ANIL AGGRAWAL'S
Internet Journal of Forensic Medicine and Toxicology
The world's first online journal devoted entirely to forensic medicine and toxicology · Published 1 January and 1 July

VOL. 16, NO. 1 · JANUARY – JUNE 2015
Downloaded from www.anilaggrawal.com · ISSN 0972-8074 (Online) · https://www.anilaggrawal.com/ij/vol_016_no_001/papers/paper009.html

Separation of different components of hair cosmetic (Hairsprays) using TLC and HPTLC.

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CITE THIS ARTICLE

Kaur A, Sahota SS and Garg RK. Separation of different components of hair cosmetic (Hairsprays) using TLC and HPTLC.. *Anil Aggrawal's Internet Journal of Forensic Medicine and Toxicology* [serial online], 2015; Vol. 16, No. 1 (January – June 2015): [about 15 p]. Available from: https://www.anilaggrawal.com/ij/vol_016_no_001/papers/paper009.html

ABSTRACT

Hairspray is one of the top most selling products among cosmetics. As it is a hair product, it has a great importance from forensic point of view because hair evidence is one of the most common types of evidence encountered in criminal investigations and cosmetics (including hairspray) are used widely today. The analysis of hair with respect to its identity, source of origin and presence of any adhering material (like hair color, dyes, hair sprays or serums etc.) is very significant in different types of Forensic cases.

In the present study, an attempt has been made to study the hair spray samples (marketed) commonly used for the separation of components using thin layer chromatography and high performance thin layer chromatography based on their polarity. Eight hairspray brands were analyzed with thirty different solvent systems through TLC and by one of the best solvent system (from TLC results), through HPTLC. It is interesting to note that same solvent [Carbon tetrachloride: Cyclohexane (90: 10)], is found to be the most suitable for the separation of components of hairsprays using both the techniques. The results are clear and well separated and can be used in Forensic investigation using these techniques in order to save time and chemicals.

Keywords: Hairsprays, HPTLC, TLC, Forensic

Introduction

Chromatography is the technique for the separation of the components based on the polarity using two phase, i.e. Mobile phase (Solvent) and Stationary Phase (Silica Gel G). This technique was first reported by Russian Botanist Tswett. In 1903, he succeeded in separating leaf pigments using a solid polar stationary phase. Thin layer Chromatography and High performance thin layer chromatography are a form of planar chromatography. Thin layer chromatography is widely used in the forensic science laboraborties for the analysis of drugs[1-4], Cosmetics[5-6], inks[7-10], etc. Over the period of time thin layer chromatography has been replaced by more sensitive, accurate, fast technique i.e High performance thin layer chromatography. It is also a planar chromatography and more refined form of thin layer chromatography (TLC), it has a wide range of applications in pharmaceutical [11-15], Botany [16-18], Forensic [1-4, 7-10], Chemistry [19-21] etc. With the advancement in the field of technology, HPTLC is hypernated with other instrumental technique to know the exact structure of the compound. In the present study an attempt has been made to separate the components of different components to narrow down the search for the suspect and use the result as corroborative evidence.

Hair sprays are widely used cosmetic product in today's times. It is also known as Hair Lacquer. It contains various natural and synthetic resins in both aqueous or alcoholic solutions and a propellant. Apart from these it also contains perfumes and small amount of lanolin. [22] This composition is filled into a pressure resistant container with a spray nozzle. The spray is dispensed from a pump or aerosol spray nozzle. For propellant driven spray applications, pressurized propellant mix is added. In case of pump sprays, often creating less fine aerosols, propellants are excluded. [23] Spraying deposits a stiff layer of the polymers on the hair after the solvent evaporates. The key property that makes polymers useful as hairspray is the holding agents and it's their ability to form films upon drying. Once hair spray is applied to the hair, the liquid drops run down the hair shaft until they reach the intersection of two hair fibers. When the drops dry at this fiber intersection, they create an invisible film that bonds hairs together. [24]

