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RESEARCH ARTICLE

Determination of Extracted Methamphetamine from Hashish Narcotic Plant by Home-made Ion chromatography System

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Abstract

A fast method was described for the determination of [(S)-N-methyl-1-phenylpropan-2-amine] methamphetamine (MMPs) as micro crystalline standard mp170-175^{o}c dissolvent in Ethanol comprehensive with extraction (MMP_{Ext}) from plant, this method using IC-UV. It is based on the spectrophotometric UV in max wavelength 250nm. The (MMP) spectra of this system was examined under the optimum conditions to obtained a good truly result for active material (MMP) from standard material and extraction plant. The detection limit (S/n=3) is 1 mg/l with relative standard deviation is 0. 19% for five replicates of 6mg/l MMPs.The linearity was in the rang 2-5 mg L^{-1} with a correlation coefficient R^2 0.999. The method has been successfully applied to the determination of (MMP) in standard (Microcrystalline) and plant extraction preparation using standard addition method and the recoveries were in the rang 97.5 % - 100.0 %

INTRODUCTION

Hashish narcotic plant are a group of natural products that are containing active compound Methamphetamine (MMP) Fig.1. Many therapeutic functions have been described to these compounds, which recognized the role of MMP as the main therapeutic for many diseases[1], Protective effects of MMP against serious disorders such as cancer, hypertension ,nerve and vomit disease have been also recognized[2]. MMP is one of the most commercially important and widely utilized sympathetic nerve. It is used as antioxidant medicine therapeutic agent, and an important safe pro-vitamin source made[3]. Most MMP is naturally present in the plant extraction form; however, some amounts of the extract compound form of Hashish are also present in some types of Taboco. Figure 1 shows the structures of MMP were separated on Ion Pac Arcus EP - C18 ; 5µm column, which is designed to provide high shape selectivity for separation of hydrophobic structurally related isomers and unique selectivity complementary to other reversed-phase columns[4].Extract able are defined as compounds that can be extracted from describe plant drug delivery device components to use as a medicament. Routine extractable testing is performed on device components to control each within the final product[5].

Routine extract able testing has been performed using Soxhlet and reflux extraction. These techniques have disadvantages associated with the handling and disposal of significant volumes of potentially flammable and hazardous organic solvents. Extractions usually proceed for 24 h and therefore must be left an attended.

Arrays of extraction apparatus consume valuable bench space. Accelerated solvent extraction is an established technique and an improvement over these traditional extraction techniques. It is a powerful technique to reliably

extract compounds from Narcotic plant materials. and use organic solvents at 30^{o}c temperatures above their atmospheric[6] .

Pressure boiling points to deliver extractions equivalent to traditional techniques, but in a shorter perior of time, This outlines the use of accelerated solvent extraction in the routine extract able analysis of atypical elastomeric device component.

Isocratic elution is used to separate the common mono-valent cations [Benzo-mono-methylamine (MMA)]with based organic compound. Divalent cations in the sample will be retained on the column along time. Minimum detection limits range from about 0.5 µg /ml in a 100-µL sample injection. The working range of concentrations extends to about 2.0-10.0 µg /ml . The low detection limits make this a quick, direct method . for quality control analysis in drugs manufacturing [7] . The most important applications of chromatography (IC-uV) is the determination of MMP compound in Microcrystalline and active material in Hashish plant. Approximately 25% of drugs on the market are developed in salt forms certain physicochemical and biopharmaceutical properties of active pharmaceutical developed salt (APD) can be improved by pairing a basic or acidic drug.[8,9] . The drug with high solubility, stable microcrystalline from, and good bioavailability. Chromatography techniques with UV detection plays an important role in the Hashish plant by 250 nm wavelength , pressure 170 bar and $25C^{o}$-$30C^{o}$ with Ion Pac Arcus EP - C18 ; 5µm , 4.5×250 mm *(P/N* 11051194 L*)* also flow APPL spectrophotometer system PD 303UV with flow rate 1.0 ml/Min was used. The isocratic 30mM,mixture of 10% ,40% and 50% from acetonitril ,Ethanol and DI water respectively . The microcrystalline as standard and Hashish plant is an important traditional chine's medicine used for the treatment such a aliments as a cute fever, head ache, anti-emetic, anti-convulsing [10] .different HPLC methods were reported for separation and determination of MMP as The major active components in Hashish plant [11] .

