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Megan Zellner,[1] M.S. and Lawrence Quarino,[1] Ph.D.

Differentiation of Twenty-One Glitter Lip-Glosses by Pyrolysis Gas Chromatography/Mass Spectroscopy*

ABSTRACT: Differentiation of 21 glitter lip-glosses from seven manufacturers was attempted by pyrolysis gas chromatography/mass spectroscopy. Samples were pyrolyzed on a ribbon probe at 800°C for 20 sec and analyzed with an Agilent® 6890N Network GC System and Agilent® 5973 Network Mass Selective Detector with MSD Productivity ChemStation® Data Analysis software. The total ion chromatograms obtained were examined and differences in the presence or absence of certain chromatographic peaks corresponding to certain pyrolysis products (e.g., styrene, cyclohexane) noted. In cases where the total ion chromatograms between lip-glosses were similar, select ion profiling was performed. Of the 21 lip-glosses, 15 were differentiated by either the total ion chromatograms alone or through select ion profiling. Considering that lip-glosses are typically worn by young women (who are disproportionately victims of sexual assault), this study offers the potential of being able to provide investigative leads in sexual assault investigations with evidentiary samples of this kind.

KEYWORDS: forensic science, lip-glosses, pyrolysis, gas chromatography, select ion profiling, mass spectroscopy

Commercially available glitter lip-glosses are a growing trend in the makeup industry increasing the likelihood of finding this type of material on physical evidence taken from crime scenes. Past research in the area of lip makeup and cosmetics has utilized many widely accepted techniques, although many of these techniques have various limitations such as sample size requirements and background interference.

Lip cosmetics typically contain mixtures of a variety of components including inorganic pigments, oils, organic dyes, and waxes (1). Traditional analytical techniques include thin layer chromatography (2) and a variety of instrumental techniques. Instrumental methods for the analysis of lip cosmetics include visible-absorption spectrophotometry and high-performance liquid chromatography for the analysis of color or dye components (3,4), gas chromatography (GC), and GC/mass spectrometry (GC/MS) for the characterization of waxes (5,6), and scanning electron microscopy (SEM) interfaced with X-ray fluorescence and X-ray energy dispersive spectroscopy (EDX) for the examination of elemental compositions of inorganic pigments (3,5). In addition, Gordon and Coulson (7) used Fourier transform infrared spectroscopy (FTIR), GC-flame ionization detector, and SEM-EDX to obtain discrimination powers of 98.3%, 93.8%, and 82.0%, respectively, on 53 different cosmetic foundation samples. A combination of all three methods produced a discrimination power of 99.7%. Salvador et al. (8) showed that characterization of glycolic and lactic acids in cosmetic products using FTIR could greatly increase the ability to differentiate cosmetics. Small silicon carbide disks have been utilized to perform diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) measurements on relatively small quantity samples of forensic interest (9). Preliminary results have indicated that this sampling technique is a simple and rapid method for obtaining good quality IR spectra for a wide range of samples including adhesives, correction fluids, paint, synthetic rubber, and lipsticks. However, the lipsticks tested were not as easily differentiated by DRIFTS due to their similar composition and specular reflection.

This study attempted to determine if the utilization of pyrolysis-GC/MS (Py-GC/MS) could discriminate between a small set of lip-gloss samples offering the potential of a cost-effective, efficient, and sensitive alternative to forensic laboratories for the analysis of this type of evidence.

Materials and Methods

Lip-Glosses

Twenty-one different lip-glosses from seven common manufacturers: Bonne Bell® (Bonne Bell, Lakewood, OH), Caboodles® (Plano Molding Company, Plano, IL), L'Oreal® (L'Oreal International, Clichy, France), Maybelline® (L'Oreal USA, New York, NY), NYC® (Coty US LLC, Morris Plains, NJ), Revlon® (Revlon, Inc., New York, NY), and Smackers® (Bonne Bell) were examined. The name of each lip-gloss is provided in Table 1. The Caboodles® Glim Glam Deluxe Duo, Caboodles® Yum Yum, and Revlon® Colorstay Overtime Sheer contained both a topcoat and a basecoat which were examined separately. Samples from an additional lot of 10 of the lip-glosses were tested for interlot variation and different colors from five of the lip-glosses were also tested to see if color affected results.

Instrumentation

An Agilent® 6890N Network GC System and Agilent® 5973 Network Mass Selective Detector with MSD Productivity ChemStation® Data Analysis software and a 30 m HP/5 ms column

[1]Forensic Science Program, Cedar Crest College, 100 College Drive, Allentown, PA 18104.