Hair sprays were developed around the time of an aerosol can in the 1940s. The first hair spray used was shellac, a resinous material derived from insects to hold hair in place. It also produced a water-insoluble film that was difficult to wash out of the hair. Synthetic polymers into hair styling helped overcome this problem. The first to package it was Chase products (an aerosol manufacturer) in 1948. Historic beauty titan Helene Curtis coined the name "Hairspray" in 1950, with the release of her wildly successful product "Spray Net". By 1964, hairspray was the most successful beauty product in the country, outselling even lipstick. However the late '60s into the '70s saw a drastic decline in hairspray sales. This was a result of a few factors like the popularity of "flower child" hair-styles in that era and it was discovered that aerosol cans contained Chlorofluoro Carbons (CFCs), which were harmful to the environment—particularly the ozone layer, as well as the women using the products. Before the 1970s, hair spray contained the propellant "vinyl chloride." This is a known carcinogen linked to cancer of the liver.

Eventually, new rules and regulations came forward for the improvement of these products, and CFCs were removed from aerosol cans completely and vinyl chloride was also replaced by other propellants.

But by the 80s, Hairspray's popularity came back. Today hairsprays are formulated as flexible, medium, and maximum hold, as there is much more of a liking towards natural hair styles. [25]

The present work has been done with the aim to analyze the hairsprays present in the area or victim that can help in association of the victim or assailant in crime investigation. This is a preliminary study and not much is reported, particularly with reference to India. It is expected that it will provide significant information that will be useful in forensic investigations.

Materials and Methods

1. **Collection of samples:** Eight (commonly used) hair sprays samples of different brands were collected from different beauty parlors and salons of Punjab.
2. **Chemicals and Solvents:** The entire reagents were of Laboratory grade purchased from Merck, Mumbai. Silica Gel G was purchased from Merck, Mumbai for Thin layer chromatography. Aluminium per coated TLC plates with silica Gel 60 F254 (MERCK KGaA) of size 20×20 cm were used for HPTLC.
3. **Analytical Configuration**: HPTLC CAMAG® analytical station. It includes- (1) Applicator Linomat 5, (2) Wincat software, (3) TLC scanner 3.

Results and Discussion

In the present study, an attempt has been made to find a better solvent system for the separation of hairspray components. A total of thirty solvent systems were used to find out that with which solvent system separation is more suitable. The solvent systems used are given in Table 1. Out of these thirty solvent systems attempted eight solvents were selected as it gave maximum separation of the components of hairspray and the visualization done with the help of iodine fuming in case of thin layer chromatography and under UV (long and short) which in case of high performance thin layer chromatography. These selected eight best solvent systems are given in Table-2.

Out of the eight selected suitable solvent systems, Solvent system [Carbon tetrachloride: Cyclohexane (90: 10)] is chosen for further HPTLC analysis of the hair sprays to separate the components as it gave better separation result. Using Solvent system [carbon tetrachloride: Cyclohexane (90: 10)], Berina gave 3 spots at hRf (16,47,54), Keabeen gave 3 spots at hRf (12,17,26), Nova gave 2 spots at hRf (12,27), Nova Gold 4 spots at hRf (9,14,57,98) and Swift gave 4 spots at hRf (30,42,48,57) while Ck, Gatsby and Sora Amore gave no results.

Same Eight samples were subjected to High Performance Thin layer chromatography. Two solvent systems were selected based on the results of Thin layer chromatography i.e. Solvent system [Chloroform: Ethanol (90: 10) and Carbon tetrachloride: Cyclohexane (90: 10)]. HPTLC results obtained and visualized under UV light at 254 nm. Out of these two solvents system selected, solvent system Chloroform: Ethanol (90: 10) gave better results, separating two components of three samples out of six samples and one component of remaining five samples. Whereas, solvent system Chloroform: Ethanol (90: 10) gave no result.

Using Solvent system Carbon tetrachloride: Cyclohexane (90: 10), Berina gave only one spot with hRf at 4, CK gave two spots at 3 and 16, Gatsby gave one spot at 3, Keabeen gave one spot at 3, Nova gave two spots at 4 and 60, Nova gold gave two spots at 3 and 9, Sora amore gave two spots at 2 and 4, while Swift gave two spots at 4 and 9 (Table-3), separating all the samples of hairsprays depending upon their polarity.

