteen samples contained significant levels of Isopar H in the total ion chromatogram.

Discussion

Norpar products have been reported in previous studies as components added during the vinyl manufacturing process (11). Vinyl manufacturers report using a range of Norpar products from Norpar 13 to Norpar 15 as plasticizers to lower the viscosity of the vinyl and make it more pliable. This study has shown that ignitable liquid products other than Norpars are being used by the vinyl flooring industry as plasticizers.

During production, the vinyl is heated in ovens in excess of 300°C. It was once thought that the ignitable liquid plasticizers were being baked off during the heating process. This study illustrates that not only are Norpar products being used as plasticizers, but also Isopar H and TXIB products. These three plasticizers can be identified in significant quantities as seen in the Armstrong's Interflex-Traditions collection (Fig. 8).

Under the conditions as described in sample preparation, burning the vinyl samples had no significant change in the total ion chromatogram. The total ion chromatograms of the unburned and burned samples were very similar except for the addition of a benzyl chloride peak at retention time 7.274 min. in the burned samples of Domco, Congoleum, Mannington and Tarkett (Fig. 5). Ion 91 was used to identify the presence of the benzyl chloride. The burned samples from the Armstrong's collections contained trace amounts of benzyl chloride that are not evident in the total

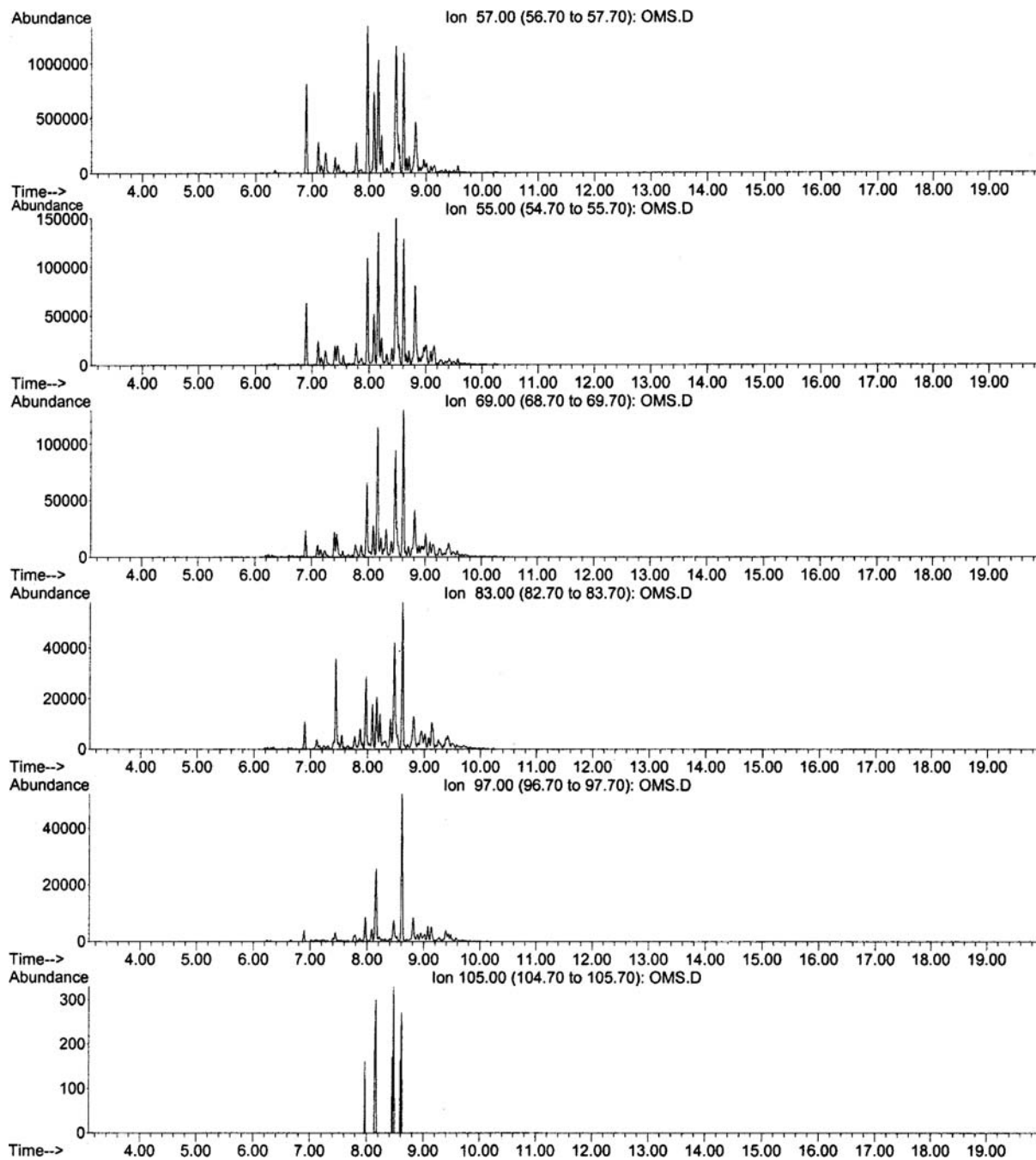


FIG. 3—Six selected ion profiles of Exxon's Odorless Mineral Spirits also marketed as an Isopar H product.

ion chromatogram, and can be identified by its retention time and extracting ion 91 from the total ion chromatogram (Fig. 10). The abundance of the Norpar product, TXIB, and Isopar H remained relatively the same in the unburned and burned samples. Burning the vinyl samples did not make it difficult to identify the ignitable liquid plasticizer used (Fig. 9).

Of the 72 vinyl samples analyzed, 68 contained significant levels of either a Norpar 13 or Norpar 14 product as seen in Figs. 4, 7 and 8. Mannington's older collections of Marquis series, Beautiflaire series, Duracon series, and Duracon II series contained trace amounts of hydrocarbons in the range of dodecane (C12) to hexadecane (C16). The hydrocarbons identified in Fig. 6 of Mannington's

Beautiflaire Series 10009 illustrates the total ion chromatogram from the above products contained significantly less amounts of n-alkanes as seen in other collection series. This study was unable to determine the origin of the normal alkanes of the older Mannington samples. These vinyl samples may contain trace amounts of a Norpar 14 or a plasticizer containing normal alkanes in the range of C12–C16. These particular Mannington samples were discontinued in the early 1990's, but were used in this study to represent older homes, trailers, and businesses that may still have this type of vinyl flooring.

As an ignitable liquid, Isopar H is marketed as charcoal starters, paint thinners, copier toners, and solvents (11). Other isoparaffin

