Journal of Forensic Chemistry and Toxicology
Volume 4 Number 1, January - June 2018
DOI: http://dx.doi.org/10.21088/jfct.2454.9363.4118.2

Original Article

Derivative Ultraviolet Spectrophotometric Studies on Ignitable Liquids

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Received on 10.11.2017, **Accepted on** 08.12.2017

Abstract

The present article describes the potential utility of derivative ultraviolet (UV) spectrophotometric technique in the discrimination of different kinds of ignitable liquids in their neat states. Derivative UV spectrophotometry is capable of differentiating these ignitable liquids as derivative spectra have more number of points for comparison than their corresponding normal zero order spectrum. Derivative spectra are relatively simple. The technique is rapid, simple, cost-effective and can be used for the screening purpose at the initial stage of investigation. Taking into account the results obtained in the present work, it is possible to suggest the use of this technique to distinguish these ignitable liquids, if recovered from arson scene or clandestine laboratories.

Keywords: Derivative Ultraviolet Spectrophotometry; Clandestine Laboratory; Arson; Ignitable Liquids; Petroleum Products; Solvent Extraction.

Introduction

An ignitable liquid (IL) is a volatile, inflammable liquid with a flash point of less than 200°F [1]. It includes charcoal lighter fluids, paint thinners, cleaning solvents, engine fuels, lamp oils, polishes and lubricants [1,2]. IL's can be broadly classified into 2 classes: petroleum and non-petroleum based IL's. Petroleum based IL's are refined from crude oil and includes petrol, kerosene and diesel. These IL's are made up of hydrocarbons. Non-petroleum based IL's are those derived from other sources and includes oxygenated solvents and naturally derived products such as turpentine. Non-petroleum based IL's can further be divided into 2 subclasses: oxygenated solvents and natural products derived from plants. Oxygenated solvents are rich in oxygenated compounds. These solvents can be single compound product (e.g. methanol, isopropanol) or complex mixtures (e.g. lacquer thinner, enamel reducer). Natural products extracted from plants categorized under the subclass of non-petroleum based IL's and includes turpentine and citrus oil extract [1].

IL's are frequently used in different daily-life situations varying from domestic use to commercial or industrial use. However, these substances can be used in criminal activities such as in committing arson, in illegal preparation of prohibited drugs and improvised explosive devices (IED's). In cases of arson, IL's are frequently used to initiate the fire due to their easy availability, simple handling, storage, transportation and cost effectiveness. These substances are frequently used as a fire accelerant in arson and in bride burning cases [1,3]. The unexplained presence of IL's strongly indicates a fire of suspicious origin. Detection and identification of these IL's are therefore helpful in determining origin and cause of fire [4].

Different analytical techniques such as Infrared (IR) spectrophotometry [5], nuclear magnetic resonance (NMR) spectroscopy [6], thin layer chromatography (TLC) [7], gas chromatography (GC) and gas chromatography – mass spectrometry (GC-MS) [8-10] can be used to detect and identify traces of IL's in samples of forensic importance. However, these techniques have many shortcomings. The drawbacks of IR spectrophotometry include the requirement of a clean, pure and moist free sample for analysis. Lack of reference database and limited access are the major limitations of NMR spectroscopy. TLC can be used to analyse only dye components of sample. However, GC and GC-MS are frequently used for the analysis of IL's due to its high sensitivity, resolution and specificity. Despite the reasonable success of GC, it suffers from certain problems: technique is destructive in nature, peaks generated from substrate and burning and pyrolysis products of it causes interference in identification, evaporation of IL's during fire causes loss of its low boiling components which further raises problems in interpretation of chromatograms [11]. Since some IL's contain number of components so long run time is required to resolve these components and hence method is time consuming. The complex nature of chromatograms makes the comparison process tedious and raises the question against the reliability of interpretation and identification. Peaks from the background substrates such as cloth, wood etc. also enhance the complexity of chromatogram and further complicate the interpretation of chromatogram [12]. Most of these problems associated with GC can be minimized or eliminated through the use of UV spectrophotometry in derivative mode.

Derivative ultraviolet spectrophotometry is an analytical technique in which normal zero order spectrum of sample is mathematically differentiated into a derivative (first or higher derivatives) and thereby enhances the "fingerprint" of a sample and provides cleaner spectrum. It isolates qualitative and quantitative information from overlapping bands of the analytes and interferences and useful for analysis of mixture of multi-components. This technique improves resolution bands, eliminates the influence of background or matrix and provides more defined fingerprints than traditional ordinary or direct absorbance spectra. It can separate superimposed curves for quantitative measurements and is able to suppress matrix effects [3,13,14]. Second derivative UV spectrophotometry improves the detectability of spectral features [15].