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TABLE 1—*List of lip-glosses utilized in study.*

Manufacturer	Lip-Gloss Name
Bonne Bell®	Clic-It
Bonne Bell®	Lipfashion
Caboodles®	Flash Glitter Lipgloss
Caboodles®	Glim Glam Deluxe Duo
Caboodles®	Yum Yum
L'Oreal®	Colour Juice
L'Oreal®	Glam Shine
Maybelline®	Shiny-licious
Maybelline®	Wet Shine Diamonds
Maybelline®	Lip Polish
NYC®	Kiss Gloss
NYC®	Roll-On Glitter
NYC®	Liquid Lip Shine
Revlon®	Skinlights
Revlon®	Super Lustrous
Revlon®	Colorstay Overtime Sheer
Smackers®	Whirly Shimmer Gloss
Smackers®	Lip Sparkler
Smackers®	Roll-On Shimmer Gloss
Smackers®	Lip Frosting
Smackers®	Lip Smacker

(0.25 mm ID with a 0.25 μm film thickness) (Agilent Technologies, Santa Clara, CA) were utilized in this study. The NIST02 database library was utilized to determine the structure of all compounds listed and scan parameters for the mass spectrometer began at 40 amu and ended at 450 amu. Prior to analysis, samples were mixed on a glass slide to ensure homogeneity. Samples were pyrolyzed on a ribbon probe at 800°C for 20 sec in a CDS Analytical Pyroprobe 5000 Series (software version 1.6.2) with a 1500 valved interface unit (CDS Analytical, Oxford, PA) maintained at 300°C.

Preparation of Instrument

A system verification of the GC/MS was performed at the beginning of each day prior to any sample runs by completing an autotune with perfluorotributylamine and tune evaluation. The pyrolysis interface unit was then connected and allowed to equilibrate to the appropriate temperature. Once the interface reached 300°C, a system blank was run for 50 min at an inlet temperature of 280°C (16.412 psi, split ratio of 30:1, helium as carrier gas). The oven temperature was initially held for 2 min at 40°C and then ramped 10°C/min to a maximum temperature of 295°C and held for 22.5 min. Once it was demonstrated that the column was clean, samples were analyzed.

Py-GC/MS of Lip-Glosses

A small amount of the lip-gloss sample, *c.* 2 mm in diameter, was placed on the ribbon probe, inserted into the interface accessory unit, and allowed to equilibrate for a period of 30 sec at which point the run was started. The probe was then kept in the accessory for another 60 sec while a probe temperature of 800°C was maintained for 20 sec. After the 60 sec, the probe was removed from the interface unit. The sample was then allowed to pass through the column for the duration of the run (50 min) before being analyzed. Triplicate runs were performed on each of the lip-glosses on different days to demonstrate reproducibility for all aspects of the study.

Results

The chromatograms obtained from each of the 21 lip-glosses (including the top and base coats for three of the samples) were

TABLE 2—*Major and descriptive pyrolysis chromatographic peaks (in minutes) from lip-glosses (peaks listed in the typical order of intensity).*

Lip-Gloss (Manufacturer's Name—Product Name)	Major Peaks (Component Identification)
Bonne Bell®—Clic-It	5.14 (styrene), 7.59 (cyclohexane), 2.27 (pentadiene)
Bonne Bell®—Lipfashion	5.14 (styrene), 14.03 (octadecene)
Caboodles®—Flashglitter	7.59 (cyclohexane), 2.27 (pentadiene)
Caboodles®—Glim Gam Top coat	7.59 (cyclohexane), 5.14 (styrene), 2.27 (pentadiene)
Caboodles®—Glim Gam Base coat	7.59 (cyclohexane), 5.14 (styrene), 2.27 (pentadiene)
Caboodles®—Yum Yum Top coat	7.59 (cyclohexane), 2.27 (pentadiene)
Caboodles®—Yum Yum Base coat	7.59 (cyclohexane), 2.27 (pentadiene)
L'Oreal®—Colour Juice	7.59 (cyclohexane), 10.09 (dodecene), 6.87 (decene)
L'Oreal®—Glam Shine	21.27 (octadecanoic acid), 14.03 (octadecene), 10.09 (dodecene), 6.87 (decene)
Maybelline®—Shiny-licious	7.59 (cyclohexane), 10.09 (dodecene), 2.27 (pentadiene), 21.27 (octadecanoic acid), 6.87 (decene)
Maybelline®—Wet Shine Diamonds	10.09 (dodecene), 21.27 (octadecanoic acid), 14.03 (octadecene), 6.87 (decene)
Maybelline®—Lip Polish	7.59 (cyclohexane), 2.27 (pentadiene)
NYC®—Kiss Gloss	7.59 (cyclohexane)
NYC®—Roll On	7.59 (cyclohexane), 21.27 (octadecanoic acid), 2.27 (pentadiene)
NYC®—Lip Shine	14.03 (octadecene), 7.59 (cyclohexane)
Revlon®—Skinlights	5.14 (styrene), 7.59 (cyclohexane), 2.27 (pentadiene), 19.03 (cycloeicosine)
Revlon®—SuperLustrous	7.59 (cyclohexane)
Revlon®—Colorstay Overtime Sheer—top coat	6.90 (cyclooctane), 3.43 (octane)
Revlon®—Colorstay Overtime Sheer—base coat	4.00 (cyclotrisiloxane), 7.03 (cyclotetrasiloxane)
Smackers®—Whirly Shimmer Gloss	7.59 (cyclohexane), 2.27 (pentadiene)
Smackers®—Lip Sparkler	19.10 (cycloeicosine), 7.59 (cyclohexane), 2.27 (pentadiene), 22.03 (octadecene)
Smackers®—Roll-On Shimmer Gloss	5.14 (styrene)
Smackers®—Lip Frosting	5.14 (styrene)
Smackers®—Lip Smacker	5.35 (heptanol), 13.93 (undecyleric acid), 19.33 (hexadecanoic acid)