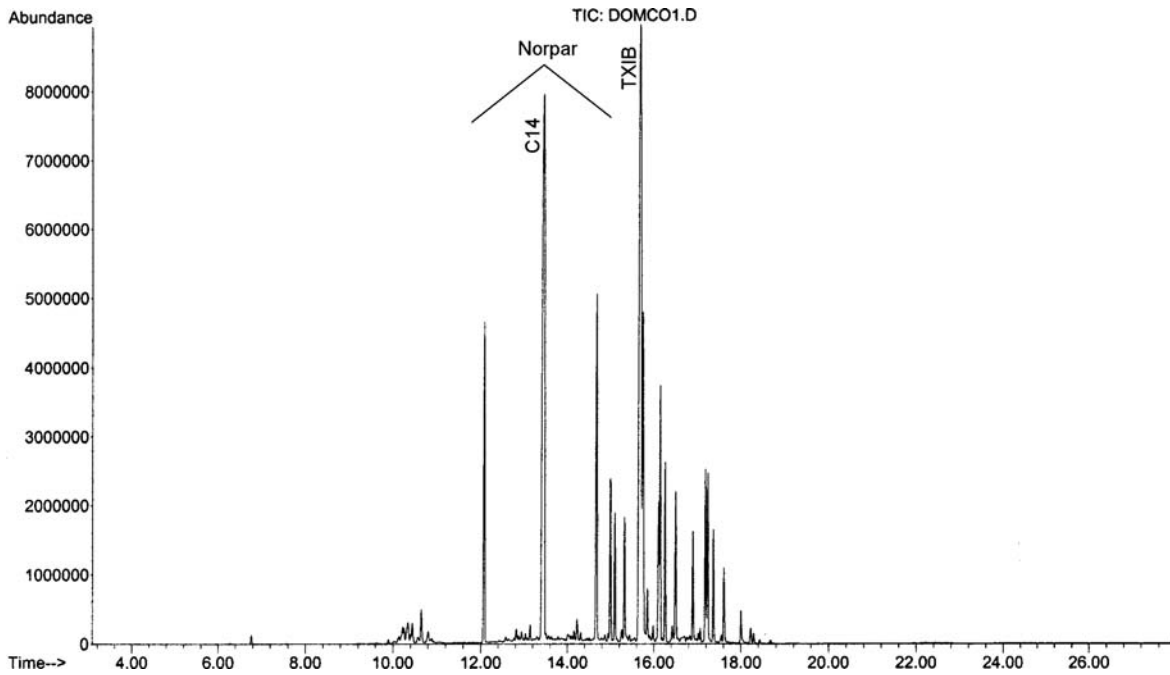


FIG. 4—Total ion chromatogram of vinyl flooring manufactured by Domco Customflor Legend Series 64273.

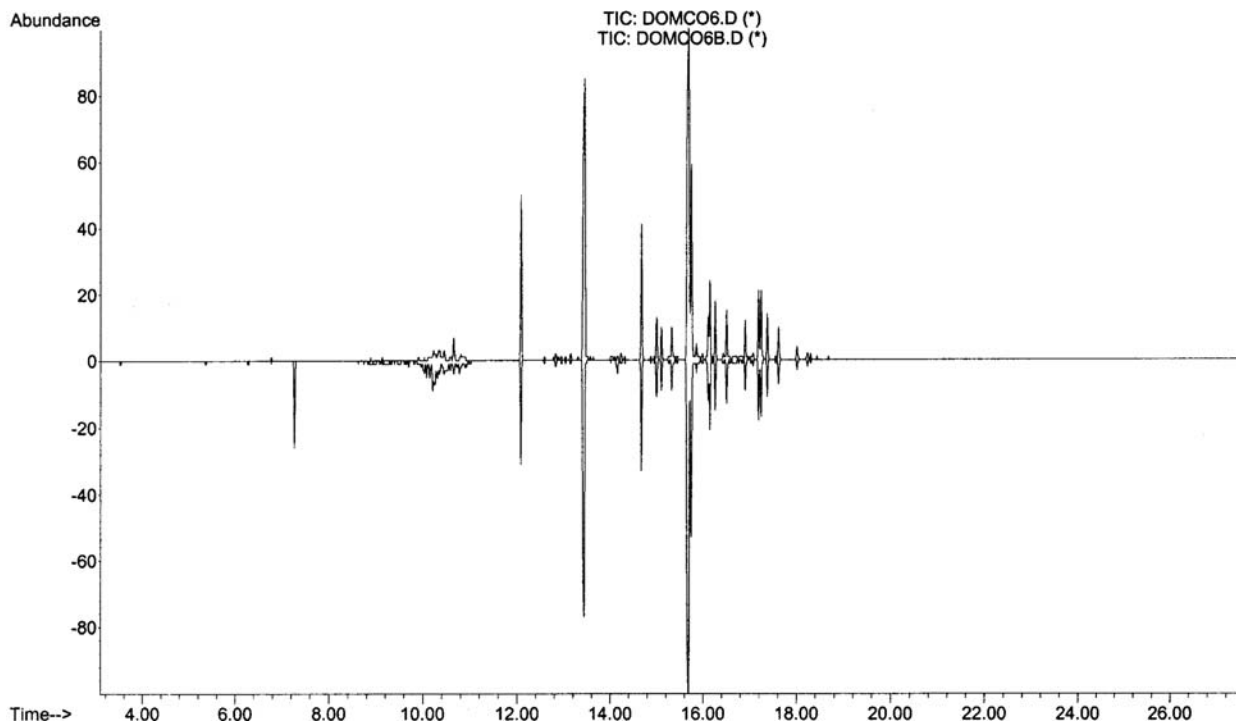


FIG. 5—Total ion chromatogram of Domco Customflor Concourse 65311. Domco 6 (top chromatogram) is unburned flooring. Domco 6B (bottom chromatogram) is burned flooring.

