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The Analysis of Tire Rubber Traces Collected After Braking Incidents Using Pyrolysis-Gas Chromatography/Mass Spectrometry

ABSTRACT: Automobile tire marks can routinely be found at the scenes of crime, particularly hit-and-run accidents and are left on road surfaces because of sudden braking or the wheels spinning. The tire marks are left due to the friction between the tire rubber and the solid road surface, and do not always demonstrate the tire tread pattern. However, the tire mark will contain traces of the tire. In this study, Pyrolysis Gas Chromatography/Mass Spectrometry was used to analyze 12 tires from different manufacturer's and their traces collected after braking incidents. Tire marks were left on a conglomerate road surface with sudden braking. The samples were pyrolysed without removal of contaminant in a micro-furnace type pyrolyser. Quantitative and qualitative analysis were performed on all the samples. All 12 samples were distinguished from each other. Each of the tire traces were identified as coming from there original source.

KEYWORDS: forensic science, tire marks, pyrolysis, rubber residues, skid

Tire rubber traces may be found at a wide variety of crime scenes, particularly hit-and-run accidents. The tire rubber trace is left on a road surface due to the friction between the tire and the road as the vehicle brakes. The amount of trace rubber left on the road is dependent on the speed of the vehicle, the condition of the tire, the condition of the road, and the temperature of the tires. The trace rubber will be in the form of black lines the width of each tire, in the direction the vehicle was traveling. Under skidding conditions, the tire is not rotating as it rubs against the road. The mark left can identify the width of the tire and the stance of the vehicle, but it may be difficult to distinguish the pattern of the tread. In circumstances where the tread of the pattern cannot be distinguished, the trace rubber that has been transferred from the tire to the road may provide a clue as to the identity of the tire. The mark left on the road is not all rubber polymer and is made up of the ingredients used to make the tire.

Tires are not 100% rubber, although rubber is the most important component. The tread of a tire has numerous ingredients that are used to make the final product. The choice of ingredients is dependent on the balance between the manufacturing conditions, cost, and the physical properties of the final product. Table 1 lists the different classes of ingredients required. For each class of ingredient required there are different possible compounds that can be used (1). There are three main rubber polymers used—Isoprene (natural rubber), 1, 3-Butadiene (BR), and Styrene-Butadiene copolymer (SBR) (2). SBR is the main rubber polymer used throughout the tire industry (3) but is not used on its own. It is always used with isoprene, BR or both, although the percentage of each used will vary. There will also be variation within the SBR polymer group by changing the ratio of styrene: BR. Three different BR monomer units can also be used—cis-1, 4; trans-1, 4; and trans-1, 2 (4).

The rubber group is the class of ingredient with the least variation, although is present in the greatest amount. The fillers and extender oils are next in abundance, and it is the carbon black filler that gives the tire its black color. The remaining ingredients are present in very small percentages. There are 1000s of different plasticizers, peptizers, and anti-oxidants that may be used in the tire formulation. The differences in the recipe used to formulate the tread can vary between manufacturers, but also within the same manufacturer. These differences can be used to distinguish between tires from different manufacturers and tires from the same manufacturer. It is also possible that tires from different manufacturers have very similar formulations.

The trace rubber residue, if correctly classified or identified, could lead to the vehicle/s involved in the crime being included in the suspect group. The trace rubber residue could also be used to exclude suspected vehicles.

Pyrolysis-gas chromatography/mass spectrometry has been shown to be an effective technique to analyze rubber. Over the last 40 years, researchers have used Py-GC to analyze rubber and vulcanized rubber. The aims of the research have varied greatly, but there has been limited research investigating the differences between products. In 1989, Ding and Liu pyrolysed their tire rubber samples at 750°C (5). At this temperature the vulcanized rubber sample is completely pyrolysed. More recently however, Sarkissian et al. found that a low pyrolysis temperature of 450°C gave better discrimination between tire samples (6), even though pyrolysis of the vulcanized rubber sample is considered incomplete at this temperature.