analyzed using the ChemStation® software included on the Agilent® GC/MS to determine if differentiation of the lip-glosses was attainable. The GC total ion chromatograms (TIC) obtained were examined and differences in the general pattern and the major and distinctive peaks present were determined. The largest and most descriptive peaks consistently present throughout all three replicate runs for each lip-gloss are tabulated in Table 2. Identifying the largest peaks in the TIC provided an objective basis for grouping or differentiation of the lip-glosses in this study. In cases where the TIC between samples was similar, select ion profiling was performed to differentiate samples.

Lip-Glosses with Distinctive TIC

Of the 21 lip-glosses, six had individually distinctive pyrolysis GC patterns (Bonne Bell® Lipfashion, NYC® Lip Shine, Revlon® Skinlights, Revlon® Colorstay Overtime Sheer, Smackers® Lip Sparkler, and Smackers® Lip Smacker). Each is listed in italics in Table 2. The TIC from each of the six (including both the top and base coats of the Revlon® Colorstay Overtime Sheer) did not follow any particular pattern and could be easily differentiated from the other lip-glosses in the study. Total ion chromatograms from the Bonne Bell® Lipfashion and NYC® Lip Shine lip-glosses are found in Fig. 1.

Two sets of two lip-glosses showed distinctive TIC patterns but could not be differentiated from each other (L'Oreal® Colour Juice and Maybelline® Shiny-licious; L'Oreal® Glam Shine and Maybelline® Wet Shine Diamonds). Lip-glosses from each set similarly could not be differentiated from each other using the ion profiling method described below. Total ion chromatograms from the two Maybelline products are found in Fig. 2.

Ion Profiling

The remaining 15 lip-glosses could not be individually differentiated. Most of these lip-glosses showed a repeating pattern of four peaks consistent with the pyrolyzed TIC of a polymer. The 15 remaining lip-glosses were grouped into separate classes by the presence or absence of dominant peaks containing styrene or cyclohexane.

In all 15 lip-glosses, ion profiling was performed in an attempt to differentiate the lip-glosses within each group. Ions 104 and 69 were chosen to profile. Ion 104 was the molecular ion peak for styrene present in the primary styrene peak found at 5.14 min. It was also found in many other peaks examined as the result of the fragmentation of other compounds. For instance, ion 104 can result from fragmentation of the compound benzimidazole-2-carboxaldehyde 1-methyl oxime. Structures for styrene and benzimidazole-2-carboxaldehyde, 1-methyl, oxime as well as