products have been reported in Zip-lock® bags and moisture barriers of laminated kraft paper (12). The identification of Isopar H in unburned and burned samples of vinyl flooring is pertinent information for fire debris analysts. Previously, the identification of Isopar H and/or Norpar in vinyl flooring was suspected to have originated from the adhesive or glue used during installation. However, this study shows the ignitable liquids identified (Nopars, TXIB, and

Isopar H) are being used as plasticizers during the manufacture of vinyl flooring and are not found in the adhesives used for vinyl installation.

From this study, a distinction cannot be made as to whether a charcoal starter containing Isopar H may have been used as an accelerant on the vinyl flooring or whether the Isopar H present was added during the vinyl manufacturing process. Comparison samples

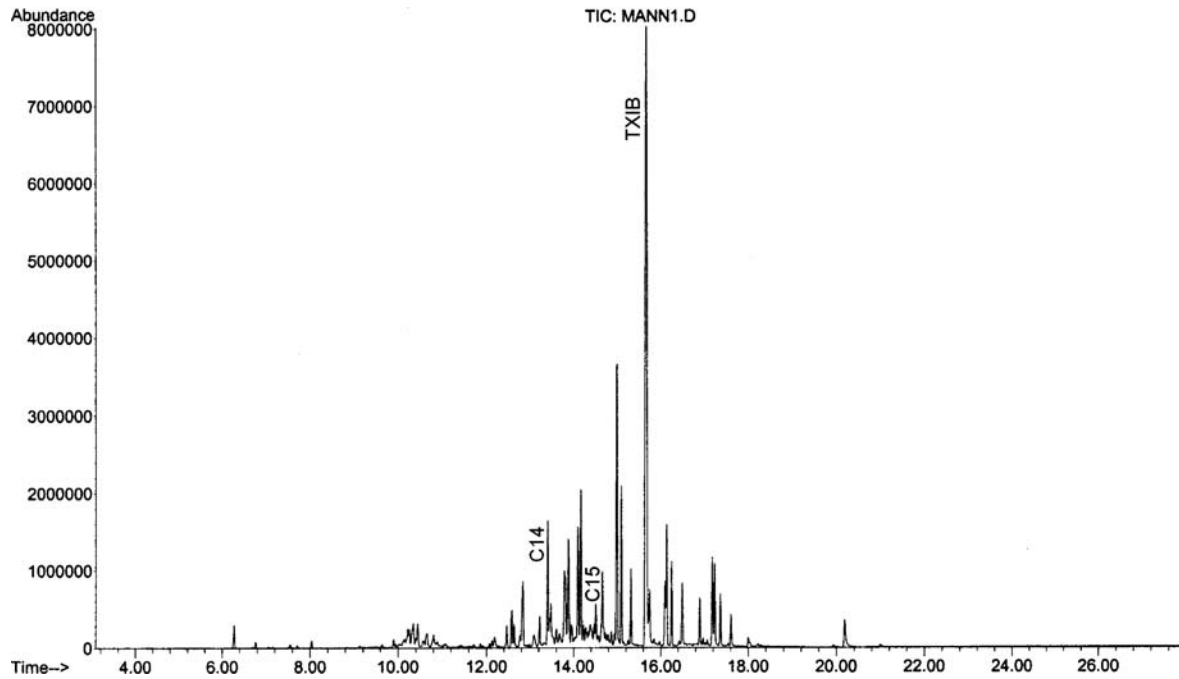


FIG. 6—Total ion chromatogram of vinyl flooring manufactured by Mannington Beautiflaire Series 10009.