Materials and Methods

Twelve tires from different manufacturers were used to create the braking marks. Each of the tires was made in the Asia Pacific region. Seven of the tires were brand new, while five of the tires had previously been used. All the tires were of the same size. The tires used are listed below.

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TABLE 1—*Classes of ingredients used in tire manufacture.*

Ingredients used in tire manufacture	
Class	Example
Rubber	Natural and synthetic
Fillers	Carbon black and silica
Extender oils	Distilled aromatic extract oils
Vulcanizing agents	Sulphur
Accelerators	Benzothiazoles
Activators	Zinc oxide and stearic acid
Processing aids	Peptizers, plasticizers, and softeners
Protective agents	Antioxidants and antiozonants

- Bridgestone (used) – Australia
- Dunlop (new) – Australia
- Federal (new) – Japan
- Firestone (used) – Taiwan
- Goodyear (used) – Australia
- Hankook (new) – Korea
- Kuhmo (used) – Korea
- Nankang (new) – Taiwan
- Sime (new) – Malaysia
- Sumitomo (used) – Japan
- Toyo (new) – Japan
- Yokohama (new) – Japan

Braking marks were left under controlled conditions. A braking mark was left by each tire with a vehicle traveling at 60 km/h on a concrete road containing gravel aggregate. The section of road used for each incident was clear of any visible tire marks. The tire rubber from the skid marks were collected directly off the road surface using tweezers. A tire residue was able to be collected from each of the four tires through one skidding incident. The section of skid between the front and rear tires was used for the front tires. During the skid, the camber of the road resulted in the vehicle veering from a straight line. This left a narrow section in which the rear tires did not overlap the front tires. This will not always be the case, depending on the driver, the road, and the speed of the vehicle, the skid may be in a straight line from beginning to end. Even in this situation it is possible to collect a brake mark residue from each of the tires. Residues collected from the starting point of the skid will originate from the rear tires, while the residue collected from the end of the skid will be from the front tires only. A section where the veered from the straight line during the skid was used to collect tire residue left from the rear tires. Figure 1 shows a section of one of the skid marks.

Pyrolysis was carried out using a Shimadzu Furnace Pyrolyser-4a Ver.2 set at 450°C, mounted on the split injector of the GC/MS. The analysis was carried out using a Shimadzu GC-17A gas chromatograph coupled to a Shimadzu GCMS-QP5050A mass spectrometer. Chromatography was performed using a J & W Scientific DB-5MS column, 60 m in length with an internal diameter of 0.25 mm. System control was carried out using the Shimadzu Class-5000 software. Mass spectra were tentatively identified using the NIST libraries.

Between 100 μg and 500 μg of sample was used for analysis. Six repeats were completed for each tire sample, and three repeats for each skid mark.

Qualitative analysis was completed using target compound identification (TCI). Target compounds were identified from the six repeat analyses of each sample, and compared with the other tire samples and the tire trace.

Peak area values were calculated using HP software. The data was weighted to remove heteroscedastic noise, and then normalized to account for the variations in sample size. Quantitative analysis was completed using a combination of principal components analysis (PCA) and linear discriminant analysis (LDA). The PCA and LDA were performed using Minitab version 13.1 software.