Lawrence [15] used second derivative UV spectrometric method to differentiate amphetamine and phenethylamine. Transformation of spectrum into derivative form enhances the resolution of peaks and thereby improves the specificity of method. Davidson and Elsheikh [16] determined ephedrine and pseudoephedrine in different kinds of pharmaceutical preparations using second and fourth derivative UV spectrophotometry. Lawrence and MacNeil [17] used second derivative UV spectrometry to differentiate amphetamine, ephedrine, meperidine, phentermine and phenethylamine. Gill et al. [18] used derivative UV spectroscopy to detect amphetamine in liver extract. It is observed that second derivative UV spectra can be used to eliminate the broad background absorption. Verweji and Bonte [19] detect the carboxyhaemoglobin in blood samples by using second derivative spectrophotometry. Cruz et al. [20] also determined carboxyhaemoglobin and total haemoglobin in carbon monoxide intoxicated patients by using third derivative spectrophotometry. Randez-Gil et al. [21, 22] simultaneously determined nitrazepam and clonazepam in urine and blood plasma samples by high order (fourth and fifth) derivative spectrophotometry. Lawrence and Kovar [23] used amplitude difference D_{308}-D_{300} and D_{286}-D_{282} to determine the concentrations of cannabinol and Δ^9-tetrahydrocannabinol in mixtures containing up to 99% of Δ^9- tetrahydrocannabinol and 50% of cannabinol. A group of scientists [24,25] identified and determined cocaine in binary mixtures of cocaine and local anaesthetics using derivative UV spectrophotometry. It was observed that technique was useful to distinguish cocaine-lidocaine, cocaine-procaine, cocaine-tetracaine and cocaine-benzocaine mixtures. Kuo et al. [26] simultaneously determined diquat and paraquat in blood, urine and tissue samples using second order UV derivative spectrophotometry. Sharma et al. [27] analyzed some commonly abused over the counter drugs by derivative ultraviolet spectrophotometric method. Kaur et al. [28] analyzed some undetonated explosives by derivative ultraviolet spectrophotometry. Saini et al. [29] compared some lipstick smears by ultraviolet-visible spectrophotometry operated in derivative mode and pointed out that derivative spectrophotometry provides more points for comparison than conventional ultraviolet spectrophotometry. Meal [30] analysed the fire debris samples by using second derivative ultraviolet spectroscopy and observed a unique and easily recognizable second derivative UV spectrum of petrol, kerosene and diesel. Absence of minima at 251nm and maxima at 261nm in second derivative spectrum of kerosene differentiate it from diesel. Zerlia et al. [31] analyzed different petroleum products using ultraviolet spectrometry and suggested that present method can be used as a tool for rapid screening of petroleum products in petroleum field without performing chromatographic separation prior to analysis by ultraviolet spectrometry. Bumbrah et al. [32] detected residues of petroleum products residues in simulated fire debris samples by derivative UV spectrophotometry. Dixit et al. [33] used second derivative ultraviolet spectrophotometry to determine naphthalene and its derivatives in petroleum fractions.

It is observed that very little work had been done on the analysis of IL's using derivative ultraviolet spectrophotometry. Therefore, in the present study, UV spectrophotometry in normal and derivative mode is used to analyse IL's. In this paper, we describe the potential utility of normal and derivative UV spectrophotometry in the analysis of IL's.

Materials and Method

Reagents and Samples

The commercially available IL's including thinners and petroleum products (petrol, kerosene and diesel) analysed in the present study were purchased from local market, petrol pump and oil depot of Patiala city, Punjab and details of samples are given in Table 1. Cyclohexane of analytical grade and Whatman filter paper were purchased from Loba Chemie, Ambala. All samples were stored in glass vials (20ml) with screw caps (Labbox, Mataro, Barcelona, Spain) and kept at 4^0C until their analysis.

Data Acquisition

Double beam UV-VIS spectrophotometer with model 1700 PharmaSpec (Shimadzu Corporation, Kyoto, Japan) was used to record the absorbance of samples. UVProbe Version 2.32 (Shimadzu Corporation, Kyoto, Japan) software was used to record and process the normal and higher order derivative UV spectra of all samples. Quartz cells of 1cm path length were used. Instrument was operated in spectrum mode to record the zero, first and second order derivative spectra of samples. All samples were scanned from 320 to 245nm region of ultraviolet band. Sample concentrations were adjusted to provide a sample absorption maximum of within unity. Cyclohexane was used as extracting solvent as well as reference. The following instrumental parameters were kept constant throughout the present study:

- Measurement mode – Absorbance
- Scanning range – 320-245nm
- Absorbance recording range – 0.00A ~ 1.00A
- Scan speed – Fast
- Scan mode – Auto
- Number of scans – 1
- Display mode – Overlay

Sample Preparation

Before preparing the samples, all IL's were filtered using Whatman filter paper in order to remove any contaminants or sediments present in it. Neat samples of IL's were prepared by dissolving 20µl filtered IL in 10ml of cyclohexane and were subjected to UV spectrophotometry and spectrum was recorded in the range of 320 to 245nm. Three different samples of each IL in their neat state was analysed three times. In this way, total of 90 spectra were recorded.

Results and Discussion

In the present study, ultraviolet spectrophotometry in normal and derivative mode is used to analyze different kinds of IL's including petroleum products. The potential utility of this technique in screening of different IL's is observed. Table 1 reflect the characteristic peak

Table 1: Description of ignitable liquids including sample code, name, class, colour, brand, manufacturer and location

S. No.	Sample Code	Name	Class	Colour	Brand Name	Manufacturer	Location
1.	S1	Crude	Thinner	Colorless	Local	NR	Patiala
2.	S2	Shine	Thinner	Colorless	SL	Kayson Thinners & Chemicals	Jalandhar
3.	S3	Lusture	Thinner	Dark Purple	Ramble	Ramble Cosmetics	Delhi
4.	S4	Welcome	Thinner	Colorless	New Wembley	New Wembley Products	Delhi
5.	S5	Axalta	Thinner	Colorless	Axalta	Axalta Coating India Pvt. Ltd.	Gujarat
6.	S6	Coats	Thinner	Dark Yellow	NR	NR	Patiala
7.	S7	Nails	Nail Polish Remover	Colorless	Camieo	NR	Delhi
8.	S8	Petrol	Petroleum Product	Orange	Hindustan Petroleum	Hindustan Petroleum	Patiala
9.	S9	Kerosene	Petroleum Product	Blue	Public Distribution Services	NR	Patiala
10.	S10	Diesel	Petroleum Product	Yellow	Hindustan Petroleum	Hindustan Petroleum	Patiala

wavelengths of different IL's in their neat states along with type of spectrum recorded.