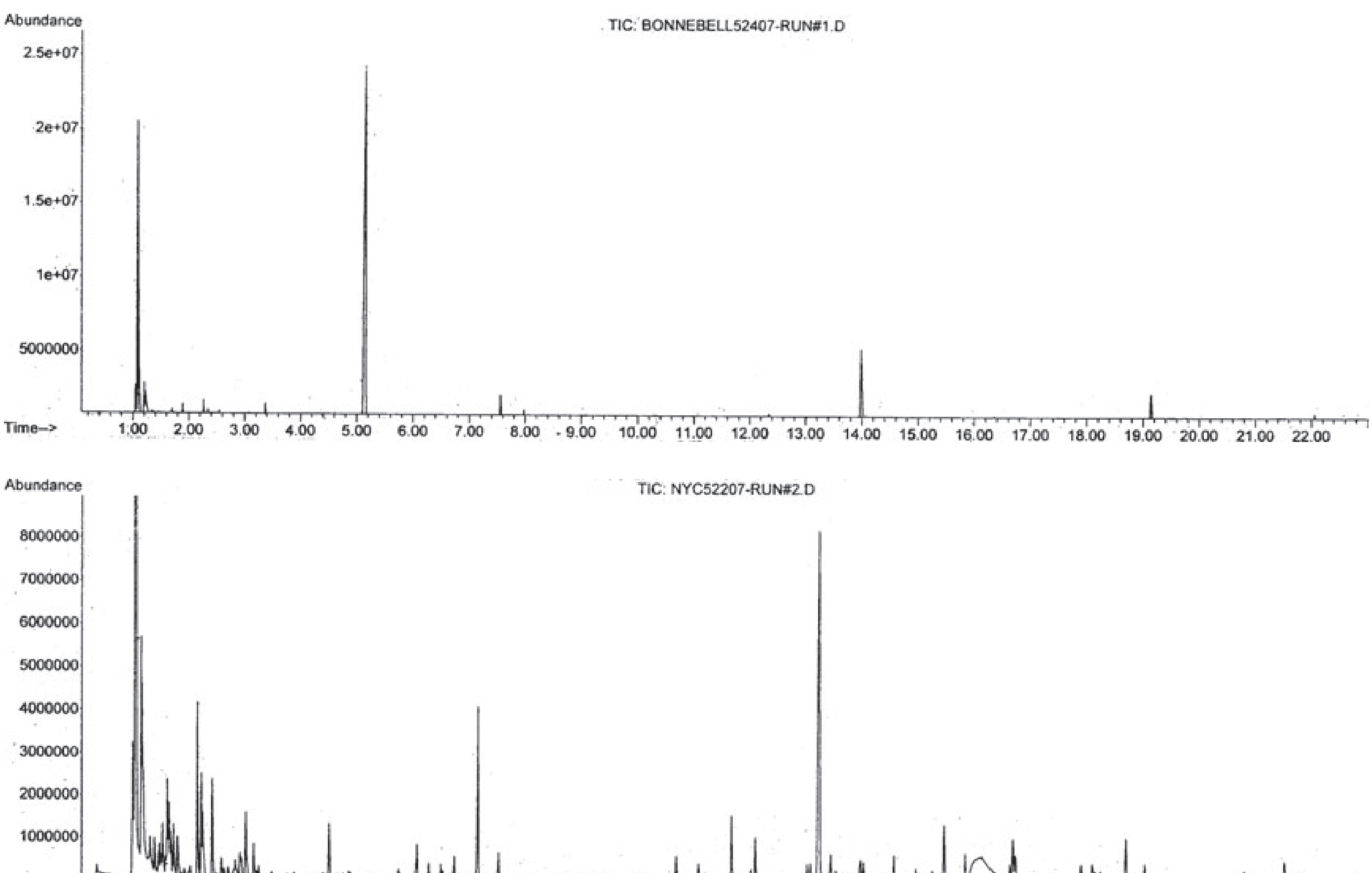

FIG. 1—*Total ion chromatograms from Bonne Bell® Lipfashion (A) and NYC® Lip Shine lip-glosses (B). The large peak around 1.0 min represents dead volume gases in the column. Both show an octadecene peak at 14.03 min with the Bonne Bell® Lipfashion showing a styrene peak at 5.14 min and the NYC® Lip Shine lip-gloss showing a cyclohexane peak at 7.59 min.*