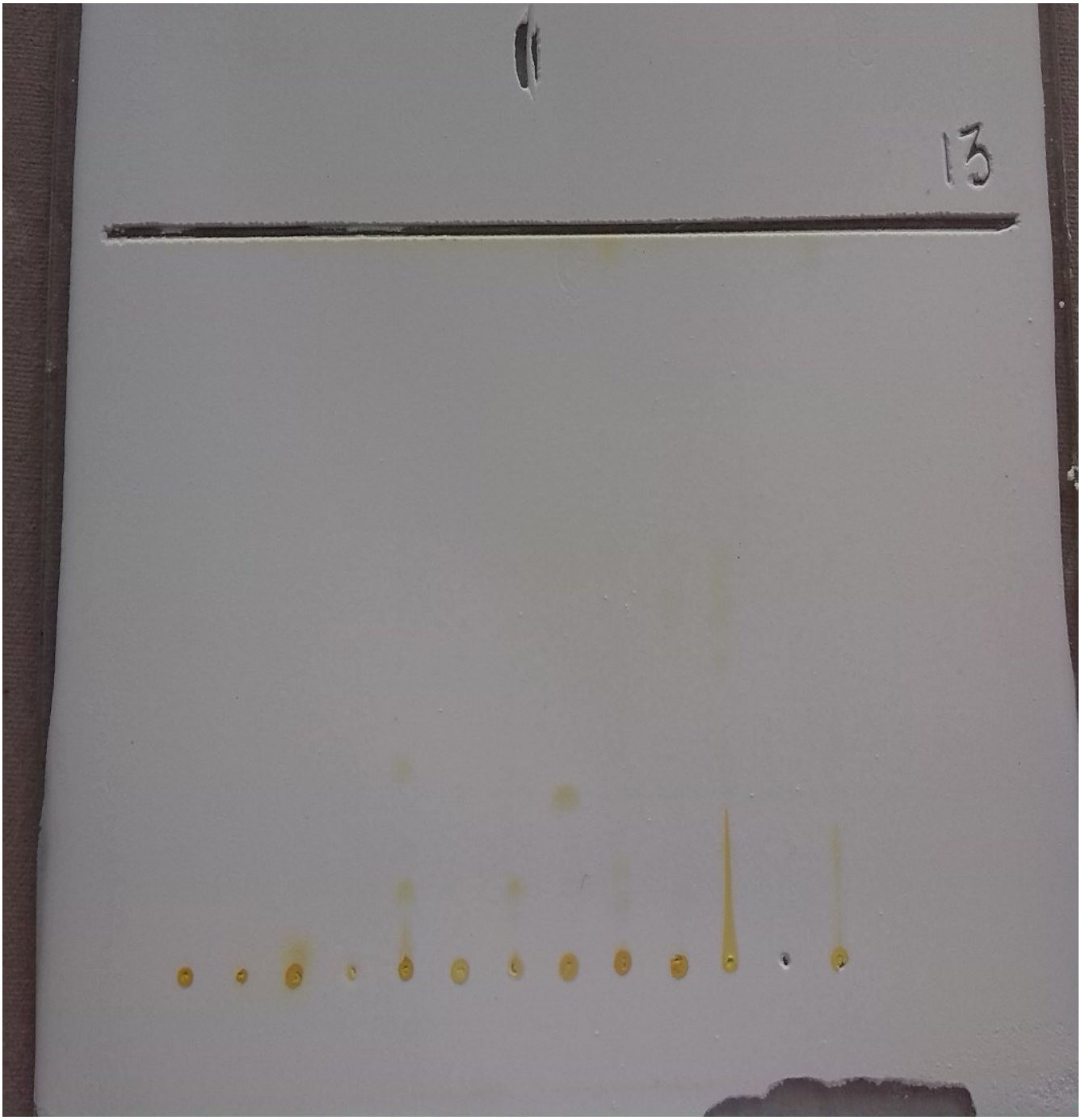


Fig. 1 -TLC result of solvent system S5

Carbon tetrachloride: Cyclohexane (90: 10)

