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The Characterization of Plastic Automobile Bumper Bars Using Fourier Transform Infrared Spectroscopy (FTIR), Pyrolysis Gas Chromatography (PGC), and Energy Dispersive X-Ray Microanalysis Combined with a Scanning Electron Microscope (SEM-EDX)

REFERENCE: Parybyk, A. E. and Kobus, H. J., "The Characterization of Plastic Automobile Bumper Bars Using Fourier Transform Infrared Spectroscopy (FTIR), Pyrolysis Gas Chromatography (PGC), and Energy Dispersive X-Ray Microanalysis Combined with a Scanning Electron Microscope (SEM-EDX)," *Journal of Forensic Sciences*, JFSCA, Vol. 35, No. 2, March 1990, pp. 281-292.

ABSTRACT: A collection of 28 plastic automobile bumper bars has been examined using Fourier transform infrared spectroscopy (FTIR), pyrolysis gas chromatography (PGC), and energy dispersive X-ray microanalysis combined with a scanning electron microscope (SEM-EDX). FTIR identified 8 polymer classes in the collection. Polypropylene-based (PP) plastics and polyurethane (PUR) plastics were the most common with approximately three quarters of the samples being of these types. PGC provided additional discrimination between the samples in the collection, particularly for the PUR samples, defining a total of 13 categories. SEM-EDX was the most discriminatory technique used and resulted in 17 categories being identified. However, the combination of the 3 techniques of FTIR, PGC, and SEM-EDX was recommended as the preferred approach as it provided a high degree of discrimination with 25 categories identified. Vehicle identification via analytical profiles of bumper bar materials was not possible, and consequently, the establishment of a database using such information for investigative purposes could not be recommended.

KEYWORDS: criminalistics, automobiles, plastics, spectroscopic analysis, chromatographic analysis, X-ray analysis

The benefits associated with the use of plastics in automobile manufacture such as lower costs, corrosion resistance, reduced vehicle weight, and design freedom [1-4] have resulted in a dramatic escalation in the range of plastic components used on motor vehicles in the last decade. Thus forensic science laboratories are often required to examine samples of plastic from vehicles involved in hit-run and other vehicle accident investigations.

The purpose of the work reported in this paper was to examine the value of Fourier transform infrared spectroscopy (FTIR), pyrolysis gas chromatography (PGC), and en-

This work was submitted in partial fulfillment of the requirements for the degree of M.S.F.S. from the George Washington University, Washington DC.

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ergy dispersive X-ray microanalysis using a scanning electron microscope (SEM-EDX) for the identification and discrimination of the materials used in plastic bumper bars. These techniques were chosen as they are generally available in forensic science laboratories [5-14] and characterize materials on the basis of both organic and inorganic content.

Although this study deals with vehicles available in Australia, the results should have wider application, particularly with the growing trend towards the "world car."

Experimental Procedure

Sample Selection

Bumper Bars—Statistics relating to new vehicle registrations and vehicle accidents² indicated that a representative collection of bumper bar samples would need to include the five major motor vehicle manufacturers in Australia, namely Ford, General Motors Holden, Mitsubishi, AMI Toyota, and Nissan. In addition, samples from some European models would complement the collection.

Samples were obtained directly from the manufacturer where possible or from reputable crash repairers. Information regarding the model of the vehicle, year of manufacture, and composition of the part was requested. However in some instances this was not available or was unreliable. Only samples for which authentic information could be obtained were included in the collection.

The plastics commonly used in vehicle manufacture are shown in Table 1. The abbreviations given are used throughout the paper. The 28 bumper bar samples collected are listed in Table 2.

Polymer reference standards—A collection of polymer reference standards was purchased from Scientific Polymer Products (Ontario, New York 14519). The standards were used to generate a library of infrared spectra and pyrograms.

Analytical Procedures

Microscopic Procedures

The bumper bar samples were examined using a stereomicroscope. The color, texture, and resilience of the samples was noted.

TABLE 1—*Key to plastics used in automotive manufacture.*

Plastic	Abbreviation
Polypropylene	PP
Ethylene propylene diene modified	EPDM
Polycarbonate	PC
Polyurethane	PUR
Polyurethane (reaction injection molded)	PUR(RIM)
Polyester	PES
Acrylonitrile butadiene styrene	ABS
Polyamide	PA
Polybutylene terephthalate	PBT
Polyphenylene oxide	PPO

²South Australian Department of Transport, personal communication, 1987.