The zero order spectrum of sample S1 shows a broad absorption region at 261.5nm (Figure 1). However, in case of its first and second order derivative, the spectra are bipolar with more points for comparison. In its first order derivative spectrum, maximum absorbance occurs at 252.0nm (Figure 2) while in second order derivative spectrum of sample S1, this characteristic peak shifts to 272.0nm (Figure 3). In case of sample S1, as such no specific pattern is observed in shifting of wavelength region of characteristic absorption peak with change in derivative order. It is observed that number of maxima and minima points increases with derivative order (i.e., from zero to second order derivative). The second order derivative spectrum has more number of maxima and minima points than their corresponding zero and first order derivative spectra (Table 2). The zero order spectrum of sample S2 shows a broad absorption region at 262.0nm (Figure 1). In its first order derivative spectrum, peak of maximum absorbance shifts from 262.0nm to 251.5nm (Figure 2) while in its second order derivative spectrum, this characteristic peak further shifts to 273.0nm (Figure 3). Similar trend, as in case of sample S1, is observed in number of maxima and minima points in respective spectra with their derivative orders (Table 2).

In zero order spectrum of sample S3, peak of maximum absorption is present at 278.0nm (Figure 1) which is shifted to 256.0nm in its first order derivative spectrum (Figure 2). In second order derivative spectrum of sample S3, this characteristic peak further shifts to 298.5nm (Figure 3). Neither increase nor decrease is observed in number of maxima points with derivative order. All spectra of sample S3 have only one maxima point irrespective of derivative order. However, second order derivative spectrum of sample S3 has more number of minima points than their corresponding zero and first order derivative spectra. Sample S4 show no characteristic peak between 320 and 245nm in its zero order spectrum (Figure 1). However, only one peak at 266.5nm is observed in its first order derivative spectrum. Some hidden peaks are resolved and observed at 272.5, 265.5, 257.5 and 252.0nm in its second order derivative spectrum (Table 2). It is observed that second order derivative spectrum of sample S4 have more points, between 320 and 245nm, for comparison

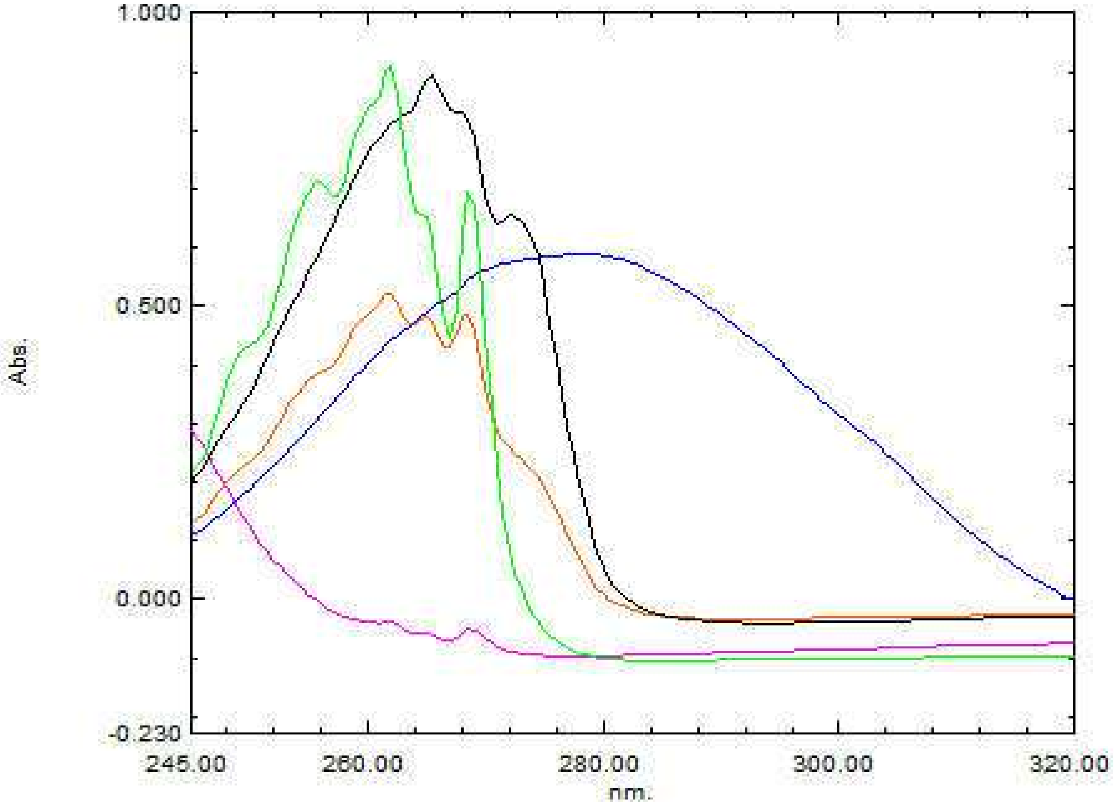

Fig. 1: Normal UV spectra of S1 (Orange), S2 (Green), S3 (Blue), S4 (Pink) and S5 (Black)

Table 2: Characteristic peaks of ignitable liquids in their normal and higher order derivative spectra (1st and 2nd)