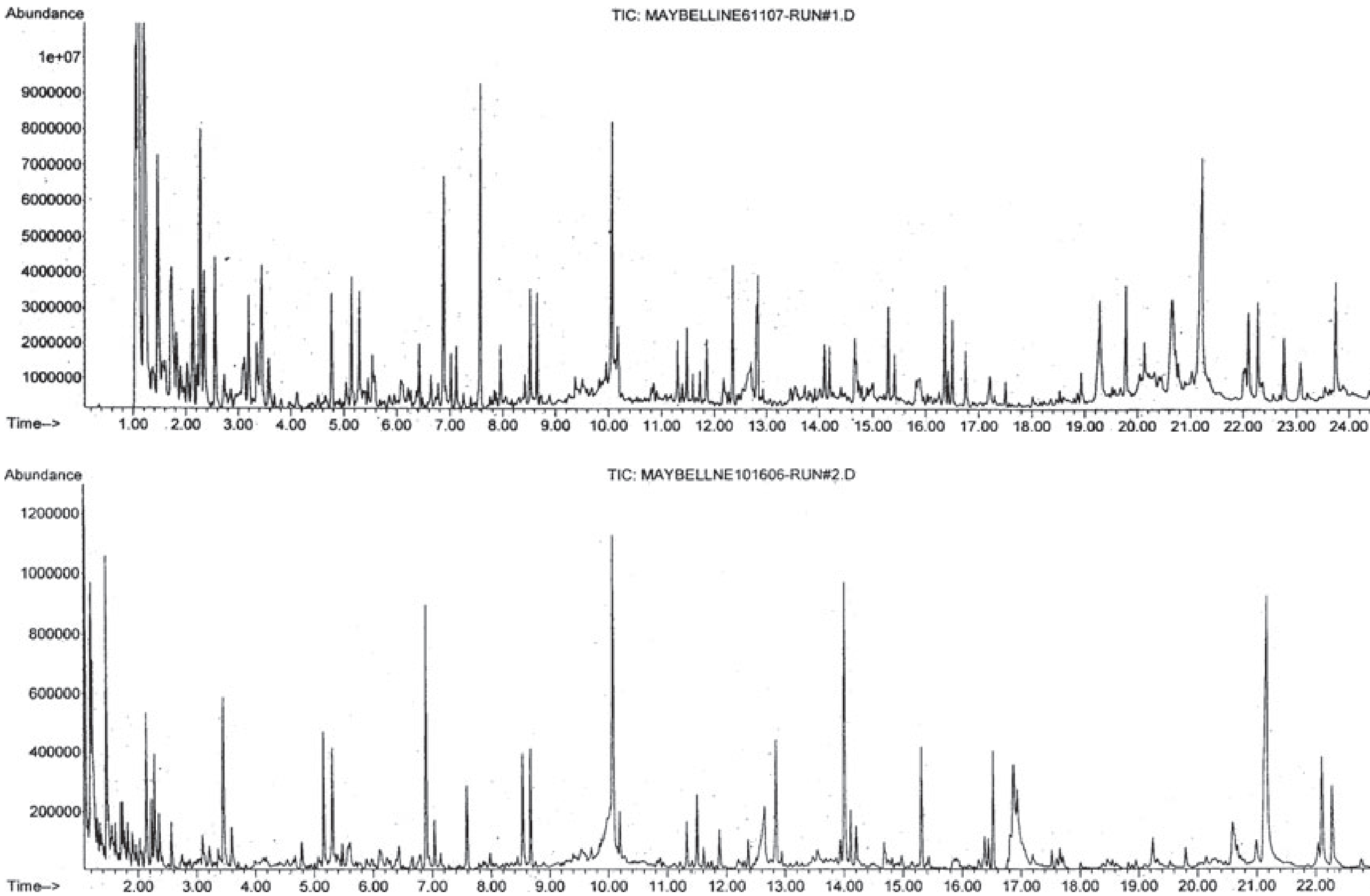

FIG. 2—*Total ion chromatograms for Maybelline® Shiny-licious (A, analytically indistinguishable from L'Oreal® Colour Juice) and Maybelline® Wet Shine Diamonds (B, analytically indistinguishable from L'Oreal® Glam Shine). The large peak around 1.0 min represents dead volume gases in the column.*

respective fragmentation pattern producing ion 104 could be found in Fig. 3.

Ion 69 was chosen as it commonly arose due to fragmentation of the monomer methyl methacrylate. The structure and fragmentation of methyl methacrylate producing ion 69 is shown in Fig. 4.

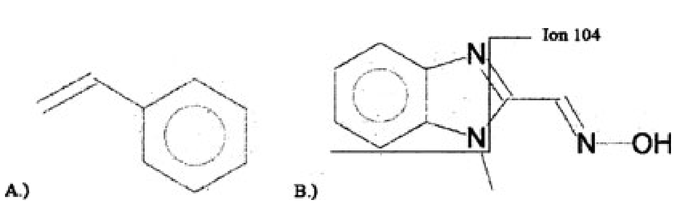

FIG. 3—*(A) Styrene (B) Benzimidazole-2-carboxaldehyde 1-methyl oxime and fragmentation pattern producing ion 104.*

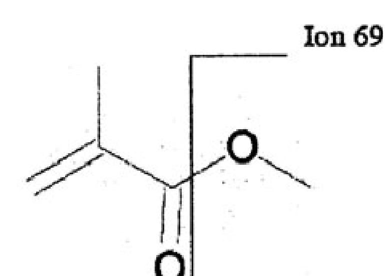

FIG. 4—*Methyl methacrylate and fragmentation pattern producing ion 69.*

In cases where differentiation could not be achieved by profiling ion 104 or 69, a third ion (ion 106) was profiled. Ion 106 was found in small abundance in many examined peaks and could arise from the fragmentation of phenyl and pyridyl groups (10).

Ion profiling results listed peaks containing ions 104, 69, and 106 for those lip-glosses containing dominant styrene and cyclohexane peaks are found in Table 3 and an example TIC of a lip-gloss in this class can be found in Fig. 5 (Caboodles® Glim Glam, base coat). Based on the methodology employed in this study, the two listed lip-glosses (Bonne Bell® Clic-It, Caboodles® Glim Glam) could not be differentiated. Furthermore, the top and base coats from the Caboodles® Glim Glam were analytically indistinguishable.