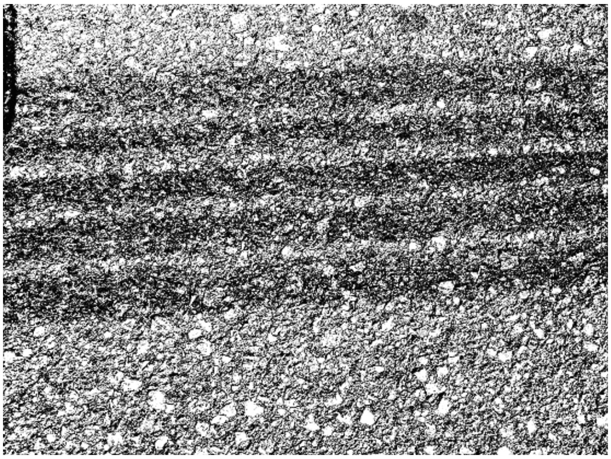


FIG. 1—*Short section of skid mark on concrete road with gravel aggregate.*

Results

Each of the 12 samples was examined for both consistencies and inconsistencies over the duration of the chromatogram and through six repeat analyses. For all 12 samples combined, 164 peaks with different mass spectra were observed. Some of these peaks did have the same retention time but were present in different samples. Table 2 lists all the identified and unidentified peaks. Any peak whose mass spectra could not be identified has been named unidentified 1, unidentified 2 etc., based on the retention time. The identified compounds include rubber polymers, accelerators, activators, aromatic compounds, antioxidants, plasticizers, softeners, polymerization by-products, processing aids, and pyrolysis products. Of all the ingredients used in manufacture it appears that carbon black plays no part in the analysis. After pyrolysis of the sample (tire or skid), the carbon black residue is left behind in the pyrolysis bucket.

For each sample the chromatograms of the skid marks were examined to identify those peaks present in both the tire and skid mark. Of the 164 peaks (identified and unidentified), only 43 were found to be consistently present in both the tire sample and skid mark sample. The 43 consistent peaks were present in at least five of the six repeat analyses from the tire, and present in at least two of the three repeat analyses of the skid mark. These peaks were used for both the TCI and LDA and are listed in Table 3. The remaining 121 identified and unidentified peaks were not present in the skid marks and were not used for comparison purposes.

Of the 43 peaks, 35 were found to be present in all 12 samples. The remaining eight peaks were used to discriminate the tires. Differences were found between 10 of the 12 tire samples. Only the Goodyear and Nankang tires could not be discriminated. Table 4 shows which of the eight peaks were present in which sample. Based on these eight peaks, 10 of the 12 skid mark samples were identified as coming from the tire that left them. As the Goodyear and Nankang tire samples could not be differentiated based on the presence or absence of these peaks, the skid marks from these tires could only be identified as originating from either the Goodyear or Nankang tire.

