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Screening of (Aminoalkylindols) Cannabinoids in Smoking Products by GC-EI/MS in Jordan: Liquid-liquid Extraction Optimization

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IN THIS work a screening study in Jordan was conducted on identification of herbal smoking mixtures containing recently new marketed synthetic cannabinoids (aminoalkylindols) (XLR-11, AB-CHMINACA, AB-FUBINACA isomers) which were responsible for a few cases for suicide accidents, individual violent action and few homicides. The smoking items were seized by customs and by the Anti-Narcotics Department in the year 2016/17. Forensic evidences including herbal smoking mixtures, cigarettes, and cigarettes waste were screened for containing these banned drugs employing GC-EI/MS in scan mode with JWH-018 and 5F-ADB as surrogates and individual aminoalkylindols reference standards. The liquid-liquid extraction optimization results indicated that dichloromethane gave the highest recovery for aminoalkylindols in herbal smoking mixtures and methanol for cigarette waste filters. The Results indicated that the marketed herbs were in all sprayed with either XLR-11 (around 45%) or combination of either both XLR-11 and AB-CHMINACA or a combination of XLR-11 and AB-FUBINACA isomers (25%) and the rest was AB-CHMINACA or AB-FUBINACA isomers pure or mixtures. These results were further confirmed by cigarette waste analysis which gave approximate results to herbs or spices. To our knowledge this is the first study of its kind in the MENA region and to be conducted in Jordan with an optimization procedure that is not reported in literature.

Keywords: Cannabinoids, Herbal smoking mixtures, Cigarettes, Cigarettes waste, GC-EI/MS

Introduction

Synthetic cannabinoids are psychoactive chemical species that mimic the effects of cannabis (marijuana) when consumed and were originally designed for therapeutic purposes serving as agonists to the same receptors that bind Δ^9- tetrahydrocannabinol (THC). The principal active ingredient of the cannabis plant [1]. In recent years synthetic cannabinoids as part of what is described as recreational designer drugs, a growing consumer phenomenon, have gained a great deal of interest worldwide and locally by the Antinarcotics Department and Forensic Laboratory. These emerging cannabinoids were originally synthesized for medical research purposes [2] but have recently been appropriated into the illicit drug market posing a challenge to law enforcement agencies and forensic laboratories; to catch up with the continuing change of chemical structure functional groups and substitution leading to vast diversity in

analogs and homologues [2,3]. Their high psychoactive effects [4], metabolites diversity [5] and their unlegislated legal status for banning have made them marketed easily among the youth as herbal smoking blends where the synthetic cannabinoids are sprayed or soaked with herbs or plant parts or tobacco. In the last four years since their appearance there was a noticeable increase in the number of new synthetic cannabinoids with diverse classes such as (XLR-11, AB-CHMINACA and AB-FUBINACA isomers) [6-9] invading different spots of the world including recently Jordan. Fast action by the Antinarcotics Department was taken to seize these drugs and send them to forensic laboratories for identification where later on a legislation bill was passed and approved by prime ministry and Jordanian Food and drug Administration to add them to the list of banned abused drugs in 2016. Several methods have been reported in the literature for analysis of synthetic Cannabinoid in different samples. These included different chromatographic methods using HPLC and GC coupled with mass detector. Wurita, et al [10] identified and quantified 5-fluoro-ADB-PINACA and MAB-CHMINACA in dubious herbal products by extraction with acetonitrile using GC-MS in scan mode. Umebachi and co-workers [11] scanned indole and indazole synthetic cannabinoids using GC-EI-MS, positive chemical ionization (positive CI)–MS, and negative chemical ionization (negative CI)–MS where methods were evaluated for the structural elucidation and identification of synthetic cannabinoid composition. Raso & Bell [12] in 2017 performed a qualitative analysis and detection of the pyrolytic products of JWH-018 and 11 additional synthetic cannabinoids including XLR-11, AB-CHMINACA and AB-FUBINACA in the presence of common herbal smoking substrates. Presley et al [13] reviewed in 2013 the analysis of synthetic cannabinoids in botanical material including analytical methods and the history of synthetic cannabinoids and how they have been detected in the illicit drug market including the classes JWH, CP, HU, AM, WIN, RCS, XLR and UR. Jang, et al [14] determined urinary metabolites of XLR-11 by liquid Chromatography–quadrupole time-of-flight mass spectrometry using β-glucuronidase to hydrolyze samples at pH = 4.5 followed by SPE. Also different research groups used high-resolution mass spectrometry for purposes of metabolites study using LC-MS; for example Castaneto, et al [15] identified AB-FUBINACA

metabolites in human hepatocytes. Scheidweiler, et al [3] used non-targeted SWATH acquisition for identifying 47 synthetic cannabinoid metabolites in human urine by liquid chromatography-high-resolution tandem mass spectrometry including XLR-11 using enzymatic hydrolysis followed by liquid extraction. Kataev, et al [16] identified the main metabolites of AB-FUBINACA in urine of consumers of smoking mixes by using GC-MS technique. Whereas others like Hutter et al [17] used different matrix to determined 22 synthetic cannabinoid in hair by using liquid chromatography tandem mass spectrometry.

In this work a screening case study in Jordan was conducted on identification of herbal smoking mixtures containing recently new marketed synthetic cannabinoids belonging to the class of aminoalkylindoles. Including XLR-11 as cyclopropylindole, and AB-CHMINACA with AB-FUBINACA isomers as indole carboxamides. These recreational drugs were responsible for a few cases for suicide accidents, individual violent actions, hospitalization, and few homicides. Seized by customs and Anti-narcotics Department in the first quarter of 2016 .Forensic evidences including herbal smoking mixtures, cigarettes, and cigarettes' waste samples were screened in the Forensic Laboratory (accredited ISO/IEC 17025:2005 from Perry Johnson Laboratory Accreditation, Inc. (PJLA) (Michigan, USA)) for containing these banned drugs. GC-MS in scan mode was employed for identification of these drugs accompanied by liquid extraction technique for sample preparation. Where it was optimized for best solvent using the guidelines in United Nations Office on Drug and Crime (UNODC) recommended methods for the identification and analysis of synthetic cannabinoid receptor agonists in seized material [18].