S.No.	Sample Code	Spectrum Order					
		Zero		First		Second	
		Maxima (nm)	Minima (nm)	Maxima (nm)	Minima (nm)	Maxima (nm)	Minima (nm)
1.	S1	261.5[a]	292.0	252.0[a]	276.5, 274.5, 271.5	280.0, 272.0[a], 265.0, 257.5, 251.0, 245.0	275.0, 268.0, 260.5, 254.0
2.	S2	262.0[a]	285.0	251.5[a]	272.0, 266.5, 263.5	273.0[a], 265.5, 258.0, 251.5, 245.5	269.0, 261.5, 254.5, 248.5
3.	S3	278.0[a]	-	256.0[a]	307.5, 301.5, 296.5	298.5[a]	304.0, 280.0, 275.5, 270.5, 263.0
4.	S4	-	277.0	266.5[a]	272.0	272.5, 265.5, 257.5, 252.0	269.0, 262.5
5.	S5	265.5[a]	293.0	254.5[a]	277.0	279.0[a], 270.5, 256.0, 249.5	274.5, 266.5, 253.5, 247.0
6.	S6	272.5, 270.5, 268.0[a]	319.5, 317.0	258.0[a]	294.5, 288.0, 278.0	298.0, 282.5[a]	293.5, 288.0, 273.5, 266.5
7.	S7	275.0[a], 258.5	-	270.0[a]	304.5, 286.5	289.5[a]	283.5, 275.0
8.	S8	261.5[a]	310.0	250.0[a], 246.0	277.0	279.0[a], 271.5, 265.0, 251.0, 245.0	274.5, 267.5, 260.5, 254.5, 248.0
9.	S9	272.5[a], 270.5, 268.5	317.5	257.5[a]	295.5, 289.0, 281.0	298.5, 283.0[a], 255.5, 252.5	293.0, 278.5, 276.0, 273.0, 269.5, 266.5, 262.0, 260.0
10.	S10	258.5[a]	315.0	245.0[a]	277.0, 271.5, 268.5	280.0[a], 270.5	274.5, 266.5, 259.0, 254.0, 247.0

"a" – Indicates wavelength of maximum absorbance
"-" – Not detected

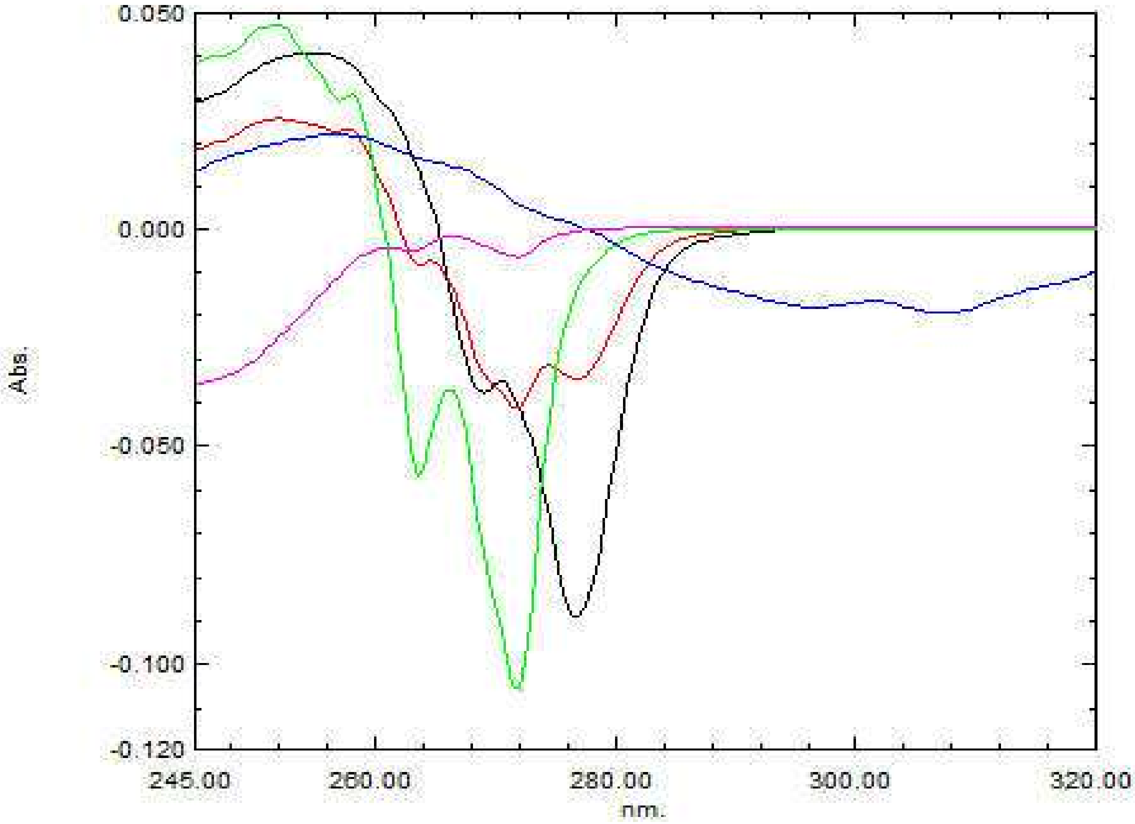

Fig. 2: First order derivative UV spectra of S1 (Red), S2 (Green), S3 (Blue), S4 (Pink) and S5 (Black)

than their corresponding zero and first order derivative spectra. The zero order spectrum of sample S5 shows a broad absorption region at 265.5nm (Figure 1). However, in its first order derivative spectrum, peak of maximum absorbance shifts from 265.5nm to 254.5nm (Figure 2) which is further shifted to 279.0nm in its second order derivative spectrum (Figure 3). The second order derivative spectrum of sample S5 also shows three maxima points at 270.5, 256.0 and 249.5nm respectively. Same numbers of maxima and minima points are present in all spectra of sample S5. The second order derivative spectrum of sample S5 contains more number of maxima and minima points than their corresponding zero and first order derivative spectra.