TABLE 2—Bumper bar samples.

Sample	Manufacturer	Model	Year	Composition
1	GMH	Camira	1987	PP/EPDM
2	GMH	Gemini	1987	PP/EPDM
4	GMH	Commodore	1987	PC
6	GMH	Commodore	1987	PC
7	AMI Toyota	Corona CS	1986	PP/EPDM
13	Mercedes	123, 240D, 230E, 280	1974–1982	EPDM, PUR
	Mercedes	124, 300E, 230E	1979–1987	PC, PP, PUR
16	GMH	Barina	1986	... ^a
20	Ford	Falcon XD	1981	PUR(RIM)
21	Fiat	132	1979	PP
22	AMI Toyota	Corolla CSX	1985	PUR(RIM)
23	BMW	E30	1983–1985	... ^a
24	AMI Toyota	Tarago	1986	PUR
25	BMW	528i	1984	... ^a
26	Mitsubishi	Magna	1985	PP/EPDM
28	Audi	GLE	1981	EPDM, PUR
29	GMH	Camira	1982–1983	... ^a
34	GMH	Camira	1982–1983 ^b	... ^a
35	Ford	Telstar	1984	... ^a
40	Mitsubishi	Sigma	1984 ^b	... ^a
42	Nissan	Pulsar	1986–1987	PP/EPDM
61	Nissan	Pintara, Skyline	1986–1987	PP/EPDM
66	Nissan	Pintara, Skyline	1986–1987	PP/EPDM
67	Ford	Falcon XF	1984–1987	PUR(RIM)
73	Ford	Commercial	1978–1986	... ^a
76	Ford	Falcon XF	1984–1987	PUR(RIM)
77	Ford	Falcon XF	1984–1987	PUR(RIM)
78	Ford	Falcon XF	1984–1987	PUR(RIM)
85				

^aSamples for which composition was not provided.

^bSamples for which the year of manufacture was identified as approximate only.

Infrared Spectroscopy

Instrumentation—A Digilab FTS 50 FTIR system equipped with the Digilab Infrared Microscope Accessory (IRMA) was used. The IRMA incorporated a $\times 32$ Cassegrainian objective and a liquid nitrogen cooled mercury-cadmium-telluride detector.

The infrared beam from the instrument was focused on the sample which was placed on the *X-Y* stage of the microscope with precise sample area isolation controlled by a rectangular aperture. Spectra were obtained in absorbance mode over the range of 700 to 3800 cm^{-1} using an aperture size of $50 \times 250 \mu\text{m}$ at resolution 4 cm^{-1} for 256 scans.

Sample Preparation—The main advantage of the microscope accessory was that small samples could be conveniently handled and the need for elaborate ultramicrosampling procedures [15] was avoided.

Standards—Preparation varied depending on the original form of the material. Standards that were in the form of fine powders were pressed into disks with KBr in a conventional 13-mm die. The coarser powders were pressed into films at 8 to 10 ton-force (71 to 89 kN) between stainless steel pellets in a standard 13-mm die for approximately 1 min, as described by Curry et al. [15]. For pelleted samples, a thin shaving was taken using a scalpel blade and flattened in the press as described above.

Bumper bar samples—These were prepared in the same way as the pelleted standards.

Measurement of Spectra—Films were placed in position on a specially prepared holder consisting of a metal plate the size of a microscope slide with four holes 4 mm in diameter

along its length. Four samples could be accommodated on the holder. Each film was supported by an adhesive tape bridge which was positioned across a hole. The metal plate was then placed on the microscope stage for measurement.

Pyrolysis Gas Chromatography

Instrumentation—A Pye Unicam Curie-point pyrolyzer was used with a Varian 3400 capillary gas chromatograph. The system was configured as shown in Fig. 1. The pyrolysis head was positioned above the injector nut of the injection port. A glass insert 11 cm long and 2-mm internal diameter (id) was positioned inside the pyrolysis head. The insert traversed the length of the head from below the septum of the pyrolysis head to the injector nut septum. The capillary column was passed through the injector nut septum to a position inside the pyrolysis head 1.4 cm above the thread of the injection port.

For pyrolysis, a disposable glass liner 10.5 cm in length was placed inside the glass insert with approximately 1 cm of the capillary column protruding into the liner. A wire loaded with sample was inserted into the disposable liner with the sample positioned at 5.2 cm below the septum of the pyrolysis head. The disposable liner was used to prevent contamination of the glass insert.