Experimental

Chemicals and Reagents

The following chemicals were purchased: ethyl acetate (minimum 99%, AppliChem company, Darmstadat, Germany), methanol and ethanol analytical grade (minimum assay 99.9%, Scharlau company, Spain), dichloromethane (DCM) (minimum assay 99.9%, TEDIA company, Texas, USA), n-hexane (95% TEDA company, USA), sodium sulfate anhydrous (98.5-101%, Scharlau company, Barcelona, Spain). The raw materials as powder for the three synthetic drugs XLR-11, AB-CHMINACA and AB-FUBINACA were purchased through the drug enforcement

department from Lipomed Inc. (Cambridge, USA). JWH-018 and 5F-ADB (Lipomed Inc, Cambridge, USA)

Samples' collection

All samples were supplied from Anti narcotic Department – Public Security Directorate Amman-Jordan, and Jordan Customs Authority. Twenty types of herbal packages, and twenty samples as cigarette waste used by suspect people for doping were all given individual codes. Herbal mixed marketed as spice were given the label herb 1- herb 20 ,cigarettes labeled as C 1-C 20 and cigarette waste given C.W 1 - C.W 20. Fig.1 shows variety of types of samples received by the Forensic Laboratory with more detailed information in Fig. S$_1$ – Fig. S$_5$.

Extraction procedure

Cigarettes and herbal samples extraction (extraction solvent optimization)

Randomly two herbal smoking samples were selected and extracted with different solvents methanol, ethanol, hexane, ethyl acetate and dichloromethane (DCM) based on the guidelines of United Nations Office on Drug and Crime (UNODC) recommended methods for the identification and analysis of synthetic cannabinoid receptor agonists in seized material [18]. This was done in purpose to find out the best extraction solvent that will be used later on for

extraction of all herbal samples. After extraction of each sample (around 0.20 g) with 2.00 ml solvent, centrifuging at 4000 rpm, the supernatant was transferred into a 2.00 ml tube after filtering by 0.45 μm filter membrane and evaporated to dryness. 1.0 ml from different solvent was added to reconstitute sample and then transferred it into a GC vial for qualitative GC-MS analysis along with JWH-018 and 5F-ADB as surrogate and quality control standards for any instrumental variations. The corresponding area for each drug peak (total ion chromatogram) was integrated, where the best extraction solvent showed the highest area of drugs' peak areas. Trials were we performed in triplicate. After choosing the best extraction solvent, twenty herbal samples were extracted with that solvent and analyzed by GC-EI/MS in the scan mode. The same procedure was conducted for the cigarettes.

Cigarette waste extraction

In order to find the best solvent with the highest recovery for cannabinoids in cigarette waste; different solvents methanol, ethanol, hexane, ethyl acetate and dichloromethane were tested. The procedure in section 2.3.1 was used and best solvent was chosen based on the highest area for each recovered cannabinoid. The reference standard material for the three synthetic drugs XLR-11, AB-CHMINACA and AB-FUBINACA were injected and analyzed on GC/MS to verify

Fig. 1. Variety of types of samples received by the Forensic Laboratory.

retention time and mass spectrum of each drugs in addition to the spectral library data confirmation. After choosing the best extraction solvent, twenty Cigarette waste's samples were extracted with that solvent and analyzed by GC-EI/MS in the scan mode for the semi-quantitative analysis.

Mass spectrometry

Gas chromatography coupled to mass spectrometry (GC-MS) from Agilent Technologies was used for analysis. A high pure helium gas (99.999%) was employed as a carrier gas at constant flow rate of 9 mL/ min, column flow rate of 1 ml/min, and a fused silica capillary column DB-5MS (30 m × 0.25 mm × 0.25 μm) film thickness. The injection volume was 1.0 μl used in the split injector (split ratio 5:1). The inlet pressure was 9.38 psi at 280 ^{0}C. The column oven temperature was programmed as follows: initial temperature at 80 ^{0}C, then increased to 295 ^{0}C with a ramp of 15 C/min, finally temperature was held at 295 ^{0}C for 10 min until elution was complete. The mass spectrometer source was operated in the electron impact ionization (EI) at 70 eV, where the temperature of ion source was 200 ºC, for the analyzer 150 ºC, and for interface was 280ºC. The GC-MS software was also supported by Cayman spectral library, Nist 11 library, and Willey 9 library.

Method precision

The analytical method repeatability was evaluated by using three herb samples and three cigarette waste samples representing different composition of the three aminoalkylindols with different peak areas were selected and injected each in triplicate. Then the percent relative standard deviation (RSD) was calculated.

Results and Discussion

Cigarettes, Mixed herbal and cigarette waste samples were analyzed by GC-EI/MS in scan mode (50-500 m/z) by choosing retention time and mass spectrum for each of the three targeted drugs as indicated in Table 1. Where each pure synthetic drug was injected individually for retention time identification and mass fragments confirmation.

Cigarettes and herbal samples

As shown in Fig. 2 and with more details in Table S$_1$, the GC-EI/MS cannabinoids peaks integration results indicated that dichloromethane achieved the highest recovery demonstrated by the highest area for cigarettes and herbal smoking samples. Where all samples were injected in one batch in addition to surrogate quality control sample for any instrumental variations. The routine analysis of herbal smoking mixtures using GC-EI/MS in scan mode with dichloromethane as optimum extraction recovery solvent is shown in Fig. 3 and Table S$_2$.