The zero order spectrum of sample S6 shows a characteristic absorption peak at 268.0nm (Figure 4). It also shows two maxima points at 272.5 and 270.0nm. However, in case of its first order derivative spectrum, maximum absorbance occurs at 258.0nm (Figure 5) while in second order derivative spectrum of sample S6, this characteristic peak shifts to 282.5nm (Figure 6). It is observed that number of maxima points decreases from zero to first order derivative spectrum. However, the number of minima points increases with increase in derivative order. The

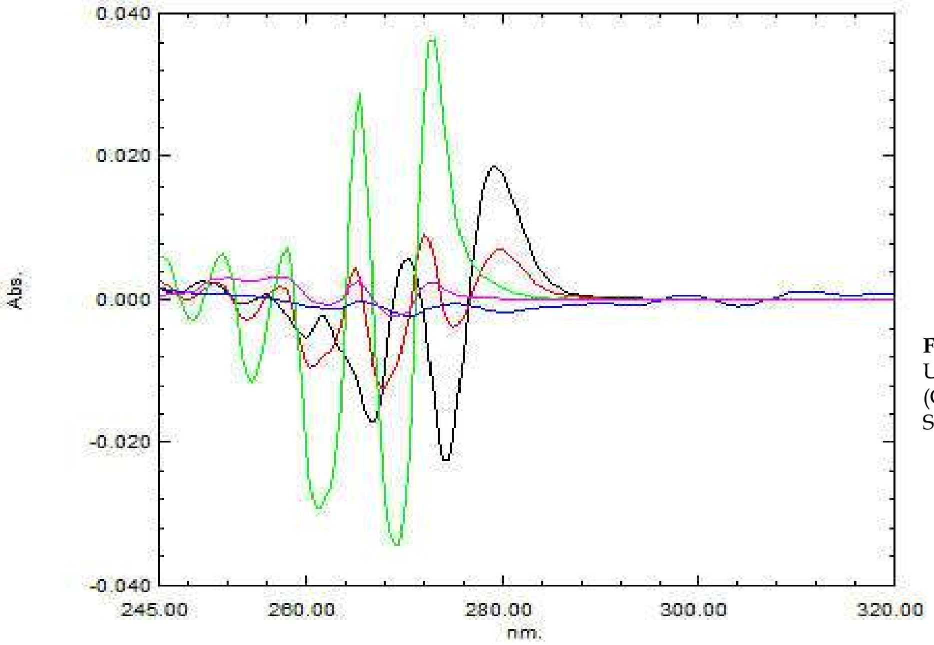

Fig. 3: Second order derivative UV spectra of S1 (Red), S2 (Green), S3 (Blue), S4 (Pink) and S5 (Black)

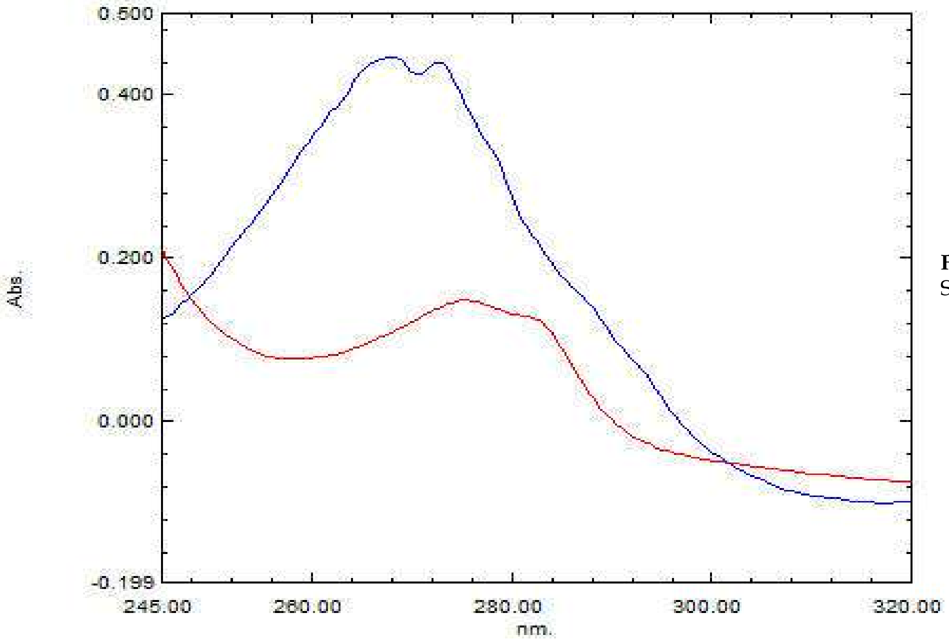

Fig. 4: Normal UV spectra of S6 (Blue) and S7 (Red)

second order derivative spectrum has more number of minima points than their corresponding zero and first order derivative spectra (Table 2). In zero order spectrum of sample S7, peak of maximum absorption is present at 275.0nm (Figure 4) which is shifted to 270.0nm in its first order derivative spectrum (Figure 5). In its second order derivative spectrum, this characteristic peak shifts from 270.0 to 289.5nm (Figure 6). The first and second order derivative spectra of sample S7 have same number of maxima and minima points but at different wavelengths.