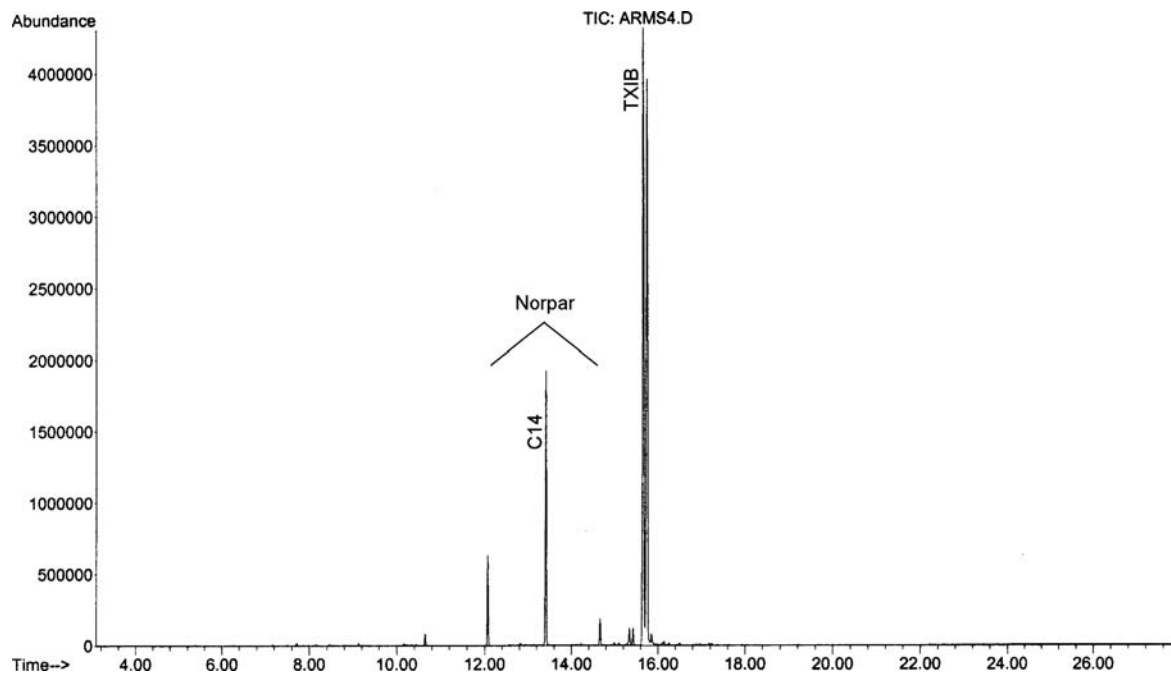


FIG. 7—Total ion chromatogram of vinyl flooring manufactured by Armstrong Ashton Series 46702.

would be helpful in cases containing vinyl flooring. Caution should be taken when reporting Isopar H and Norpar products in vinyl flooring as an ignitable liquid. At the very least, a disclaimer state-

ment should be added to all fire debris reports that contain vinyl flooring as a matrix in which an Isopar H product and/or Norpar product is identified. Further studies are needed in this area.