Where the percentage of each drug in the sprayed powder on each herbal smoking mixture is indicated based on the area ratios of the corresponding peak for each drug in herb to the total area of the three drugs. Where in all, some samples contained only XLR-11 (45%) some contained AB-CHMINACA or AB-FUBINACA (30%) and some samples contained two cannabinoids (XLR-11 with AB-FUBINACA or XLR-11 with AB-CHMINACA) (25%). Fig. 4-6 shows the GC-EI/MS chromatograms for

TABLE 1. Mass fragments of the selected cannabinoids and their retention times

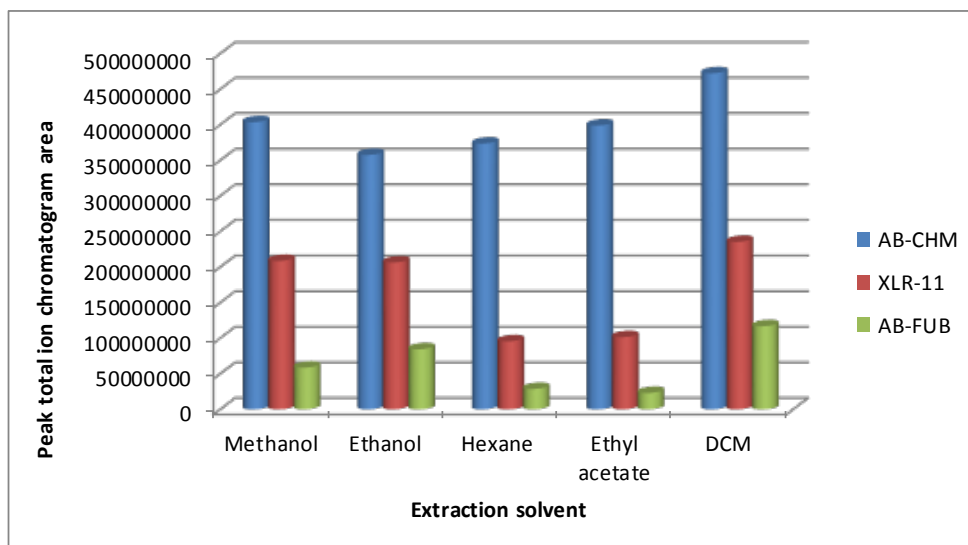| Synthetic drugs	Retention time	Mass fragments (m/z)
XLR-11	15.24 min	
XLR-11 Degradant	15.24 min	232,144,314
AB-CHMINACA	18.18 min	241,312,145
AB-FUBINACA	16.44 min	
AB-FUBINACA isomer 1	16.42 min	
AB-FUBINACA isomer 2	16.42 min	
AB-FUBINACA isomer 3	16.42 min	109,252,324
AB-FUBINACA isomer 4	16.42 min	
AB-FUBINACA isomer 5	16.42 min	
AB-FUBINACA isomer 6	16.42 min	

Fig. 2. Extraction solvent type for spice or herbal smoking mixtures Vs area response for (XLR-11, AB-CHMINACA, AB-FUBINACA isomers)
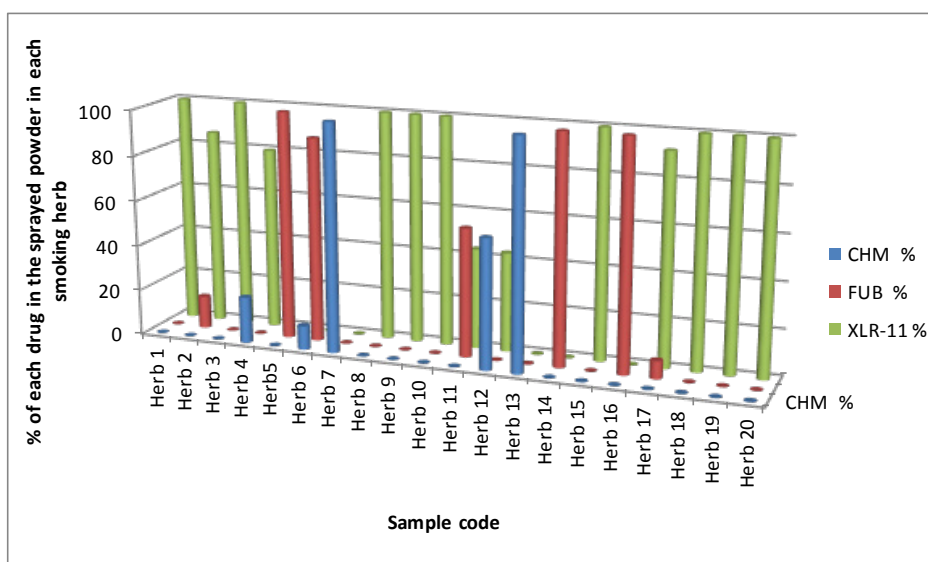

Fig. 3. Percent of each drug in the sprayed powder of each smoking herbal mixture.

samples Herb 2, Herb 3, Herb 6 and Herb 13 with the corresponding mass-spectrum for each component or drug. Where chemical structures and mass fragmentation patterns are indicated for the samples and are reported in previous studies [19-21]. Nicotine detected in the samples is coming from the tobacco which is used as the blending herb. Neophytadiene also showed up in few samples like herb 13 as indication for using cured and aged tobacco. Glycerin also showed up in herb 13 where often sprayed tobacco of the drugs' mixture of powder is mixed with glycerin to make the sample a little bit saggy or wet to be smoked by water pipe (Hookah). Cigarettes gave

nearly identical results since they were almost packed from same spices packing.