The zero order spectrum of sample S8 shows a broad absorption region at 261.5nm (Figure 7). This characteristic absorption peak shifts from 261.5nm to 250.0nm in its first order derivative spectrum (Figure 8) which is further shifted to 279.0nm in

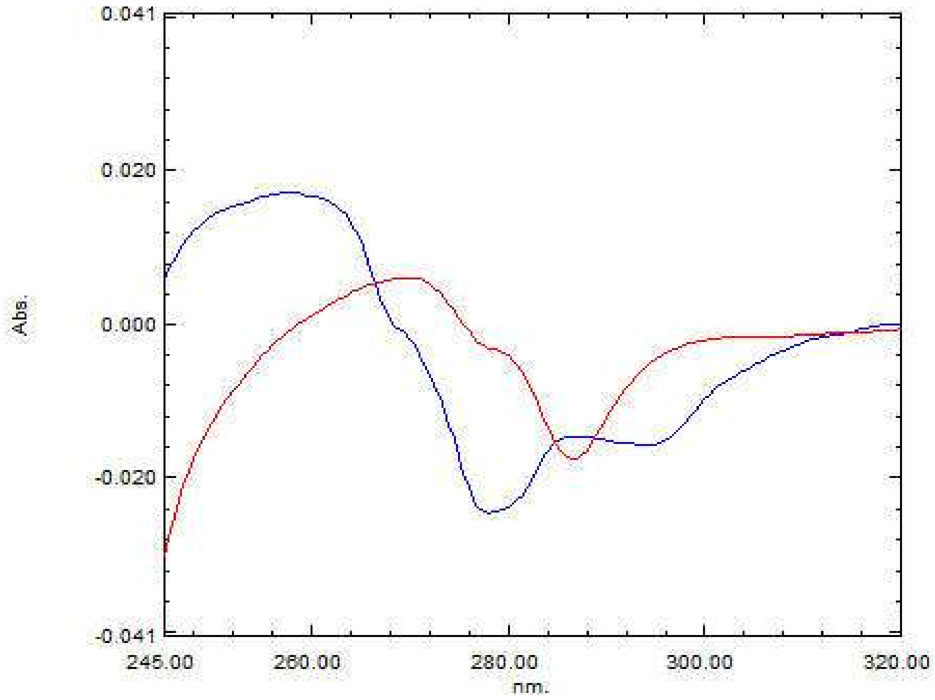

Fig. 5: First order derivative UV spectra of S6 (Blue) and S7 (Red)

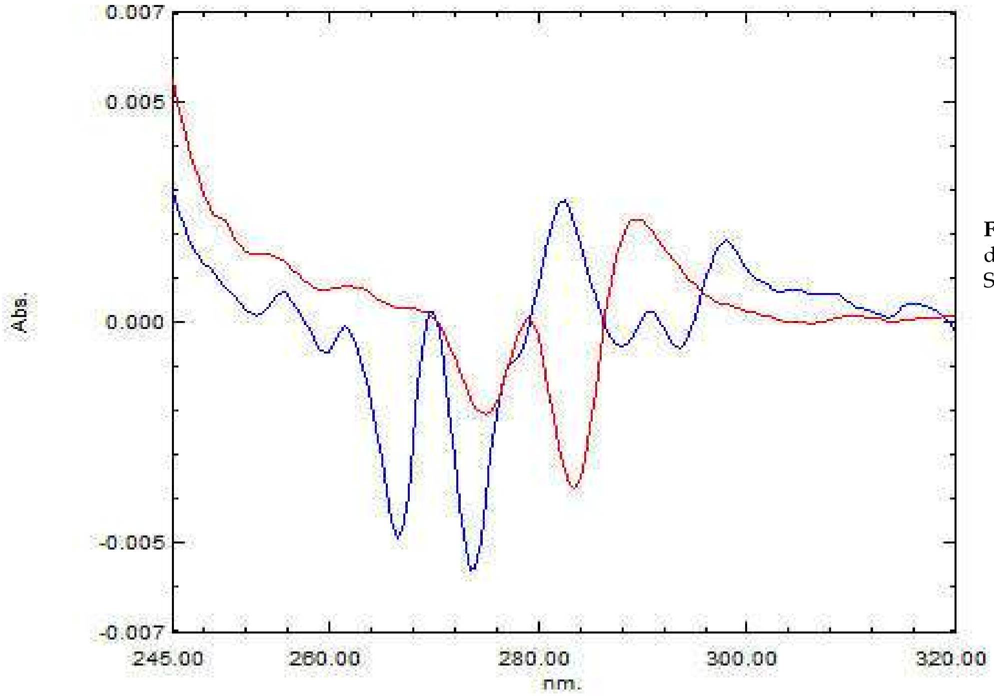

Fig. 6: Second order derivative UV spectra of S6 (Blue) and S7 (Red)

its second order derivative spectrum (Figure 9). The second order derivative spectrum of sample S8 also shows maxima points at 271.5, 265.0, 251.0 and 245.0nm respectively. It is observed that number of maxima points increases with increase in derivative order of spectrum. The second order derivative spectrum has more number of minima points than their corresponding zero and first order derivative spectra (Table 2). Meal [30] observed characteristic strong minima at 274nm in second order derivative spectrum of petrol. In a similar study, Bumbrah et al. [32] observed characteristic strong maxima at 251.3nm and minima's at 274.1 and 248.0 in second order derivative spectrum of petrol. The zero order