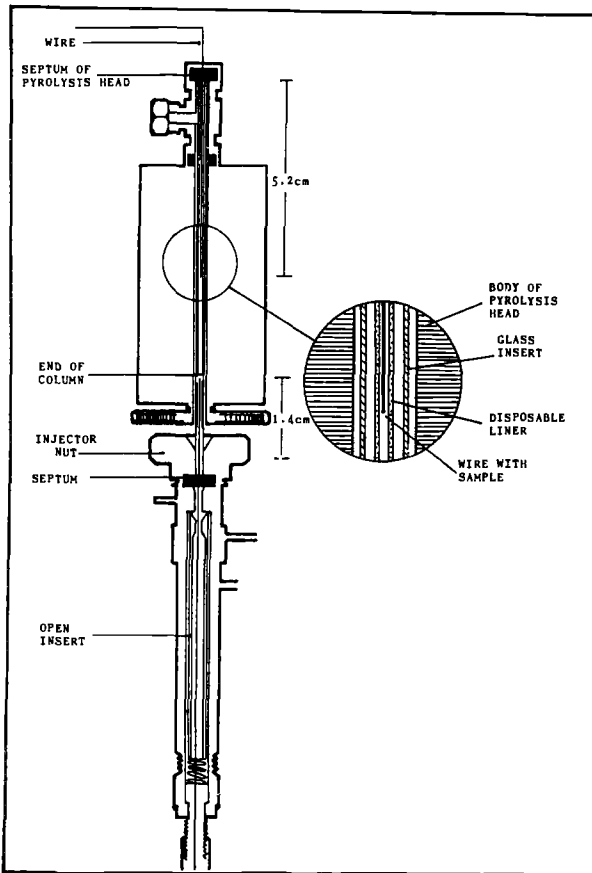


FIG. 1—Diagram of the interface between the Curie-point pyrolysis head and the capillary injector.

With this configuration and operation in the splitless mode, efficient transfer of pyrolyzate and, consequently, a high degree of sensitivity was attained. A summary of the instrumental parameters is given in Table 3.

Sample Preparation—Standards from the polymer reference collection were prepared for analysis using the following technique. One end of a Curie-point wire, approximately 10 mm in length, was flattened by placing it between two steel blocks and applying a pressure of approximately 1 ton-force (9 kN) for 5 s using an infrared (IR) press.

The flattened wire and a disposable glass liner were cleaned by heating in a bunsen flame before use. A loop was formed in the flattened end of the wire using forceps. A thin shaving of the plastic sample was taken using a scalpel blade, and a piece of the shaving approximately 0.25 mm² in area was then clamped into position in the wire loop by applying pressure with the base of a scalpel handle. The loaded wire was then placed in a disposable glass liner and the distance of 5.2 cm from the position of the sample to the end of the liner was measured and marked by bending the wire over (see Fig. 1). The remaining exposed wire was then passed through the septum of the cap of the pyrolysis head and the entire assembly inserted into the head.

Energy Dispersive X-Ray Microanalysis

Instrumentation—A Hitachi S-450 Scanning Electron Microscope and a Kevex Microanalyst 7500 energy dispersive X-ray analysis system were used. Spectra were generated at an accelerating voltage of 20 kV with a sample area of approximately 2 mm² counted for 100 s live time.

Sample Preparation—Pieces of the bumper bar samples were embedded in resin briquettes and polished using 200-grade silicon carbide paper followed by diamond abrasive paste. The polished briquette was coated with carbon by vacuum deposition.

TABLE 3—*Instrumental parameters for PGC.*

PYROLYSIS CONDITIONS	
Pye Unicam Curie-point pyrolyzer	
Pyrolysis wire: Fe with Curie-point of 770°C	
Pyrolysis time: 10 s	
Sample position on wire: 5.2 cm	
Disposable glass liner: 10.5 cm	
Sample size: approximately 0.25 mm ²	
GAS CHROMATOGRAPH CONDITIONS	
Varian 3400 GC	
Column: 25-m BP1, 330- μ m id, 0.25- μ m film thickness	
Oven: 40°C (1 min) \rightarrow 250°C (15 min) at 15°C/min; 30-min run time	
Injection port: 220°C	detector: FID, 300°C
Inlet mode: splitless	insert: open
Carrier gas: Nitrogen; 19-cm/s linear flow velocity	
DATA HANDLING	
COMPAQ DESKPRO personal computer	
ADI PX 22 color monitor	
DAPA software (data acquisition plotting and analysis, DAPA Scientific Software, P.O. Box 58, Kalamunda, Western Australia, 6076)	
LDC/Milton Roy plotter	
Display conditions: attenuation 25; chart speed 10 mm/min	

Results and Discussion

Microscopic Examination

On the basis of color, texture and resilience, four categories of sample were identified in the collection. These results are summarized in Table 4.