Cigarette waste

GC-EI/MS integration results indicated that methanol is the most appropriate solvent for extraction of our target cannabinoids (XLR-11, AB-CHMINACA, AB-FUBINACA) from cigarettes' filters as indicated in Fig. 8 and Table S3. Although methanol was a good solvent for the targeted cannabinoids but had no effect to dissolve the cellulose acetate fiber filters in cigarettes in contrast to dichloromethane. The results of the 20 samples of cigarette waste
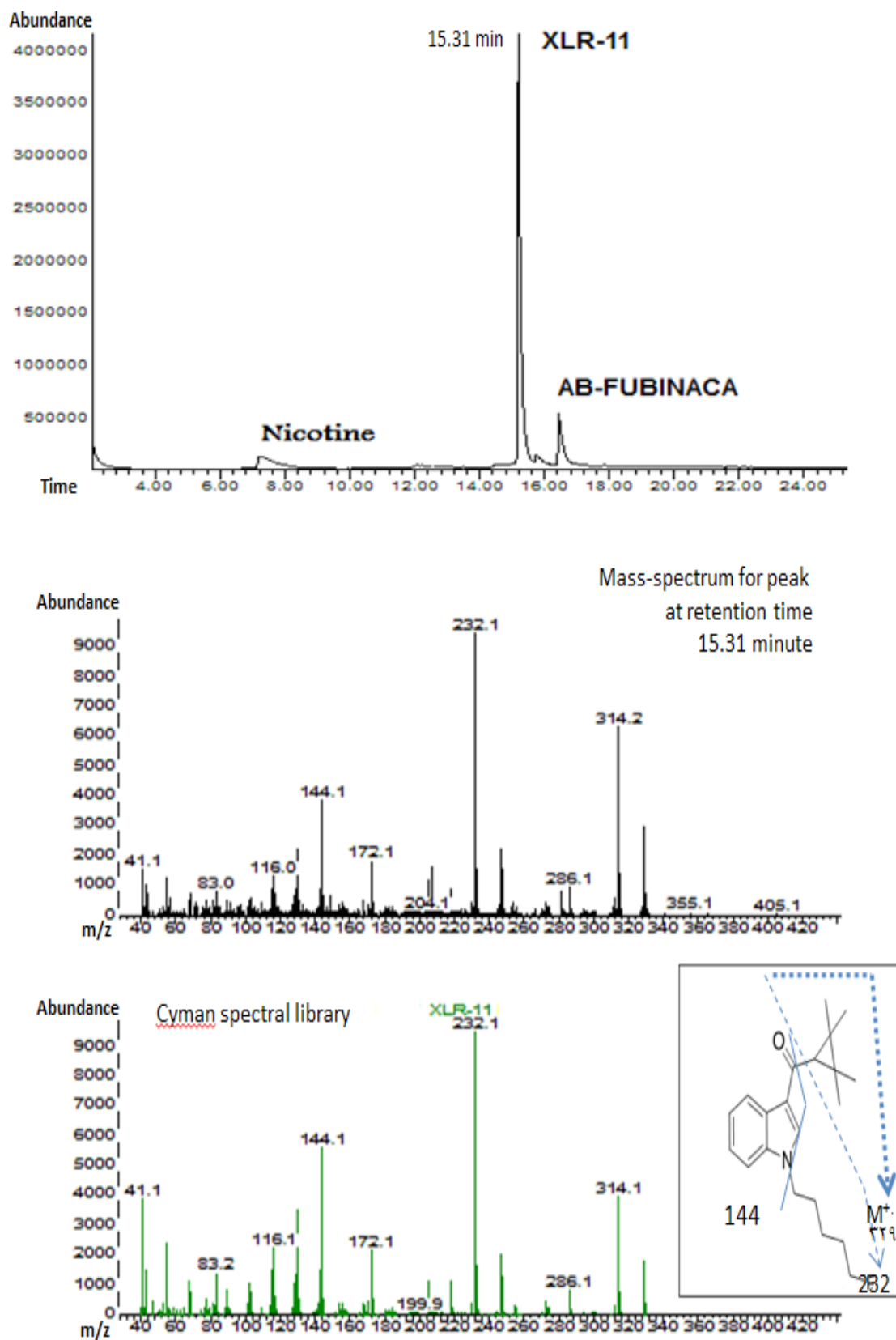
Fig. 4. GC-EI/MS chromatogram for sample coded (Herb 2) with the corresponding mass spectrum for peak at retention time 15.31 minute (XLR-11).
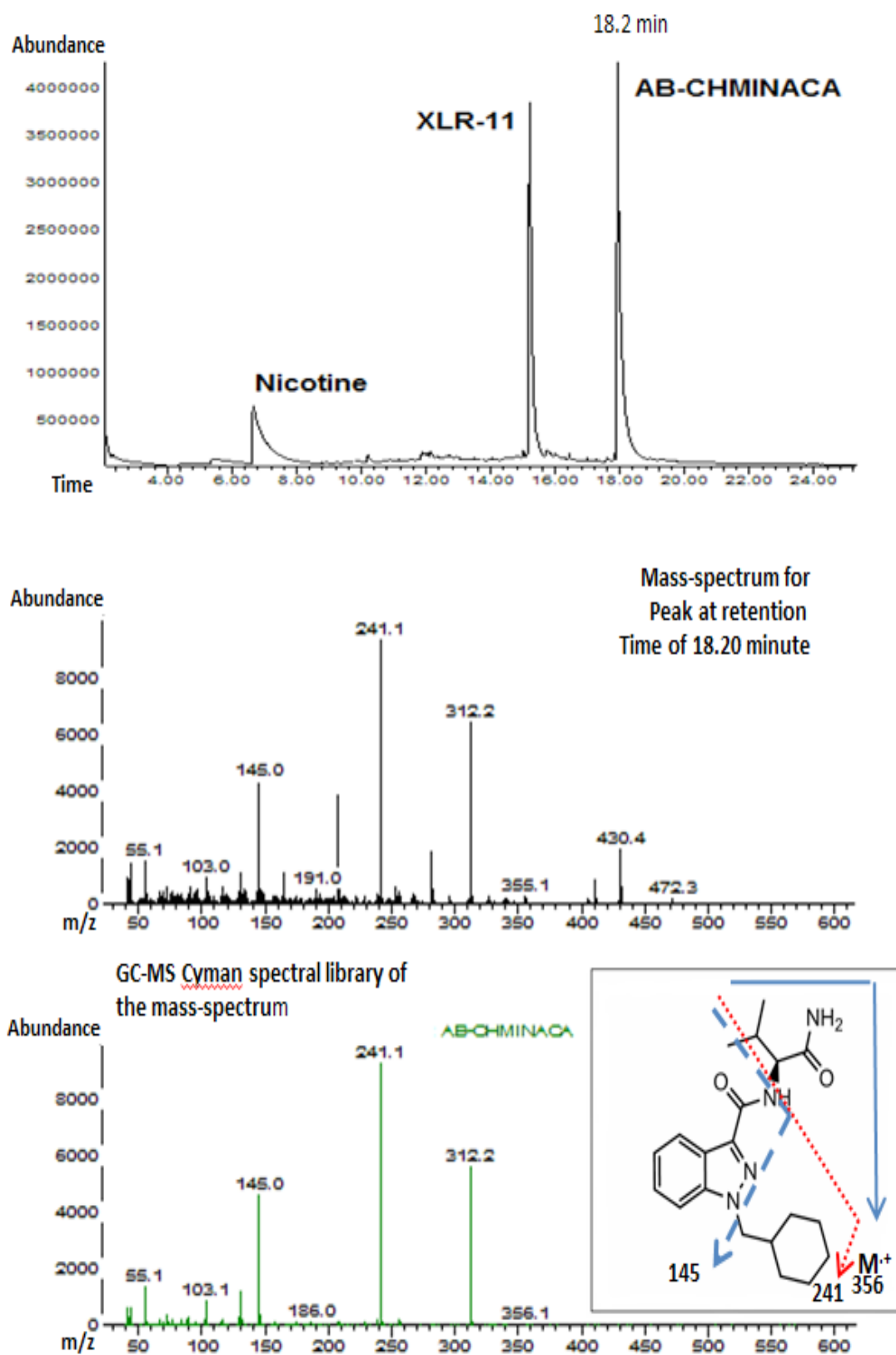
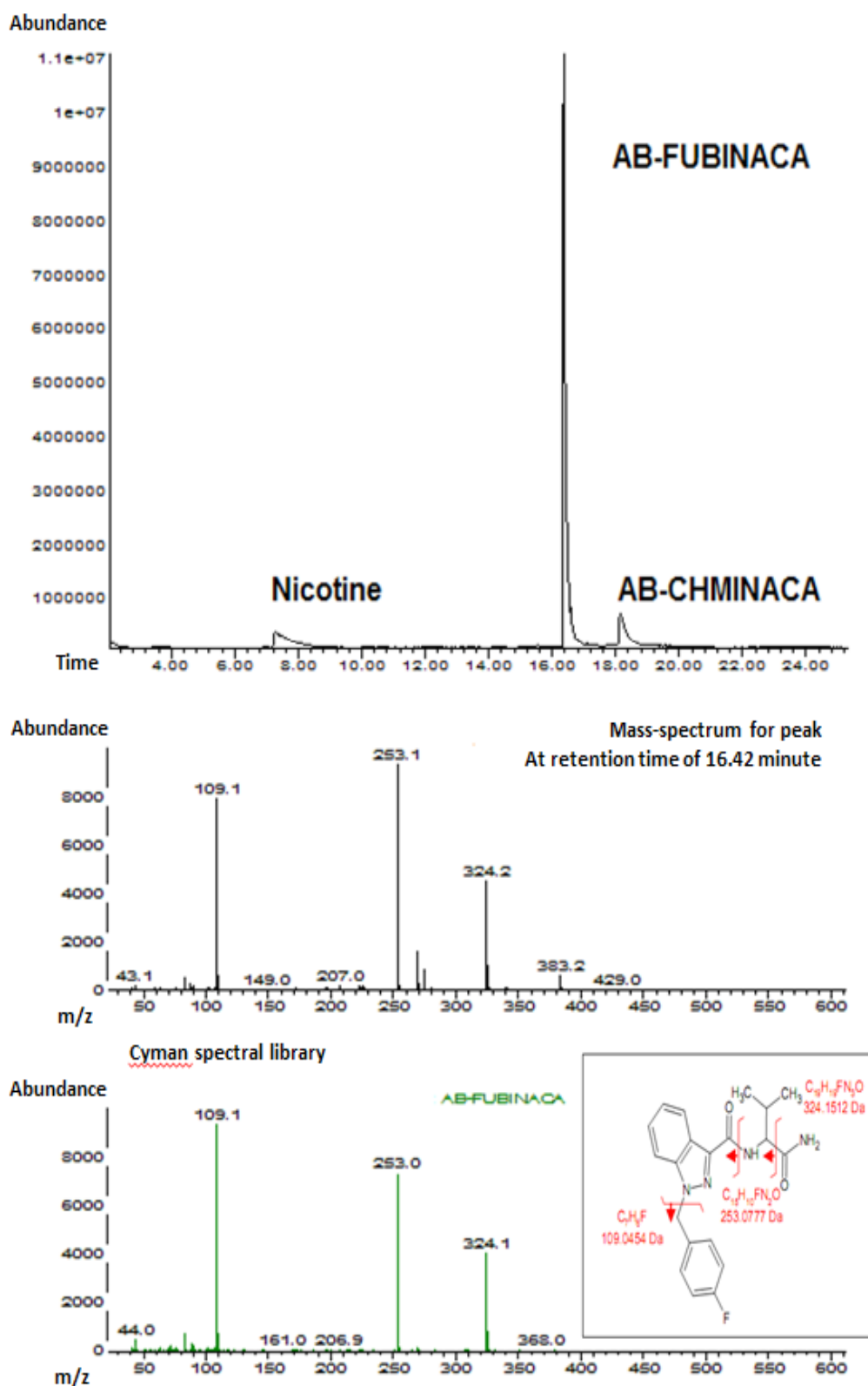**Fig. 5. Spectrum of peak at retention time 18.20 minute (AB-CHIMNACA)**