TABLE 2—*Identified and unidentified compounds observed in all 12 tire samples.*

Peak No.	Retention time (minutes)	Compound	Peak no.	Retention time (minutes)	Compound	Peak no.	Retention time (minutes)	Compound	Peak no.	Retention time (minutes)	Compound
1	2.95	1,3-Butadiene	42	20.083	Unidentified 17	83	26.767	Unidentified 42	124	30.35	Unidentified 78
2	3.25	Isoprene	43	20.317	Biphenyl	84	26.867	Unidentified 43	125	30.367	Unidentified 79
3	3.517	Unidentified 1	44	20.617	Unidentified 18	85	27.183	Unidentified 44	126	30.4	Unidentified 80
4	6.633	Toluene	45	20.617	Unidentified 19	86	27.283	Unidentified 45	127	30.417	Benzothiazole Disulfide
5	8.083	4-Vinylcyclohexene	46	20.617	Unidentified 20	87	27.333	Unidentified 46	128	30.433	Unidentified 81
6	8.517	1-Ethylcyclohexene	47	20.733	Unidentified 21	88	27.583	Unidentified 47	129	30.45	Unidentified 82
7	8.767	Ethylbenzene	48	20.767	Unidentified 22	89	27.883	Unidentified 48	130	30.467	Unidentified 83
8	9	1,2-Dimethylbenzene	49	20.783	Unidentified 23	90	27.833	Unidentified 49	131	30.467	Unidentified 84
9	9.517	Styrene	50	20.8	Unidentified 24	91	27.883	Docosane	132	30.467	Unidentified 85
10	11.033	Propylbenzene	51	21.267	Acetone anil	92	28.15	Hexadecane	133	30.5	Unidentified 86
11	11.033	D-limonene	52	21.417	Unidentified 25	93	28.133	Unidentified 50	134	30.517	Unidentified 87
12	11.683	Methylstyrene	53	21.417	Dimethyl phthalate	94	28.233	Unidentified 51	135	30.533	Unidentified 88
13	12.233	2,5,6-Trimethyl-1,3,6-heptatriene	54	21.55	Unidentified 26	95	28.2	Unidentified 52	136	30.567	Unidentified 89
14	12.65	8-Methylene-dispsiro 2.0.2.5 undecane	55	21.567	2,2,4-Trimethyl-3,4-dihydroquinoline	96	28.55	Antioxidant 2246	137	30.583	Octadecane
15	12.733	Limonene	56	21.75	1-Pentadecene	97	28.667	Unidentified 53	138	30.583	Unidentified 90
16	13.433	Butylbenzene	57	21.917	Pentadecane	98	28.717	Unidentified 54	139	30.617	9-Octadecanoic acid methyl ester
17	14.65	1,5-Diethenyl-3-methyl-2-methylenecyclohexane	58	22.1	Unidentified 27	99	28.75	Octadecane	140	30.683	Unidentified 91
18	15.467	4-Pentenylbenzene	59	22.117	Butylated hydroxytoluene	100	28.817	Unidentified 55	141	30.683	Unidentified 92
19	15.617	1,2,3,4,5,8-Hexahydronapthalene	60	22.733	Unidentified 28	101	28.933	Hexadecanoic acid	142	30.783	6-Hydroxynobiline
20	16.15	1,2,4-Trivinylcyclohexane	61	22.733	Unidentified 29	102	29.2	Unidentified 56	143	30.8	Unidentified 93
21	16.383	1-Phenylcyclopentene	62	23.383	Unidentified	103	29.2	Unidentified 57	144	30.85	Unidentified 94
22	16.567	1,1-Dimethylindene	63	23.483	Nonadeacne	104	29.3	Unidentified 58	145	30.933	Unidentified 95
23	17.15	Unidentified 2	64	23.617	Diethyl phthalate	105	29.467	Unidentified 59	146	31.217	Unidentified 96
24	17.15	Unidentified 3	65	23.883	Unidentified 30	106	29.483	Unidentified 60	147	31.233	Oleic Acid
25	17.55	Unidentified 4	66	23.95	*p*-Octylphenol	107	29.517	Unidentified 61	148	31.25	Unidentified 97
26	17.567	Unidentified 5	67	24.333	Unidentified 31	108	29.55	Unidentified 62	149	31.35	Unidentified 98
27	17.683	Unidentified 6	68	24.533	Unidentified 32	109	29.567	Unidentified 63	150	31.381	Unidentified 99
28	17.683	Hexylbenzene	69	24.617	Unidentified 33	110	29.6	Unidentified 64	151	31.383	Unidentified 100
29	17.867	4-Hexenylbenzene	70	24.7	Unidentified 34	111	29.617	Unidentified 65	152	31.417	Unidentified 101
30	17.933	Unidentified 7	71	24.833	1,3-Diphenylpropane	112	29.783	Unidentified 66	153	31.517	Unidentified 102
31	18.05	Unidentified 8	72	25.117	Unidentified 35	113	30.017	Unidentified 67	154	31.517	Stearic acid
32	18.233	Unidentified 9	73	25.133	Heptadecane	114	30.1	Unidentified 68	155	31.55	Unidentified 103
33	18.25	Unidentified 10	74	25.167	Unidentified 36	115	30.167	Unidentified 69	156	31.683	Unidentified 104
34	18.267	Unidentified 11	75	25.183	Unidentified 37	116	30.25	Unidentified 70	157	31.767	Unidentified 105
35	18.65	1-Phenyl-1-cyclopentanol	76	25.333	3-Hydroxyphenylbenzoate	117	30.283	Unidentified 71	158	31.85	Unidentified 106
36	19.05	Unidentified 12	77	25.4	Isobutyrophenone	118	30.267	Unidentified 72	159	31.867	Unidentified 107
37	19.05	Unidentified 13	78	25.867	Unidentified 38	119	30.267	Unidentified 73	160	31.883	Unidentified 108
38	19.067	Unidentified 14	79	26.3	Unidentified 39	120	30.283	Unidentified 74	161	31.917	Unidentified 109
39	19.3	4-Phenylcyclohexene	80	26.35	Unidentified 40	121	30.3	Unidentified 75	162	31.933	Unidentified 110
40	20.1	Unidentified 15	81	26.4	Unidentified 41	122	30.3	Unidentified 76	163	31.933	Unidentified 111
41	20.1	Unidentified 16	82	26.517	2-Methylhexadecane	123	30.35	Unidentified 77	164	31.95	Unidentified 112