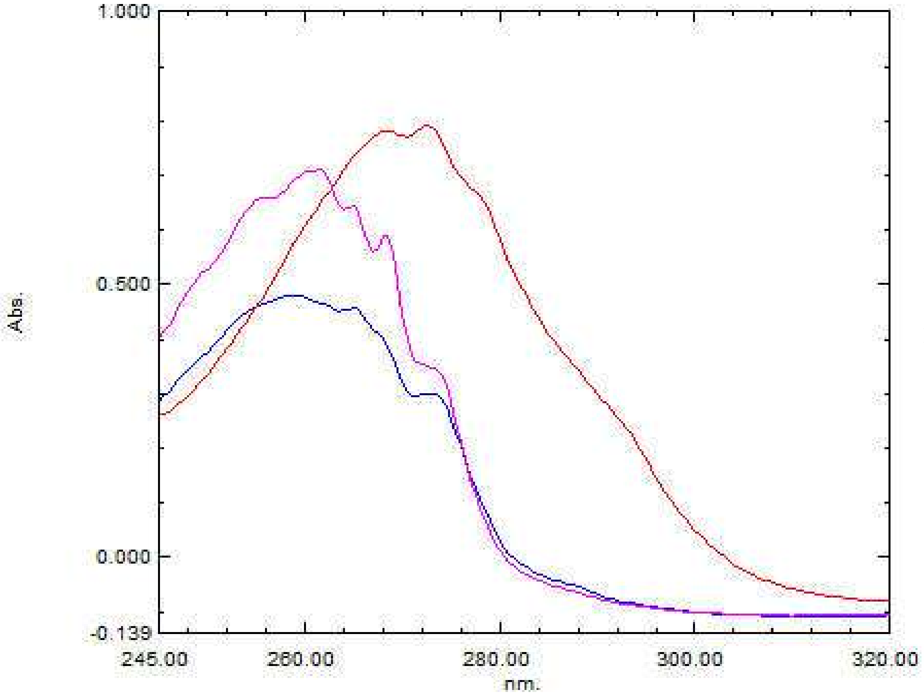

Fig. 7: Normal UV spectra of S8 (Pink), S9 (Red) and S10 (Blue)

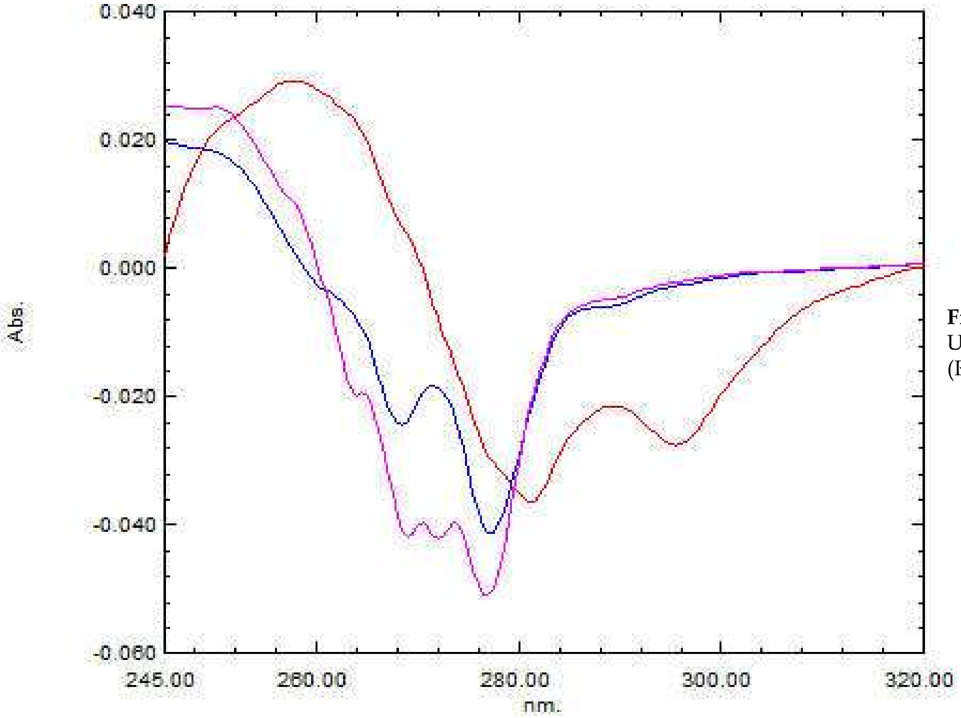

Fig. 8: First order derivative UV spectra of S8 (Pink), S9 (Red) and S10 (Blue)

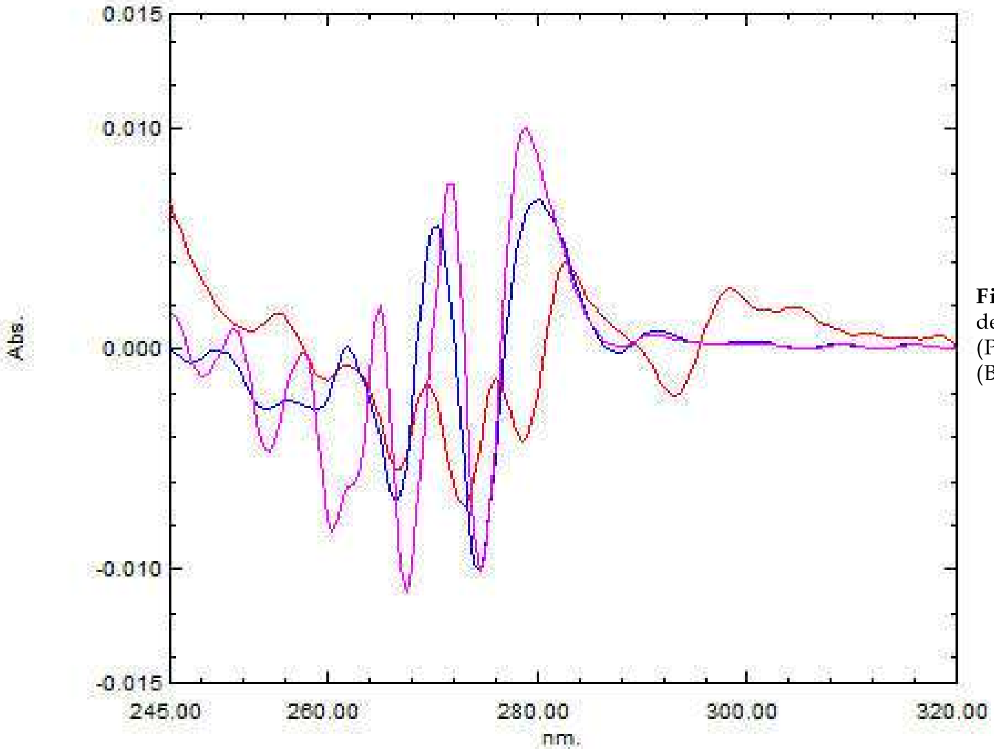

Fig. 9: Second order derivative UV spectra of S8 (Pink), S9 (Red) and S10 (Blue)