Fig. 6. **GC-EI/MS chromatogram for sample coded (Herb 6) with the corresponding mass-spectrum of peak at 16.42 minute (AB-FUBINCA).**

extracted by methanol and analyzed by GC-EI/MS are shown in Fig. 9 and Table S₄. Where the percentage of each drug in the sprayed powder on each cigarette waste is indicated based on the area ratios of the corresponding peak for each drug in cigarette waste to the total area of the three drugs. Most samples indicated the presence of XLR-11 degradant where it is expected due to the smoking process (burning and heat) when XLR-11 is converted to XLR-11 degradant where compounds containing cyclopropyl group undergo thermal degradation leading to a thermodynamic product where the cyclopropyl ring was thermally opened [19] where m/z fragments 247, 314, and 329 are noticed with higher abundances compared the intact compound XLR-11. The structure of XLR-

11 degradant gives rise to a prominent fragment ion that is 15 amu greater than the base peak of XLR11. This pattern is consistent with McLafferty rearrangement of the degradant which does not occur with the parent compound [22-24]. Fig. 10 shows the GC-EI/MS chromatogram for sample C.W 2 as an example with the corresponding mass-spectrum.

GC/MS instrumental precision was checked by injecting each sample in triplicate, after that the present relative standard deviation was obtained for each three runs for the three cannabinoids under investigation (XLR-11, AB-FUBIACA, AB-CHEMINACA) where in general RSD < 10% as acceptance criteria. As a result, it is shown that
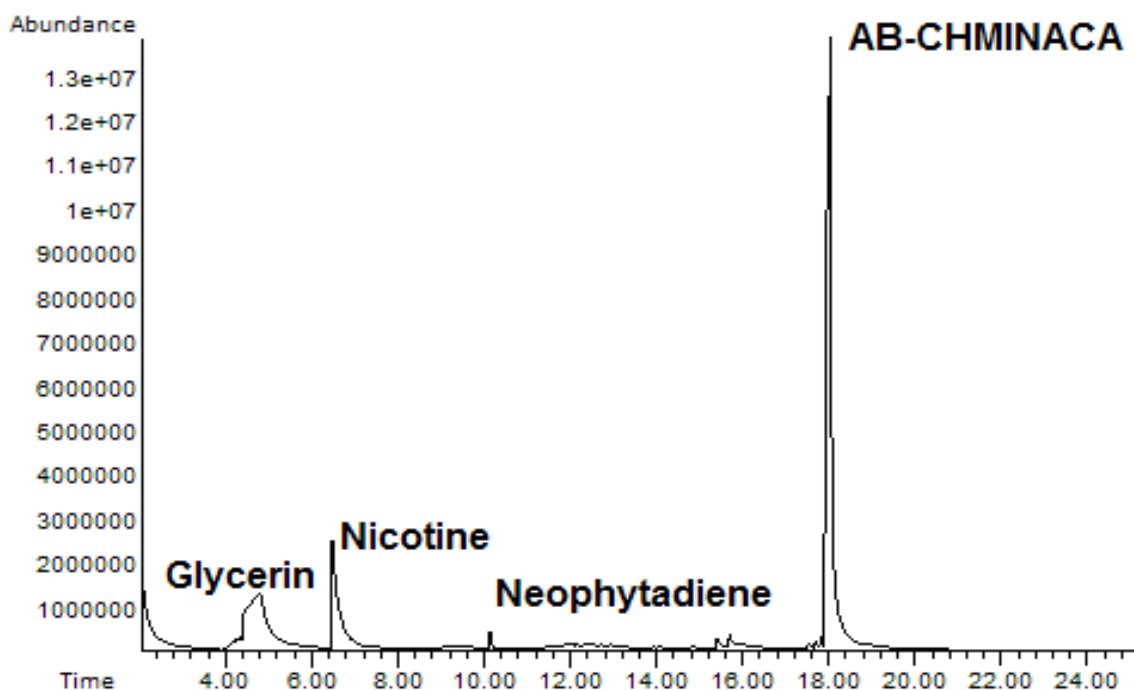

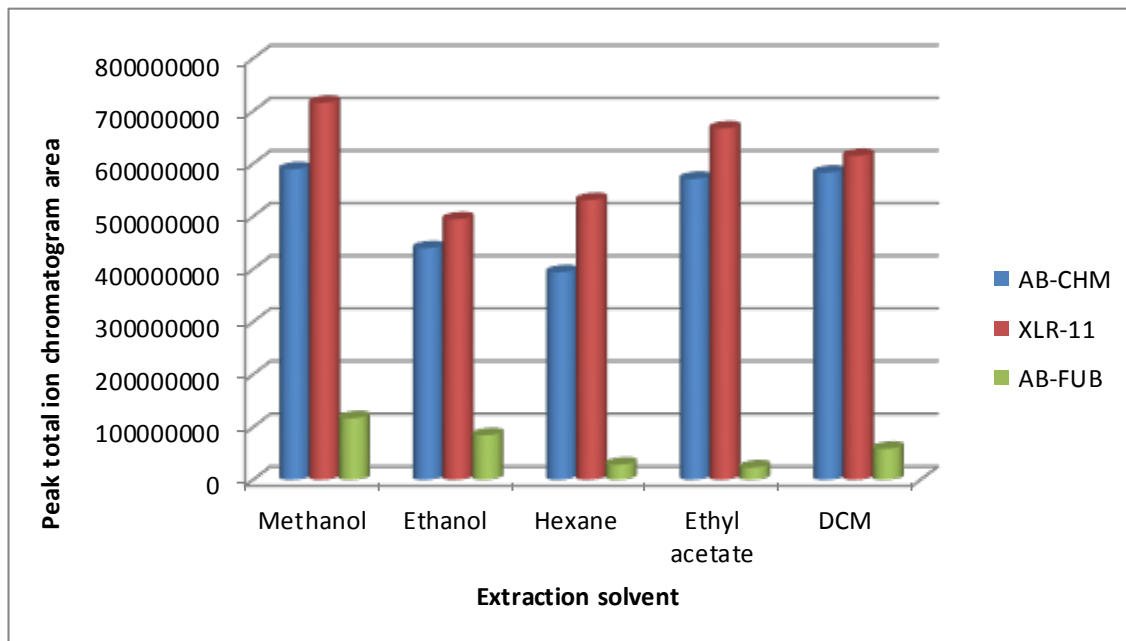**Fig. 7: GC-EI/MS chromatogram for sample coded (Herb 13).**