These HPLC methods allow analysis at (MMP) samples by using flow PD-303UV system High sensitivity and] Accurate [13,14] measurement for (MMP) in plant matrices require a robust (MMP) extraction method and a sensitive analytical method for (MMP) quantification. Analytical method that have been used for determining (MMP_{EXT}) in plant matrices include: colorimetric based on catalytic reactions microscopic 8 MPX, IC with UV detection as absorption spectrometry [15] . A number at (MMP) extraction methods have also been developed for (MMP) from Hashish plant – Microwave digestion in open or closed vessels with perchloric acid, tetramethyl ammonium hydroxide, Oxygen combustion, alkaline extraction, acid digestion (Hydrochloric acid, acetic acid) [16], precipitation with evaporation methanol acetonitrile and ultra-centrifugation, but the mean method is UV detection . (IC-uV) has the advantages at 1.0 ml / min flow rates and low eluent consumption and available IC system without additional consumable replacement costs. The (IC-uV) system is designed to run continuously, saving time spent on equilibrating and recalibration typically need after each start up[17,18].

Goal

To develop an efficient and comprehensive IC - UV with semi-automated system for identification and determination of derivative amine MMP in Hashish extracted , biochemical ,pharmaceutical and antibiotic.

Experimental :-

Ion Pac Arcus EP - C18 ; 5µm , 4.5×250 mm *(P/N* 11051194 L*)* was chosen for this separation which phenyl groups are structurally similar to the phenyl groups and aromatic structures contained in the MMP. The MMP separation in the sample can be completed within 18 min as Run time by using a 10% acetonitrile ,40% Ethanol , 50%, DI. Water as amixture eluent with 30 mM methane sulphonic acid .

Equipment:-

A home-mad IC-UV system including

- LKB Bump 2150 –HPLC ,Bromma
- Ion Pac column Arcus EP - C18 ; 5µm , 4.5×250 mm *(P/N* 11051194 L*)*
- Metrohme Electric injection valve with 100 µL loop inject in system before flow cell quartz.
- A PD 303 UV Detector
- Equipped with 18µl flow cell (Helma . uk)

Reagents and standards :-

- Acetonitrile (CH_3CN), HPLC grade ,BDH chem.LTD 562240A
- Deionized (DI) water, 18.2Ω-cm resistivity.

- Ethanol (C_2H_5OH), HPLC grade ,BDH M/ 405/17 LTD 116967 Cas 67-56-1
- Methane sulfonic Acid (MSA), analytical grade, Aldrich;51,684-8 LTD ;S 67573
- Hashish narcotic plant and Microcrystalline methamphetamine as standard

This reagents and the prepared standards can be used freshly [19] .

Sample preparation:-

Hashish plant and the (MMP) sample were supplier from Basrah Governorate police / Criminal Evidence Dept . / Criminal Lab. by document No.1032 in 18- 1- 2015.

Accurately 5g of sample mass was placed in a 100 ml volumetric flask. Add 30 mL of Ethanol / water (1:1 V/V) after 48 hours in an stirrer Bath, cool to Room temperature filter through a 0.4 um membrane prior to injection [20]

The step two after extraction plant is IC method to develop an efficient and quality control method parameters , Table 1 illustrated summarized the resolution and repeatability favorable for the home – made uv-IC. The System suitability of the proposed method was studied through method development by calculating theoretical plates, peak symmetry factor and tailing factor.

Result and Discussion:-

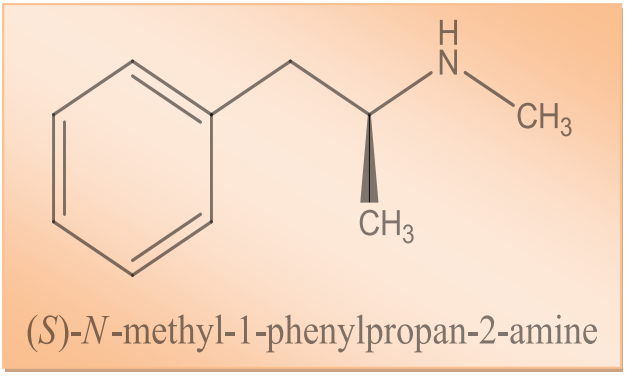

Figure: 1 MMP structure

Table 1: method parameters

Parameters	***Conditions***
Description Column	***Ion Pac Arcus EP - C18 ; 5µm , 4.5×250 mm (P/N 11051194 L)***
System Suitability Requirement	***USP Tailing Factor @ 5 %Peak Height < 1.3 Resolution between any analyte and I.S. is > 1.4 Plates > 16000- 16,810***
Isocratic Mobil phase	***10% acetonitril ,40%Ethanol ,50% DI Water +30 mM methansulfonic acid***
Detection System	***UV detection***
Wavelength Maximum	***250 nm***
Flow Rate	***1.0 mL / min***
Temperature	***25- 30 °C***
pressure Background	***170 Bar***
Run Time	***18 min***
Injection Volume	***100 µL***