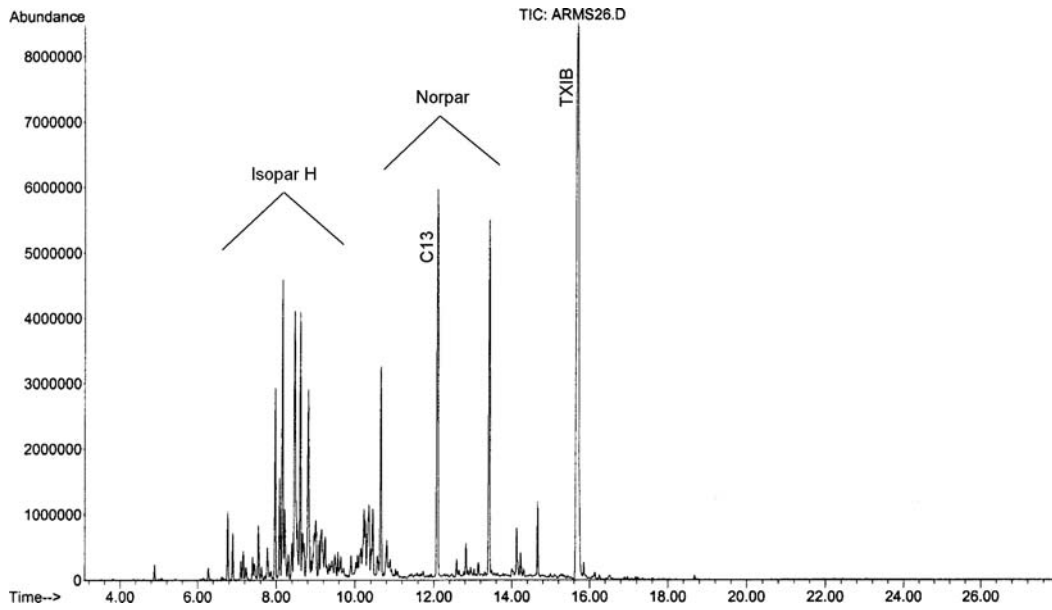


FIG. 8—Total ion chromatogram of vinyl flooring manufactured by Armstrong Interflex-Traditions Series 60221.