Fig. 8. Extraction solvent type for cigarette waste vs area response for (XLR-11, AB-CHMINACA, AB-FUBINACA isomers)
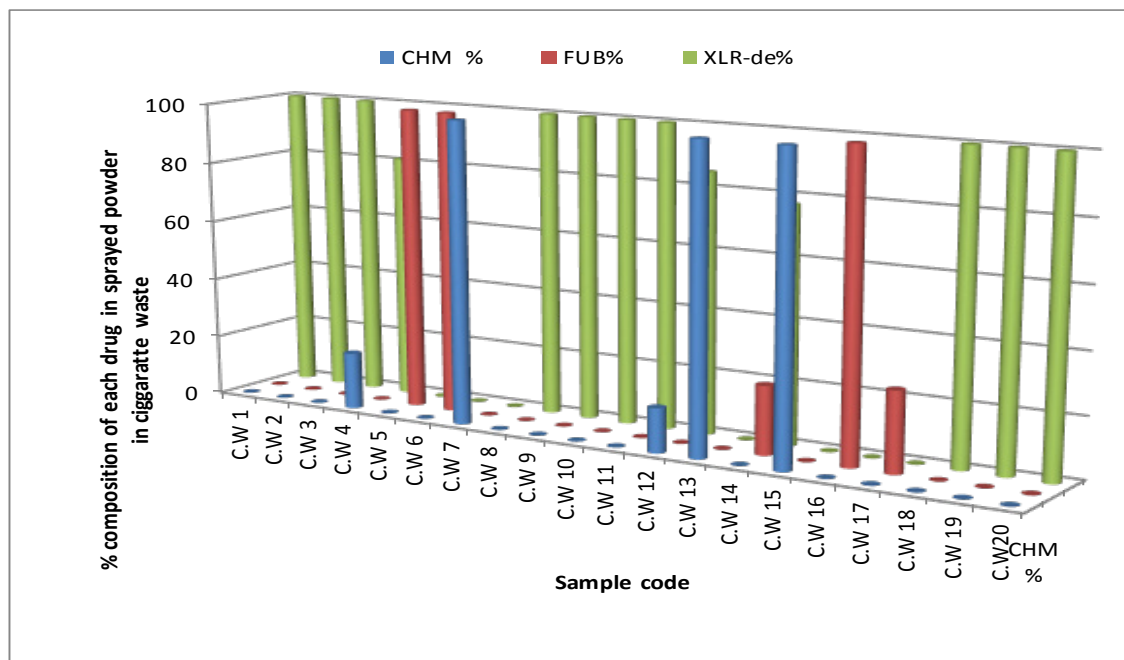

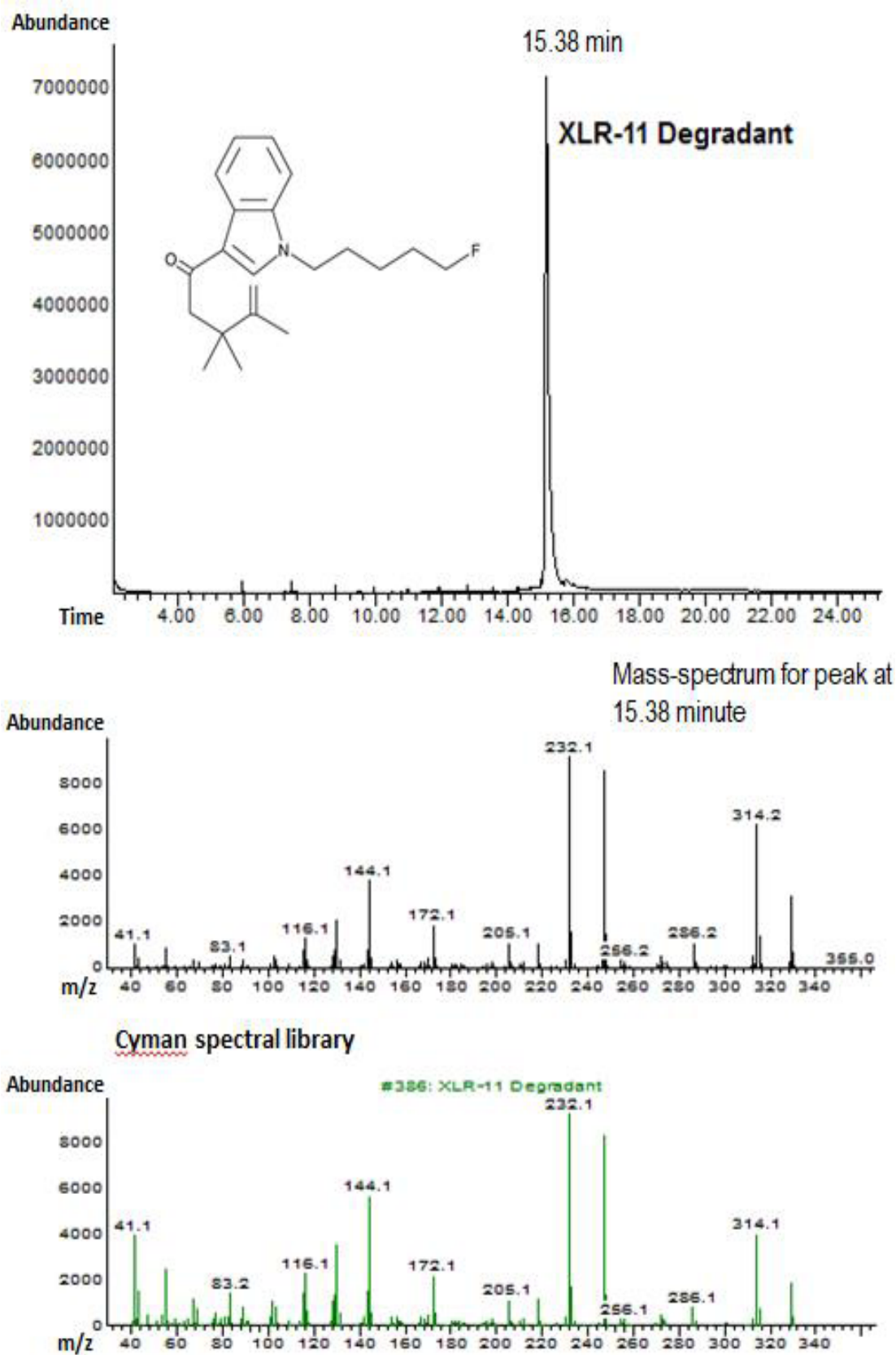**Fig. 9. Percent composition of each drug in sprayed powder in cigarette waste.**