Conclusion

Eight hairspray samples were analyzed by thirty different solvent systems through TLC and by one of the best solvent system (from TLC results), through HPTLC. Solvent system [Carbon tetrachloride: Cyclohexane (90: 10)], is found to be the most suitable for the separation of components of hairsprays using TLC and HPTLC. From the results of thin layer chromatography and High performance thin layer chromatography it is concluded that different components of hair sprays could be separated using these techniques. In this study it was found that using the same solvent system for analysis of hair sprays using these techniques, the result of the separation of the

components varies. It is interesting to note that components which could be separated by thin layer chromatography were separated using high performance thin layer chromatography. CK, Gatsby and Sora Amore gave no results using TLC, while Using HPTLC analysis gave better results. In this study a preliminary attempt has been made to separate the different components of hairsprays as the related information is not available.

1. **Conflict of Interest**: None
2. **Source of funding**: Self
3. **Acknowledgement**: We are thankful to Sophisticated Instruments Centre, Punjabi University, Patiala for providing infrastructure to conduct the study.

Table-1 Showing Solvent systems used in the present study

Solvent System Codes	Solvent System	Ratio
S1	Chloroform : Methanol	90 : 10
S2	Cyclohexane ; Chloroform	80 : 20
S3	Chloroform : Ethanol	90 : 10
S4	Chloroform : n-Hexane	75 : 25
S5	Carbon tetrachloride : Cyclohexane	90 : 10
S6	Carbon tetrachloride : n-Hexane	75 : 25
S7	Acetonitrile : Water	80 : 20
S8	Hexane : Toluene : Acetic acid	50 : 50 : 2
S9	Carbon tetrachloride : Ethanol	90 : 10
S10	Carbon tetrachloride : Methanol	90 : 10
S11	n-Hexane : Toluene : Chloroform	20 : 20 : 60
S12	Acetonitrile : Methanol	80 : 20
S13	Chloroform : Acetone	70 : 30
S14	Chloroform : Benzene : Diethyl ether	80 : 10 : 10
S15	Chloroform : Toluene : Acetonitrile	80 : 15 : 5
S16	Dichloromethane : Cyclohexane : Ethanol	65 : 25 : 10
S17	Dichloromethane : Toluene : Methanol	60 : 20 : 20
S18	Dichloromethane : Cyclohexane	80 : 20
S19	Dichloromethane : Toluene	80 : 20
S20	Chloroform : Toluene	70 : 30
S21	Dichloromethane : n-Hexane	70 : 30
S22	Chloroform : Diethylamine	90 : 10
S23	Chloroform : Methanol : Water	100 : 30 : 2
S24	Chloroform : Methanol : Water : Acetic acid	50 : 3 : 30 : 8
S25	Dichloroethane : Chloroform	60 : 40
S26	Dichloroethane : Carbon tetrachloride	60 : 40
S27	Dichloroethane : Ethanol	70 : 30
S28	Dichloroethane : Methanol	70 : 30
S29	Cyclohexane : Ethyl acetate	70 : 30
S30	Chloroform : Ethyl acetate	80 : 20

Table-2 Showing Best solvent systems

Solvent System Codes	Solvent Systems	Ratio
S2	Cyclohexane ; Chloroform	80 : 20
S3	Chloroform : Ethanol	90 : 10
S5	Carbon tetrachloride: Cyclohexane	90: 10
S7	Acetonitrile : Water	80 : 20
S8	Hexane : Toluene : Acetic acid	50 : 50 : 2
S9	Carbon tetrachloride : Ethanol	90 : 10
S10	Carbon tetrachloride : Methanol	90 : 10
S28	Dichloroethane : Methanol	70 : 30

Table-3 Showing Results of HPTLC of hairspray samples

S. No	Sample	Peaks	Rf
1	Berina	1	4
2	CK	1	3
		2	16
3	Gatsby	1	3
4	Keabeen	1	3
5	Nova	1	4
		2	60
6	Nova Gold	1	3
		2	9
7	Sora amore	1	2
		2	4
8	Swift	1	4
		2	9

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Tables