TABLE 3—*The 43 consistent compounds used for both TCI and linear discriminant analysis.*

Peak no.	Retention time (min)	Compound	Peak no.	Retention time (min)	Compound
1	2.983	1,3-Butadiene	55	21.567	2,2,4-Trimethyl-3,4-dihydroquinoline
2	3.239	Isoprene	57	21.917	Pentadecane
3	3.517	Unidentified 1	59	22.117	Butylated hydroxytoluene
4	6.631	Toluene	60	22.733	Unidentified 28
5	8.09	4-Vinylcyclohexene	62	23.383	Unidentified
6	8.489	1-Ethylcyclohexene	63	23.483	Nonadeacne
7	8.767	Ethylbenzene	66	23.95	p-Octylphenol
9	9.516	Styrene	67	24.333	Unidentified 31
10	11.033	Propylbenzene	68	24.533	Unidentified 32
11	11.036	D-limonene	69	24.617	Unidentified 33
12	11.697	Methylstyrene	70	24.7	Unidentified 34
15	12.734	Limonene	71	24.833	1,3-Diphenylpropane
17	14.653	1,5-Diethenyl-3-methyl-2-methylenecyclohexane	73	25.183	Unidentified 37
19	15.617	1,2,3,4,5,8-Hexahydronapthalene	77	26.3	Unidentified 39
20	16.15	1,2,4-Trivinylcyclohexane	79	26.4	Unidentified 41
26	17.567	Unidentified 5	80	26.517	2-Methylhexadecane
28	17.683	Hexylbenzene	82	26.867	Unidentified 43
35	18.65	1-Phenyl-1-cyclopentanol	98	28.817	Unidentified 55
38	19.067	Unidentified 14	99	28.933	Hexadecanoic acid
39	19.3	4-Phenylcyclohexene	105	29.517	Unidentified 61
43	20.317	Biphenyl	110	29.783	Unidentified 66
51	21.267	Acetone anil			

TABLE 4—*Presence and absence of eight peaks throughout all 12 tire samples.*

Sample peak no.	Bridgestone	Dunlop	Firestone	Federal	Goodyear	Hankook	Nankang	Sime	Toyo	Yokohama	Khumo	Sumitomo
3	No	Yes	Yes	No	No	No	No	No	No	No	No	No
10	Yes	Yes	Yes	No	Yes	Yes	Yes	Yes	Yes	Yes	Yes	No
11	Yes	No	No	Yes	Yes	No	Yes	Yes	Yes	Yes	No	Yes
55	No	No	No	No	No	Yes	No	No	No	Yes	Yes	No
63	No	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
66	No	No	No	No	Yes	Yes	Yes	Yes	No	Yes	No	No
70	No	No	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
105	No	No	No	No	No	Yes	No	Yes	No	No	No	No

Linear discriminant analysis is able to classify data by measuring the squared distance between an observation and a group to decide class membership. Depending on the number of variables present in a dataset, all these variables may be used, or the principal components can be used. In this case LDA could not be performed using all 43 variables as there were not enough samples. Principal component analysis was used to reduce the data set. PCA was used to transform the original data set into principal components using a linear algorithm. From the scree plot, it could be observed that 90% of the variability (eigenvalue >1) was contained within the first eight principal components. The scores of the first eight PC's were calculated and these values were used to perform the LDA. The dataset was reduced from 43 variables to eight PC's.