Ion profiling results listed peaks containing ions 104, 69, and 106 for those lip-glosses containing only a dominant cyclohexane peak are found in Table 4 and an example TIC of a lip-gloss in this class (Revlon® SuperLustrous) could be found in Fig. 6 (styrene peaks may still be found in some of the lip-glosses in this

TABLE 3—*Ion profiling of similar pattern lip-glosses with dominant styrene and cyclohexane peaks.*

Lip-Gloss (Manufacturer's Name—Product Name)	Ion 104 Peaks	Ion 69 Peaks	Ion 106 Peaks
Bonne Bell®—Clic-It	5.14	2.54, 7.59	4.76
Caboodles®—Glim Glam, top coat	5.14	2.54, 7.59	4.76
Caboodles®—Glim Glam, base coat	5.14	2.54, 7.59	4.76

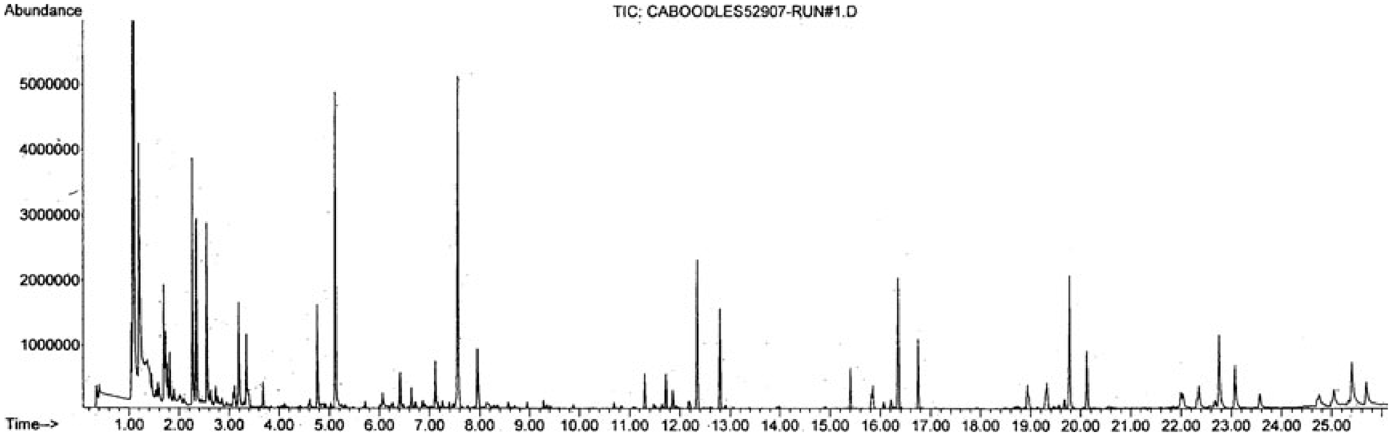

FIG. 5—*Total ion chromatogram from Caboodles® Glim Glam base coat. Lip-glosses with this pattern show a styrene peak at 5.14 min and cyclohexane peak at 7.59 min. The large peak around 1.0 min represents dead volume gases in the column.*

TABLE 4—*Ion profiling of similar pattern lip-glosses with dominant cyclohexane peak and without dominant styrene peak.*

Lip-Gloss (Manufacturer's Name—Product Name)	Ion 104 Peaks	Ion 69 Peaks	Ion 106 Peaks
Caboodles® Flashglitter	15.00, 5.14	2.39, 7.59	9.30
Caboodles® Yum Yum—top coat	5.14, 15.00	2.39, 7.59	4.76, 9.25
Caboodles® Yum Yum—base coat	5.14	7.59, 2.53, 2.39	NA
Maybelline®—Lip Polish	15.00, 5.14, 11.94	2.39, 7.59	NA
NYC®—Kiss Gloss	11.86	7.59, 2.59	NA
NYC®—Roll-On	5.14, 6.75	7.59	NA
Revlon®—Super Lustrous	No Peaks	7.59, 2.59, 2.39	NA
Smackers®—Whirly Shimmer Gloss	15.00, 5.14	2.39, 7.59, 19.16	NA

NA, not applicable.