spectrum of sample S9 shows a characteristic absorption peak at 272.5nm (Figure 7). It also shows two maxima's at 270.5 and 268.5nm. However, in case of its first order derivative spectrum, maximum absorbance occurs at 257.5nm (Figure 8) while in its second order derivative spectrum, this characteristic peak shifts from 257.5nm to 283.0nm (Figure 9). Second order derivative spectrum of sample S9 also shows maxima's at 298.5, 255.5 and 252.5nm respectively. It is observed that number of minima points increases with increase in derivative order of spectrum. The second order derivative spectrum has more number of minima points than their corresponding zero and first order derivative spectra. However, similar correlation is not observed between number of maxima points and derivative order (Table 2). Meal [30] observed characteristic strong minima at 276nm in second order derivative spectrum of kerosene which is in accordance with the present study. Bumbrah et al. [32] also observed strong maxima points at 272.7nm, 268.1nm and 257.8nm in zero and first order derivative spectra of kerosene. Strong minima points at 317.5nm and 289.1nm are also observed in zero and first order derivative spectra of kerosene. In zero order spectrum of sample S10, peak of maximum absorption is present at 258.5nm (Figure 7).

This characteristic absorption peak shifts from 258.5nm to 245.0nm in its first order derivative spectrum (Figure 8) which is further shifted to 280.0nm in its second order derivative spectrum (Figure 9). It is observed that number of minima points increases with increase in derivative order of spectrum. The second order derivative spectrum has more number of minima points than their corresponding zero and first order derivative spectra (Table 2). Bumbrah et al. [32] also observed strong maxima and minima points at 270.2nm and 247.2nm in second order derivative spectrum of diesel.

It is observed that appearance of strong maxima and minima at 272.0nm and 268.0nm in second order derivative spectrum of sample S1 are specific and can be used to differentiate it from other samples. Similarly, maxima at 273.0nm and minima's at 261.5nm and 248.5nm, in second order derivative spectrum of sample S2, are specific. In second order derivative spectrum of sample S3, strong minima's at 304.0nm and 280.0nm can be used to differentiate it from other samples. Although the zero order spectrum of sample S4 is unique in itself and did not match with rest of samples yet the presence of strong maxima and minima at 252.0nm and 262.5nm in its second order derivative spectrum are useful to differentiate it.

In second order derivative spectrum of sample S5, presence of maxima and minima at 249.5nm and 253.5nm can be used to differentiate it from other samples. In second order derivative spectrum of sample S6, maxima and minima at 282.5nm and 288.0nm are specific. Similarly, in second order derivative spectrum of sample S7, maxima and minima at 289.5nm and 283.5nm can be used to discriminate it from other samples. Sample S8 can be easily distinguished from other samples by observing strong maxima and minima's at 271.5nm, 267.5nm and 248.0nm in its second order derivative spectrum. Similarly, sample S9 can be easily discriminate from other samples by observing strong maxima's and minima at 283.0nm, 255.5nm and 278.5nm in its second order derivative spectrum. In second order derivative spectrum of sample S10, presence of strong minima at 259.0nm is specific and can be used to discriminate it from other samples.

It is important to note that peak shifts from zero to first order derivative spectrum are close while a significant change is observed in peak shifts from first to second order spectrum of all samples. Thus, peak shifting is a useful parameter to distinguish different IL's on the basis of their second order derivative spectra. Therefore, peak shifting along with the visual inspection of normal and higher order derivative spectra are useful to discriminate IL's. The presence or absence of maxima or minima point in derivative spectra (first and second) can be used to establish the differentiation between samples and can be helpful in exclusion of suspected IL. Although the technique is incapable of identifying the IL yet it is very useful and effective in screening. Derivative spectra not only provide more points for comparison than their corresponding normal zero order spectrum but it also enhances the certainty in exclusion of samples. Transformation of normal UV spectrum into derivative (first and second) spectra also enhances the discriminating potential of this technique. Derivative UV spectrophotometric technique can be very useful and helpful for the screening of IL's. In addition to this, the technique can be used for initial screening purpose and final identity can be established by analyzing the same sample with more sophisticated instrumental techniques such as GC-MS and GC-MS-MS.

Conclusion

Derivate Ultraviolet spectrophotometry is a simple, easy, cost-effective, non-destructive, well established analytical technique for the analysis of IL's as it provides much better fingerprints of IL's than the conventional UV spectrophotometry by resolving the overlapped and hidden peaks in spectra of IL's. It is a rapid and reliable screening technique and can be used to exclude the sample by visual comparison of derivative spectra of sample with their corresponding standard at the initial stage of analysis process.

Acknowledgements

Authors are thankful to University Grants Commission (UGC) for providing financial grant in terms of Senior Research Fellowship (SRF) to one of the authors (GSB).

Conflict of Interest

The authors declare that they have no conflict of interest.

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