The LDA correctly classified all the tire samples including the Goodyear and Nankang tires. Of the 36 brake marks (three for each sample), 28 were identified as originating from the correct sample. Each of the Goodyear and Nankang brake marks were identified as being from the Toyo tire. Of the three Bridgestone brake marks, only one was identified as being from the Bridgestone tire. The other two Bridgestone brake marks were the only two of the 36, not to be identified 100%. One was identified as having an 83% change of originating from the Dunlop tire and a 17% chance as being from the Bridgestone tire. The other one was identified as having a 65% chance of originating from the Yokohama tire and a 35% chance as being from the Bridgestone tire.

Discussion

Based on the six repeat analyses the tire samples were found not to be homogenous. Differences were observed in the chromatograms of the same sample after the 25-min mark. This area of the chromatogram was generally found to contain antioxidants, long-chain organic acids, and straight chain carbon compounds. The differences observed were peaks that would only appear in less than half of the six analyses. This would suggest that there is a much smaller amount of these compounds, compared with the consistent compounds, present in the tire. These low-level compounds lead to difficulties in both potential identification and discrimination. In both the TCI and LDA these inconsistently present peaks were noted but not included. The six repeat analyses allowed for the inconsistent peaks to be identified and their presence explained without being involved in either the TCI or LDA. For a peak to be considered consistent, it was required to be present in five of the six repeat analyses of the tire sample. The inconsistent peaks, if required, could be used as secondary identifiers.

During the skidding process a large amount of smoke and heat were generated. Burnt rubber could clearly be smelt. Therefore it is not surprising that structural differences could be observed between the samples collected from the tire and the samples collected from the road. The samples collected from the tire are soft and resilient. The samples collected from the road are hard and brittle. This is shown in Fig. 2. These physical differences between the source

FIG. 2—*Sample collected from skid mark.*

material and the collected material may be the cause of differences observed in the chromatograms between the tire sample and skid mark sample for all of the tires tested.

For each sample, there were no peaks present in the skid mark chromatogram that had not been present in the tire chromatogram, however, there were peaks present in the tire chromatogram that were not present in the skid mark chromatogram. Almost all of the inconsistent peaks were absent from the skid mark chromatograms. As these peaks are not present in the skid mark, the skid process must result in these compounds being lost or not being left behind. This also meant that for identification purposes, as these inconsistent peaks were not present in the skid mark chromatograms and only the consistent peaks in the tire sample were present in the skid mark chromatogram, the inconsistent peaks and the heterogeneity of the tire sample could be ignored. The only inconsistent peak that was also present in the skid mark sample was antioxidant 2246. As stated earlier, this could be used as a secondary identifier if required.

A second difference observed was with changes in mass spectra of peaks at similar retention times, between the tire sample and skid mark sample. This occurred with the peaks at 19.05 min and 22.1 min. At these two retention times, the mass spectra for all 12 skid mark samples were similar. However, there were clear differences in the mass spectra at the two retention times in the tire samples. Figures 3*a* and 3*b* are the mass spectra of peaks at 19.05 min in two of the tire sample chromatograms. Figure 3*c* is the mass spectrum of the peak at 19.05 min in the skid mark chromatogram for all 12 skid mark samples. There are clear differences between all the spectra.

Figures 4*a* and 4*b* are mass spectra of peaks at 22.1 min in tire sample chromatograms. Figure 4*c* is the mass spectra of the same peak but in the skid mark chromatogram. Figures 4*a* and 4*c* are similar, however there are differences between Figures 4*b* and 4*c*.

Even though there are some clear changes in mass spectra for peaks at these retention times, the presence and size of the peaks is consistent for all 12 samples. For this reason both of these peaks were used as primary identifiers.