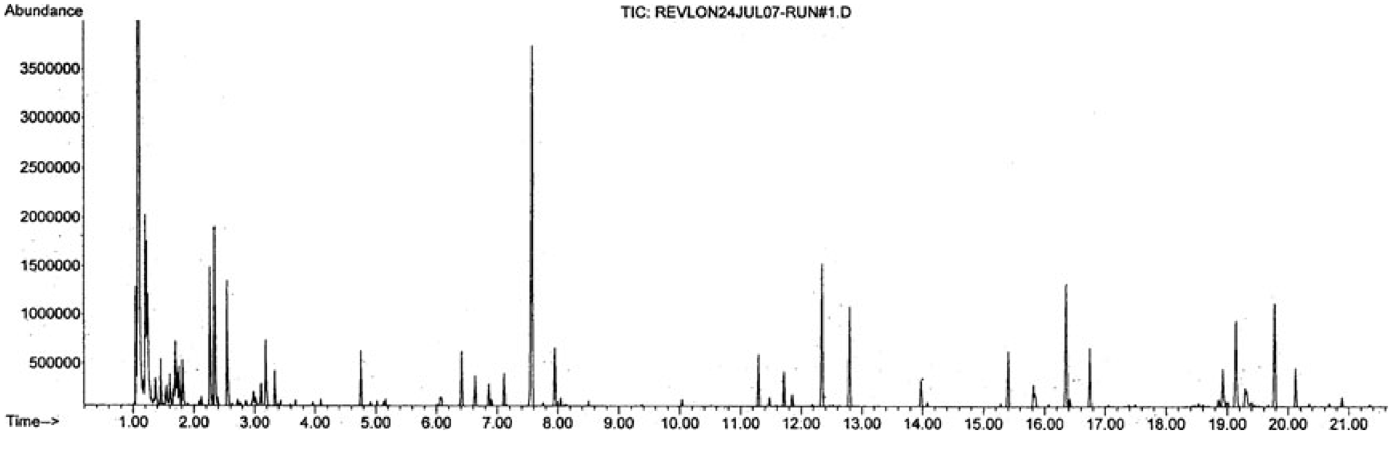

FIG. 6—*Total ion chromatogram from Revlon® Super Lustrous lip-gloss. Lip-glosses with this pattern show a large cyclohexane peak at 7.59 min without a dominant styrene peak. The large peak around 1.0 min represents dead volume gases in the column.*

class but were not dominant in any of the triplicate runs). Of the eight samples (seven lip-glosses, one with a top and base coat), five were differentiated on the basis of ion 104 alone. Ion 69 was able to differentiate one of the other three (Smackers® Whirly Shimmer Gloss), while ion 106 could differentiate each of the other two (Caboodles® Flashglitter, Caboodles® Yum Yum—Top Coat). Therefore, the ion profiling conducted could individually differentiate the eight lip-glosses in this group.

Ion profiling results listed peaks containing ions 104 and 69 for the two lip-glosses containing only a dominant styrene peak are found in Table 5 and an example TIC of a lip-gloss in this class can be found in Fig. 7 (Smackers® Lip Frosting). The two were differentiated on the basis of ion 69 alone. No peaks containing ion 69 were present in one of the two lip-glosses (Smackers® Roll-On Shimmer Gloss), while the second lip-gloss showed one peak (Smackers® Lip Frosting).

TABLE 5—*Ion profiling of similar pattern lip-glosses with dominant styrene peak and without cyclohexane peak.*

Lip-Gloss (Manufacturer's Name—Product Name)	Ion 104 Peaks	Ion 69 Peaks
Smackers®—Roll-On Shimmer gloss	5.14	No peaks
Smackers®—Lip Frosting	5.14	3.44

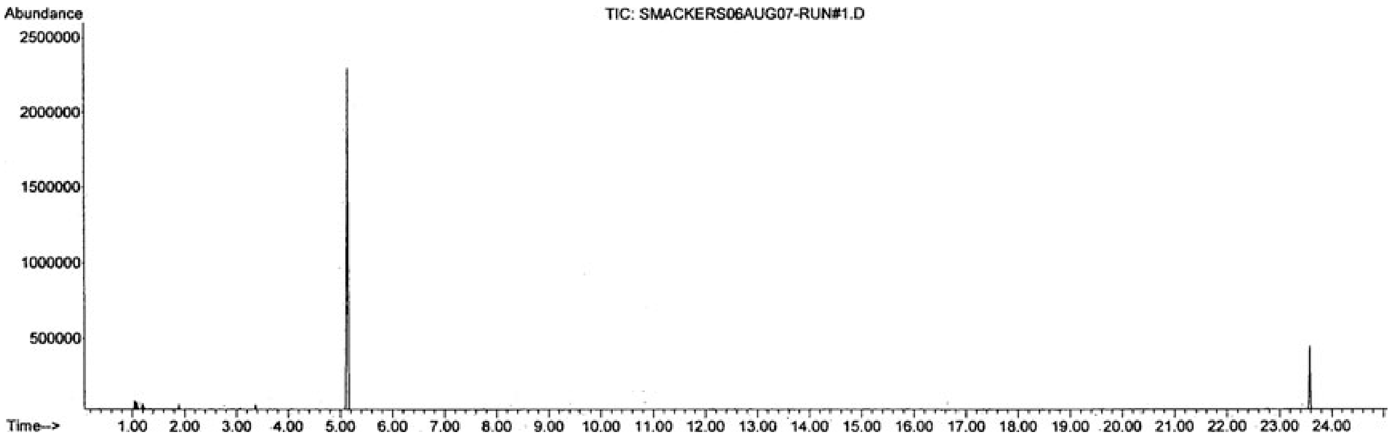

FIG. 7—*Total ion chromatogram from Smackers® Lip Frosting. Lip-glosses with this pattern show a styrene peak at 5.14 min and no cyclohexane peak.*