In terms of broad color description, approximately two thirds of the samples were black, with the remainder gray. Texturally, the samples were either solid or foamed with the degree of rigidity of the foamed samples varying from slightly flexible to rigid. Approximately one third of the samples was foamed.

Cross-sectional examination clearly distinguished the foamed samples from solid samples. However, when viewing only the outer skin of the bumper samples, it was not possible to make this distinction. It is likely that the type of sample generally submitted in forensic science casework, normally smeared material, will not provide sufficient microscopic detail to allow identification of the sample as from either a foamed or a solid plastic.

Infra-Red Spectroscopy

A total of eight different classes of polymer were identified in the bumper collection. A summary of the results of the FTIR analyses is given in Table 5.

The nine samples identified as PP/EPDM showed considerable variation in the relative intensity of the band at 1016 cm^{-1} . However, a similar variation was observed between different films prepared from the same sample. Variation in film thickness arising from the sample preparation technique was a possible cause of this lack of reproducibility [16]. Use of a diamond cell may solve this problem but one was not available for this work. Thus the band at 1016 cm^{-1} could not be used as a basis for discrimination between PP/EPDM samples.

Eight of the bumper bar samples were identified by FTIR as polyurethane in composition. The spectra showed the characteristics of polyester urethanes with strong ab-

TABLE 4—*Results of microscopic examinations.*

Color	Texture/Resilience	Sample
Black	solid	1, 7, 20, 23, 24, 28, 29, 35, 40, 42, 66
Gray	solid	2, 4, 6, 16, 22, 34, 61, 67
Black	foam, flexible	13, 76
Black	foam, rigid	21, 25, 26, 73, 77, 78, 85

TABLE 5—*Summary of FTIR results.*

Composition	Sample
PP/EPDM	1, 2, 7, 23, 28, 40, 61, 66, 67
ABS	4
PC/PBT	6
PUR	13, 21, 25, 73, 76, 77, 78, 85
PC	16
PP	20, 22, 29, 34, 42
PA	24
PES	26, 35

sorbances at approximately 3300, 1735, 1695, 1540, and 1220 cm^{-1} . The spectra were not categorized further into subclasses on the basis of the FTIR results.

The remaining samples fell into six groups: five PP samples, two PES samples, one ABS sample, one PA sample, and one sample which was a mixture of PC and PBT. Neither the polyester nor polyamide samples were identified beyond their generic polymer class.

It is interesting to compare the data in Table 5 with the manufacturer's information in Table 2. Clearly, considerable caution must be exercised in placing reliance on manufacturer's information. In four cases (Samples 4, 6, 23, and 29) the analytical findings differed markedly from the expected compositions. In two of the cases (Samples 13 and 16) there is scope for accepting the manufacturer's information given the diversity of polymeric materials used by this manufacturer in the bumper bar region of these vehicles.

Pyrolysis Gas Chromatography

The results of the PGC of the bumper bar samples are given in Table 6. Thirteen different pyrogram types were defined by PGC.

To a large extent, PGC was able to confirm the identifications made using FTIR for each of the polymer classes, the exceptions being PUR, PA, and PES. This could be rectified by establishing a more extensive library using appropriate standards. In addition, with respect to the polypropylene samples, PGC did not differentiate between PP and PP/EPDM.

The main strength of the PGC technique was its ability to provide further discrimination within those polymer categories which could only be characterized according to generic class using FTIR, specifically PUR and PES samples.

The PP samples produced two types of pyrogram. The distinction between PP Type I and PP Type II was evident in the volatile, low molecular weight fraction of the pyrolyzates. Although not a dramatic difference, it was reproducible as demonstrated by repeat analyses performed six weeks apart on Samples 1 and 2.

The pyrograms from the PUR samples were categorized into five different types. PUR Types I, II, and III showed little similarity to each other or to Types IV and V. PUR Types IV and V produced largely similar pyrograms which, although distinguishable from each other, differed only in minor components. Figure 2 illustrates the five types of PUR pyrograms obtained. Similarly, the two PES types were easily distinguished on the basis of their pyrograms.