The third difference observed was at 28.90 min on the chromatogram. For the tire samples that had a peak at this retention time, the MS was identified as being due to hexadecanoic acid. The size of this peak varied between the samples. However, in the brake mark samples the peak at this retention time was completely different. The new mass spectrum was identified as being dibutyl phthalate (Figures 5*a* and 5*b*). This peak was also used as a primary identifier in both the TCI and LDA as the change in compounds was evident in all samples that had hexadecanoic acid.

Conclusion

All 12 of the tires could be discriminated using LDA, and 10 of the 12 tires could be discriminated using target compound identification. It was found that tires are not homogenous and that differences will be seen in the repeat analysis of a sample. Physical changes were also observed between the tire sample and the skid mark sample.

In the chromatograms, differences could also be observed between the tire samples and skid mark samples. These differences were mainly in the last 7 min of the chromatogram where specific

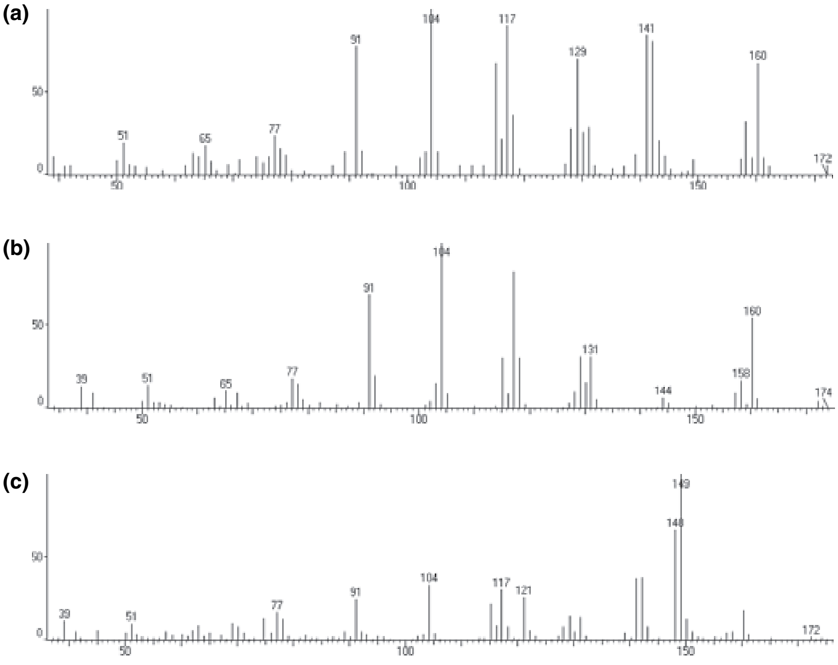


FIG. 3—*(a) Mass spectra at 19.05 min in chromatogram of tire sample 1. (b) Mass spectra at 19.05 min in chromatogram of tire sample 2. (c) Mass spectra at 19.05 min in chromatogram of skid mark sample 7.*