TABLE 6—*Summary of lip-glosses tested in color variation study.*

Lip-Gloss (Manufacturer's Name—Product Name)	Color #1	Color #2
Bonne Bell®—Clic-It	Vogue (dark pink)	Glam Mode (clear)
L'Oreal®—Colour Juice	Berry Burst (medium pink)	Sugar Coated (clear)
Maybelline®—Shiny-licious	Strawberry Tart (red)	Lolly Pink (light pink)
NYC®—Roll-On Glitter	Kiwi (green)	Strawberry (red)
Revlon®—Super Lustrous	Pink Afterglow (dark pink)	Sparkly Champagne (gold)

Lot to Lot Variation

New samples of 10 of the lip-glosses (L'Oreal® Colour Juice, Maybelline® Shiny-licious, Maybelline® Wet Shine Diamonds, Maybelline® Lip Polish, NYC® Kiss Gloss, NYC® Liquid Lip Shine, Revlon® Colorstay Overtime Sheer, Revlon® SuperLustrous, Smackers® Lip Sparkler, and Smackers® Lip Frosting) that had been used in the initial study were purchased and tested to determine whether manufacturer's formulations varied over the time of the study. These new samples were purchased almost 2 years after the initial samples. Both the top and the base coat of the Revlon® Colorstay Overtime Sheer gloss were analyzed separately.

As in the initial study, samples were run in triplicate on different days to ensure reproducibility. The TICs obtained were examined using the software provided with the GC/MS and differences in the overall TIC pattern and major and descriptive peaks present were noted.

Results showed that the TICs for both lots of each of the 10 lip-glosses were analytically indistinguishable. Therefore, it was determined that manufacturer's formulations did not vary during the time frame of this research and showed no interlot variation.

Variation Due to Color

As lip-glosses of the same product class are manufactured with different colors, the effect of the color of the lip-gloss on the chromatographic results was investigated. One new color for five of the different lip-glosses originally examined were purchased and tested. The lip-gloss and manufacturer color name are located in Table 6 with a description of the color for each gloss indicated in parenthesis.

As in the initial study, samples were run in triplicate on different days to ensure reproducibility. The TICs obtained were examined using the software provided with the GC/MS and differences in the overall TIC pattern and major and descriptive peaks present were noted.

Results for each of the new colors showed analytically indistinguishable TICs from the original total ion chromatogram obtained in the initial study. This indicates that the color of the lip-gloss is likely to have no effect on the TIC obtained.

Discussion and Conclusion

Of the 21 lip-glosses tested in this study, 15 could be differentiated by examining either the total ion chromatogram or through ion profiling. Three sets of two lip-glosses showed that they were analytically indistinguishable from each other (L'Oreal® Colour Juice and Maybelline® Shiny-licious; L'Oreal® Glam Shine and Maybelline® Wet Shine Diamonds; Bonne Bell® Clic-It and Caboodles® Glim Glam) but could be differentiated from all the other lip-glosses in the study. Therefore, Py-GC/MS showed the potential to be a valid, reproducible method for the differentiation of types of glitter lip-glosses. By including the data obtained from the lip-glosses in this study as well as other brands not discussed in this research (e.g., Covergirl®, Rimmel®, Max Factor®, and Clinique®), a database of Py-GC/MS data for lip-glosses could be produced. This database would allow scientists to conclude with a reasonable degree of scientific certainty the type of lip-gloss found at the crime scene or on a piece of physical evidence.

This potential utility of using Py-GC/MS for use with this type of evidence was further demonstrated through a small blind study. Five lip-glosses were randomly selected for analysis by Py-GC/MS from the 25 utilized in this research. All five of the lip-glosses were correctly identified using the method described. Furthermore, considering that all descriptive chromatographic peaks occurred prior to 25 min, analysis time could be cut in half making the method more suitable for use in a typical casework laboratory.

This research is significant because the lip-glosses analyzed in this study are typical of those worn by young women who are disproportionately victims of sexual assault. Through consultations with a rape crisis coordinator, an instance was discovered where a victim of a sexual assault helped to identify her attacker through the blue eye shadow she was wearing the night of the attack (Personal communication, Melodie Brooks, Rape Crisis Coordinator, Mercy Memorial Hospital System, Monroe, MI). Further development of this study could conceivably help investigators of sexual assault crimes with cosmetic-based evidence.

One potential caveat to this study is how substrate material could affect the analysis and results. In this study, all test samples came directly from the lip-gloss tube rather than from a substrate which would likely be the case at a crime scene or on a piece of physical evidence. It is conceivable that material from substrates, either chemical or biological (11), may interfere with chromatogram interpretation. Chromatograms of substrate material should be produced in these instances in order to subtract out the substrate contribution in the test sample. Furthermore, the utilization of extracted ion profiling can further increase the specificity of the test by limiting chromatograms to a set of ions consistent with lip-gloss products. In the case of possible biological interferences, validation work on the effect of microbes and skin cells should be performed.

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Additional information and reprint requests:
Lawrence Quarino, Ph.D.
Cedar Crest College
100 College Drive
Allentown, PA 18104
E-mail: laquarin@cedarcrest.edu