Fig. 10. GC-EI/MS chromatogram for sample coded (C.W 2).

TABLE 2. GC-EI/MS results for triplet injection samples and RSD.

Herb 1		Herb 5	
Injection #	Peak area	Injection #	Peak area
1	110044285	1	315755277
2	112910722	2	310609755
3	115475675	3	316761375
Mean	112810227	Mean	314375469
RSD	2.41	RSD	1.05

Herb 13		C.W 4	
Injection #	Peak area	Injection #	Peak area
1	85757993	1	448422675
2	83746321	2	458061147
3	86682179	3	467756920
Mean	085395497	Mean	458080247
RSD	1.76	RSD	2.11

C.W 6		C.W 8	
Injection #	Peak area	Injection #	Peak area
1	353733879	1	177288575
2	328372538	2	187198948
3	342049191	3	181845250
Mean	341385202	Mean	182110924
RSD	3.72	RSD	2.72

all values have RSD less than 10% as shown in Table 2.

Conclusions

This study indicated that the best solvent for extraction the targeted Cannabinoids in herbal smoking mixtures was dichloromethane, and for cigarette waste cellulose fiber filters was methanol and it had no effect on dissolving the filters. Concerning the newly marketed synthetic cannabinoids for the year 2016/17 that are belonging to the aminoalkylindols class; the GC-EI/MS data indicated that almost all herbal smoking mixtures and loaded cigarettes seized by customs and Antinarcotics Department are containing the recently new marketed synthetic cannabinoids (XLR-11, AB-CHMINACA, AB-FUBINACA isomers). These marketed herbs were sprayed with one cannabinoid either XLR-11 (45%) or AB-CHMINACA or AB-FUBINACA isomers (30%) or combination of either XLR-11 or AB-CHMINACA or a combination of XLR-11 and AB-FUBINACA isomers (25%). Herbal smoking mixtures sprayed with the XLR-11 were the one with largest quantities seized, reflecting their doping in the locally and by surrounding countries to Jordan. Supporting to this finding; the results of analyzing cigarette waste samples seized from people charged with doping indicated that most of these filters contained XLR-11 and its degradant (reported as XLLR-11 degradant) (around 50%) or AB-CHMINACA or AB-FUBINACA isomers as individual components (35%) and to a lower extent mixtures with the other two cannabinoids (15%). The banning action in Jordan in 2016 for these three recreational drugs and the intensive investigation for the components of smoked herbal material changed the pattern for available marketed designer drugs entering Jordan for the year 2017 like α-Pyrrolidinopentiophenone (PVP) or flaka and AMP-PICA, which mandates continuous update and identification for new drugs entering the market.

Conflict of interest

None

Appendix A: supplementary data

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تعرف على مركبات الحشيش الصناعي (الامينوالكل اندولز) في منتجات التدخين باستخدام
كروماتوغرافيا الغاز – مطياف الكتلة واستخلاص السائل بالسائل

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في هذا العمل اجريت دراسة حالة في المملكة الاردنية الهاشمية للكشف عن مخاليط التدخين للتعرف على
مركبات الحشيش الصناعي الجديدة (الامينوالكل اندولز) وهي أكس ال ار-11، أب-فابيناكا و أب-كاميناكا
والتي كانت مسؤولة عن عدد قليل من حالات الانتحار ، اعمال عنف فردي وعدد قليل من جرائم القتل. تم ضبط
وتحريز العينات من قبل الجمارك واقسام مكافحة المخدرات في عام 2016/2017. تم فحص الأدلة الجنائية
المتضمنة عينات مخاليط التدخين العشبية، والسجائر ، ومخلفات السجائر للكشف عن هذه المخدرات المحظورة
باستخدام كروماتوغرافيا الغاز –مطياف الكتلة GC-EI / MS من خلال طريقة المسح العام باستخدام مادة
جي دبليو اتش -18 و 5-فلورو أ د ب كبدلاء ومعايير مرجعية للامينوالكل اندولز. باستخدام تقنية استخلاص
السائل بالسائل تبين أن ثنائي كلورو الميثان أعطى أعلى مستوى كفائة استخلاص للامينوالكل اندولز في مخاليط
التدخين العشبية والميثانول لمخلفات فلاتر السجائر. أوضحت النتائج أن كل الأعشاب التي تم تسويقها قد تم
رشها باستخدام اكس ال ار -١١ (حوالي ٤٥٪) أو مزيج من كلا اكس ال ار-١١ و أب-كاميناكا   أو مزيج من
أيزومرات أب-فابيناكا و اكس ال ار-١١ و كان الباقي أيزومرات أب-فابيناكا و أب-كاميناكا بصورة نقية أو
مخاليط. تم تأكيد هذه النتائج بشكل أكبر من خلال تحليل مخلفات السجائر التي أعطت نتائج تقريبية للأعشاب
أو التوابل. على حد علمنا هذه هي الدراسة الأولى من نوعها في منطقة الشرق الأوسط وشمال إفريقيا والتي تم
إجراؤها في الأردن باستخدام طريقة لم يتم ذكرها في الأدبيات.