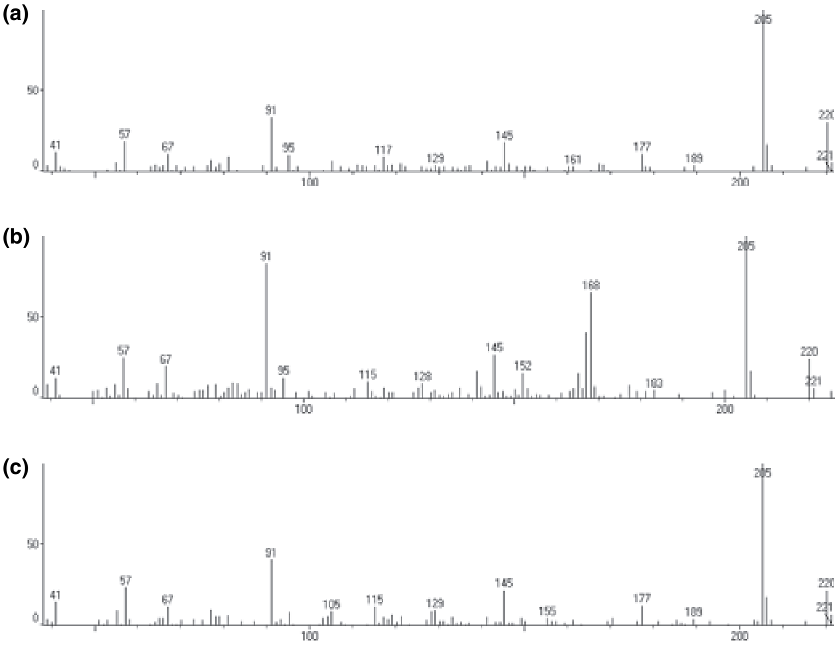
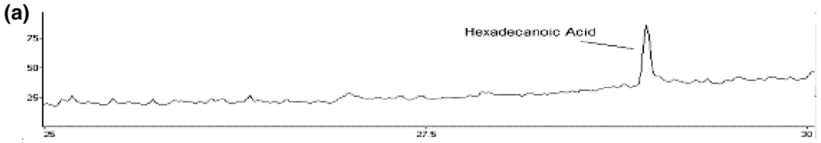
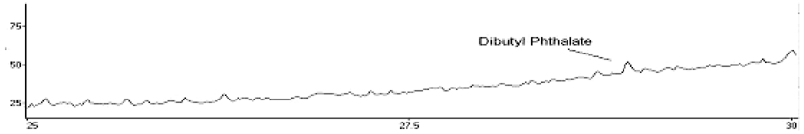


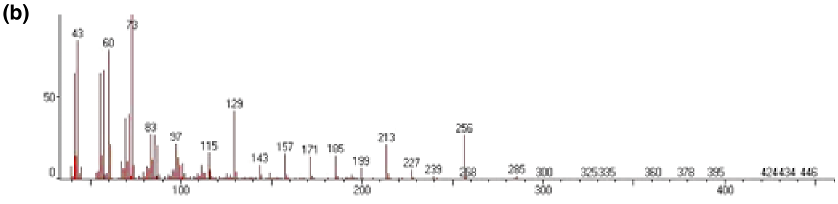
FIG. 4—*(a) Mass spectra at 22.10 min in chromatogram of tire sample 1. (b) Mass spectra at 22.10 min in chromatogram of tire sample 4. (c) Mass spectra at 22.10 min in chromatogram of skid mark sample 5.*



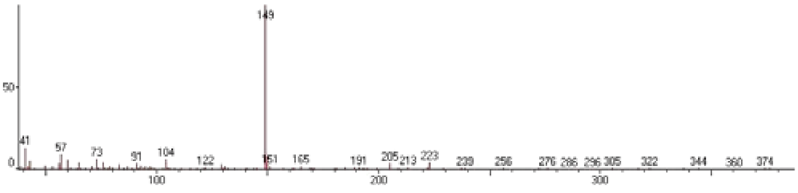
Section of Goodyear Tyre Pyrogram



Section of Goodyear Residue Pyrogram



Hexadecanoic Acid



Dibutyl Phthalate

FIG. 5—*(a) Position of peak in both Goodyear tire and skid mark samples. (b) Different spectra of peaks with the same retention time. This shows a change from the tire sample to the skid mark sample.*

peaks present in the tire sample would either be absent or different in the skid mark chromatogram. Peaks present in the tire sample but absent in the skid mark sample could be ignored as the compound has been lost or not left behind during the skid process. Only those peaks present in both the tire and skid mark sample were used for potential identification.

Using target compound identification 10 of the 12 brake mark samples could be identified, while only nine of the 12 could be identified using LDA. Because of the changes occurring during the skid, skid mark samples are more likely to be identified correctly when compared with other skid mark samples rather than the tire itself.

Acknowledgments

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