Clearly, the technique has provided good quality data. This could perhaps be further

TABLE 6—*Summary of PGC results.*

Composition	Sample
PP Type I	1, 42, 66, 67
PP Type II	2, 7, 20, 22, 23, 28, 29, 34, 40, 61
ABS	4
PC/PBT	6
PUR Type I	13
PUR Type II	21
PUR Type III	25
PUR Type IV	73, 77, 78, 85
PUR Type V	76
PC	16
PA	24
PES Type I	26
PES Type II	35

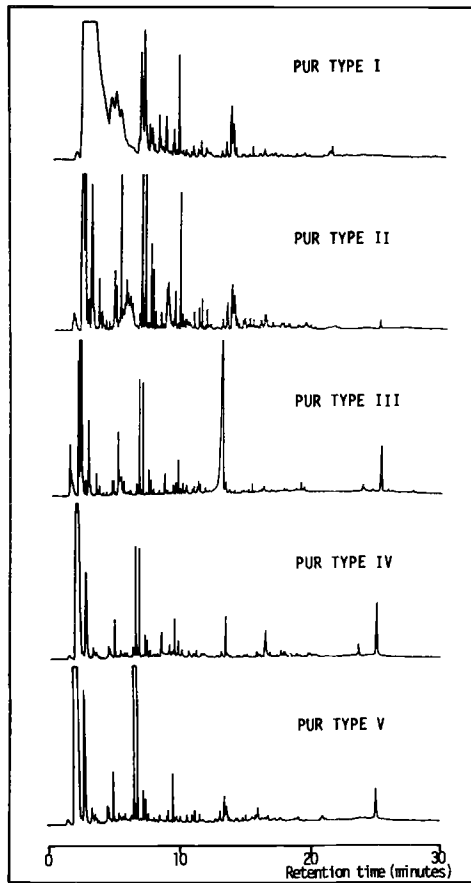


FIG. 2—The five types of PUR pyrograms obtained from the bumper bar collection.

improved by introducing a modification such as cryogenic focusing as advocated by a number of workers including Jacques and Morgan [17], Moncur et al. [18], and Wampler and Levy [19]. However, comparison of published data with those obtained during this study suggests that this approach, although worthy of exploration, is not likely to produce marked improvements.

Energy Dispersive X-Ray Microanalysis

Discrimination of the samples was made only on the basis of significant qualitative differences in the X-ray spectra produced. Initial evaluation of the spectra showed that the samples fell into three groups:

1. Those with elements present in major amounts. High count rate spectra were produced with peaks that showed a large signal relative to background (about 5000 counts per channel).
2. Those with elements present in minor amounts. The spectrum profile showed a significant bremsstrahlung compared with that of Group 1. X-ray peaks were clearly visible above the background.
3. Those with elements present in trace amounts. The spectrum profile was now dom-

inated by the brehmsstrahlung and peaks were visible above the background but significantly smaller than in Group 2.

Further inspection of the spectra within each group showed obvious qualitative differences in the elemental profiles. On this basis the samples could be divided into 17 categories as shown in Table 7. Thus SEM-EDX proved to be the most discriminatory of the three techniques used. Figure 3 is an illustration of the differences in elemental profiles that were obtained from three PP/EPDM samples which had not been discriminated by FTIR or PGC.

The discriminatory value of the technique is, however, very much dependent on sample preparation and presentation to the instrument. Differences in relative amounts of elements detected were noted when surface-mounted samples were compared with polished samples in briquettes, particularly for the foamed Samples 21 and 26. The nonideal nature of casework samples may reduce the degree of discrimination achievable using SEM-EDX unless care is taken to present them as polished samples.

Comparison of FTIR, PGC, and SEM-EDX

In comparing the value of the three techniques of FTIR, PGC, and SEM-EDX for the analysis of plastic bumper bar samples, it is important to consider two aspects. First, the type of information the technique provides and, second, the degree of discrimination the technique is capable of achieving. With respect to the latter, clearly any conclusions drawn are constrained to some extent by the size of the collection examined, although an indication of the usefulness of the technique is likely to be apparent.

FTIR proved to be a useful technique for characterizing the bumper bar samples

TABLE 7—Summary of SEM-EDX results.