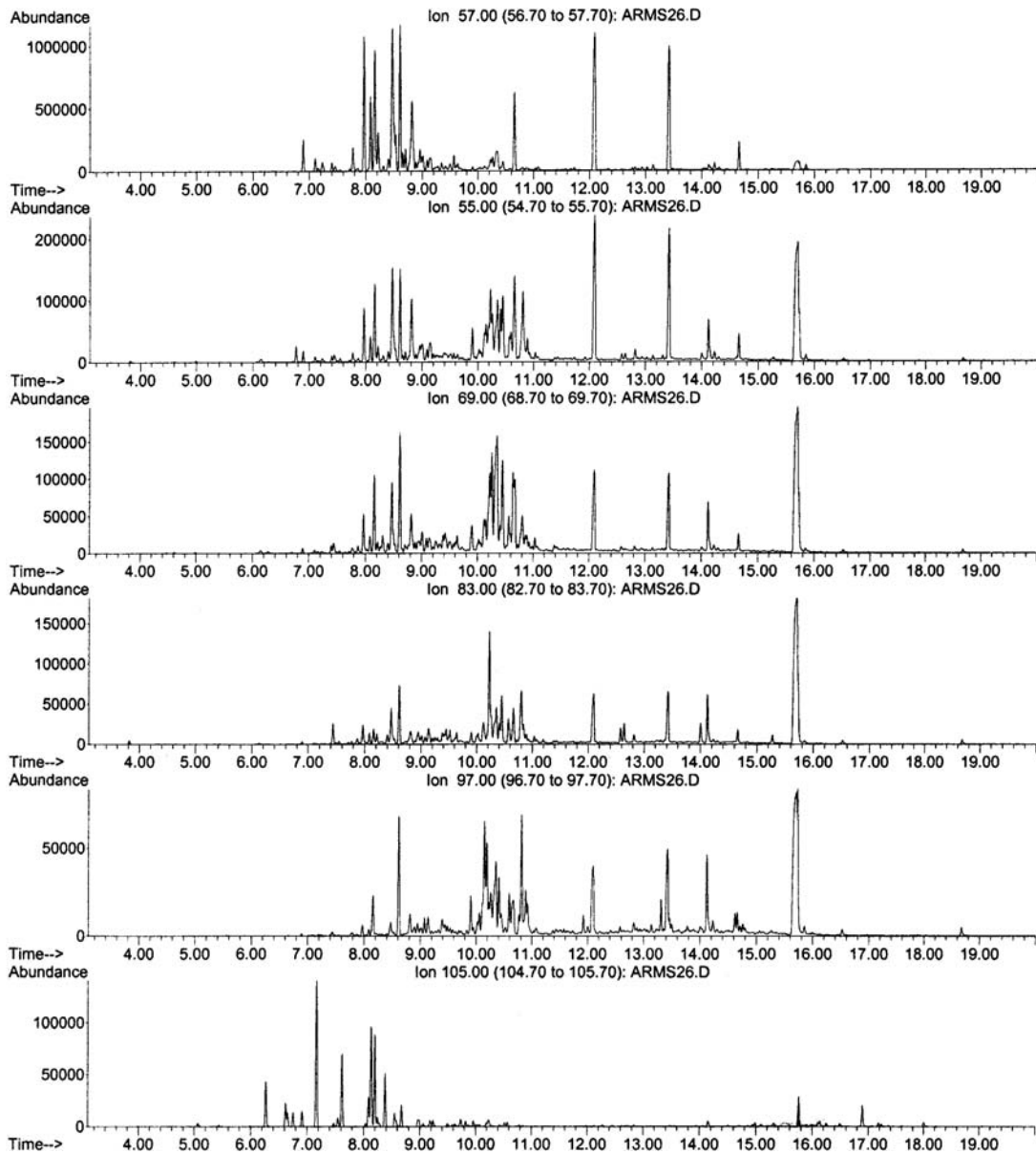


FIG. 9—Six selected ion profiles of Armstrong Interflex-Traditions Collection Series 60221.

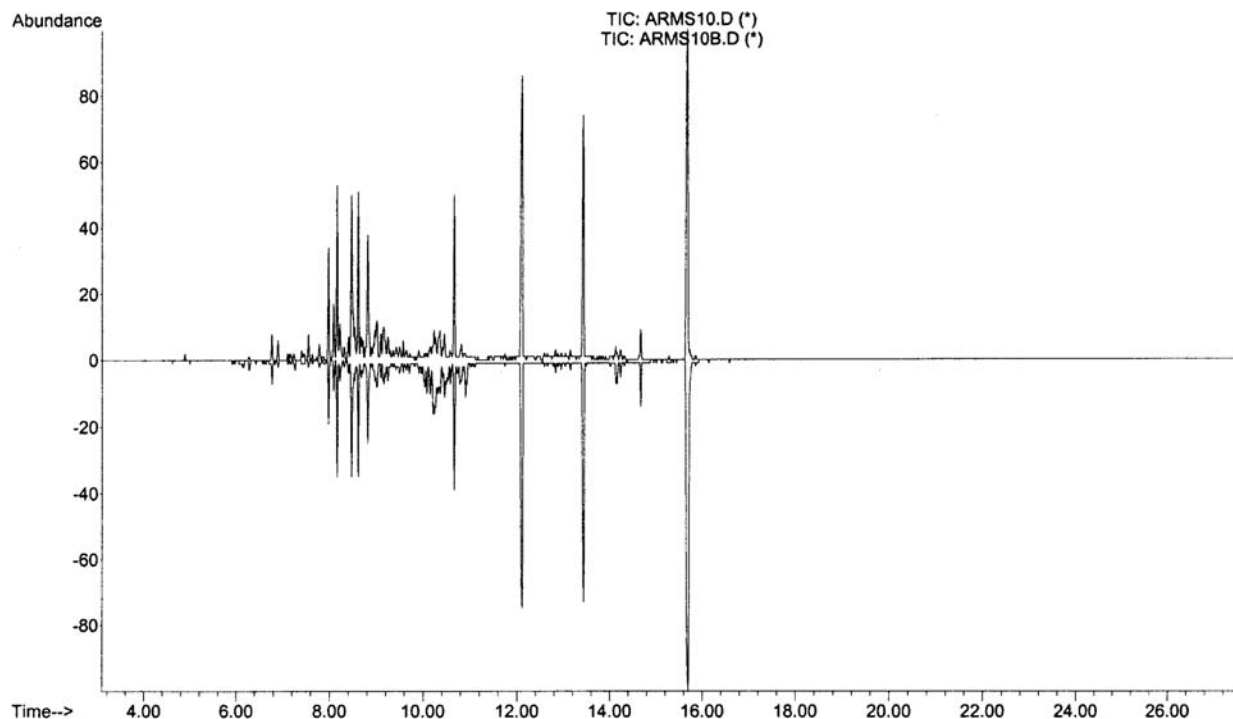


FIG. 10—Total ion chromatogram of Armstrong's Interflex-Traditions Collection Marble Dot Series 60103. Arms10 (top chromatogram) is unburned flooring and Arms10B (bottom chromatogram) is burned flooring.

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