Elements Present ^a	Sample
GROUP 1	
Si Al	7, 40
Si Ti	16
Si Mg	61
Cl Ca Al	26
Cl Ca	35
GROUP 2	
Al Si Ti	2, 22
Al Si	20
Al Si Cl	21, 24, 29, 73, 76, 78
Al Si Mg	23
Al Si Cl Ti	34
Al Cl Si	25
Al Ti Mg	67
Ti	4, 6
GROUP 3	
Si Mg	1, 28
Si Al Cl	13
Si Al	66
negligible	42, 77, 85

^aElements listed in descending order and restricted to those necessary to provide discrimination.

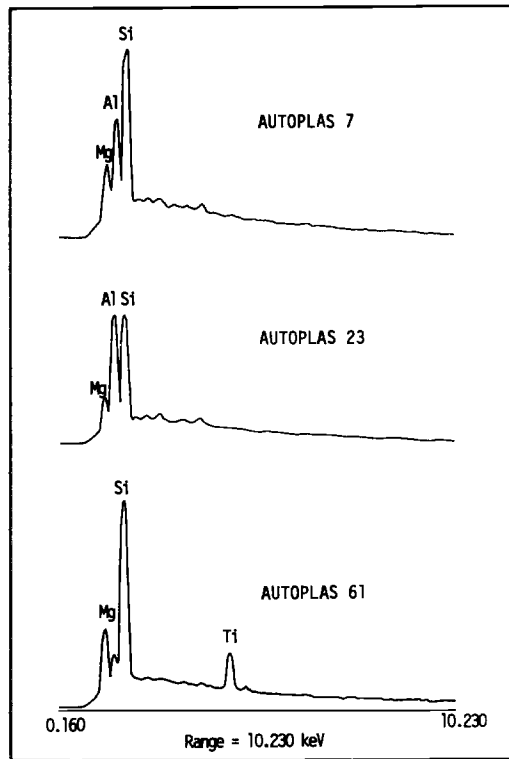


FIG. 3—A selection of elemental profiles from PP/EPDM samples in the bumper bar collection.

according to polymer class but did not provide good discrimination within a polymer class. The 28 samples in the collection were divided into 8 categories by FTIR.

The value of PGC as a technique for identification of the polymer class was very much dependent on the extent of the available reference collection of pyrograms. Where such pyrograms were available, reliable identifications could be made. The main advantage of the technique, however, lay in its ability to provide good discrimination between samples within a polymer class, particularly the polyurethanes and the polyesters. PGC divided the 28 samples of the collection into 13 categories.

SEM-EDX can provide information on the identity of fillers and pigments but does not identify plastics per se. However, it was the most discriminatory of the 3 techniques resulting in 17 categories from the 28 bumper bar samples.

The capability of identifying an unknown sample with a high degree of discrimination is a prerequisite for techniques used in forensic science casework. To generate this type of information it is often necessary to apply more than one technique to the analysis. Table 8 presents a summary of the discrimination achieved within the bumper bar collection using combinations of the three techniques of FTIR, PGC, and SEM-EDX.

Using the combination of the three techniques, three pairs of samples could not be distinguished: 7 and 40, 73 and 78, and 77 and 85.

Referring to Table 2 it can be seen that the two Samples 7 and 40 were from two different models by two different manufacturers and produced two years apart. Samples 73, 77, 78, and 85 were all obtained from Ford Australia and said to have been used for all Ford Falcon XF models over the period of manufacture from 1984 to 1987, with

TABLE 8—Comparison of the discrimination achieved using FTIR, PGC, and SEM-EDX.

Technique	Number of Categories Identified
FTIR	8
PGC	13
SEM-EDX	17
FTIR + PGC	15
FTIR + SEM-EDX	23
PGC + SEM-EDX	24
FTIR + PGC + SEM-EDX	25

Samples 73 and 77 from front bumper bars and Samples 78 and 85 from rear bumper bars.

Conclusions

The results reported in this paper show that the combination of SEM-EDX with either PGC or FTIR offers a high degree of discrimination between bumper bar samples from different vehicles. However, the combination of the three techniques of FTIR, PGC, and SEM-EDX can be recommended as a means of providing reliable identification of polymers used in bumper bar manufacture and a high degree of discrimination between samples from different vehicles. These findings are in agreement with similar reported studies [8,20–22].

The results also indicated that analysis of a fragment of bumper bar plastic was unlikely to provide information that would allow identification of a specific make or model of vehicle. Thus the analytical profiles would not be useful as an investigative aid and therefore the establishment of a data base for this purpose is not warranted.

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