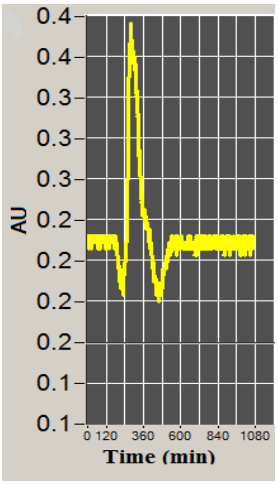

Figure2: Chromatogram of MMPs

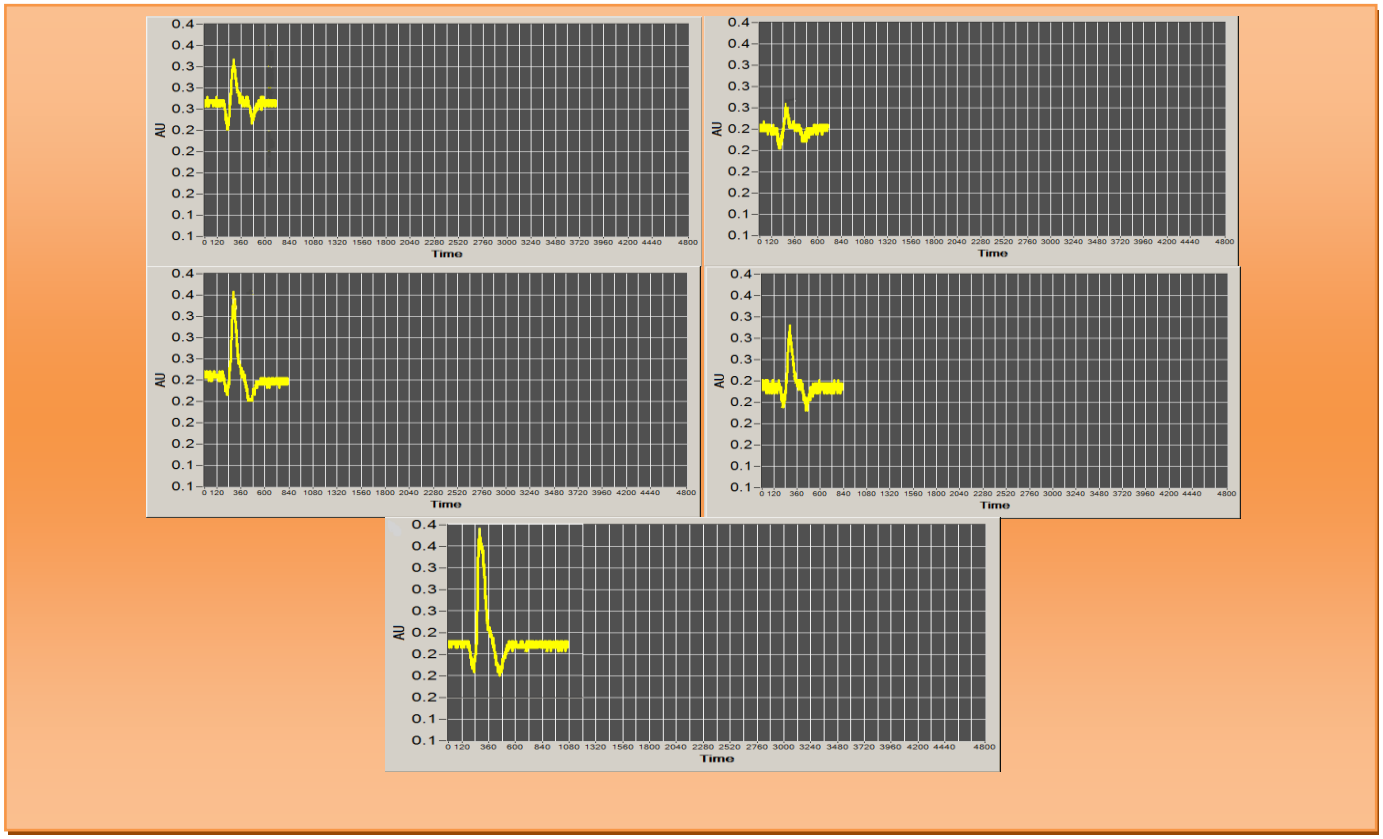

Figure3: Linearty chromatograms of MMP

Table 2: Regression statistics of the proposed method with LLOD,LLOQ, Intercept and Slope for Microcrystalline (MMP_S) material as standard and (MMP_{EXT}) from extraction narcotic plant .

compound	Retention Time(min)	R^2	Std Er[a]	Std Er Est[b]	Intercept	Slope	LLOD ng mL^{-1}	LLOQ ng mL^{-1}
MMP_S	4	0.999	0.766	0.730	-0.381	55.142	9.60	32.00
MMP_{EXT}	4	0.997	.7310	0.695	-0.382	05.142	9.50	31.66

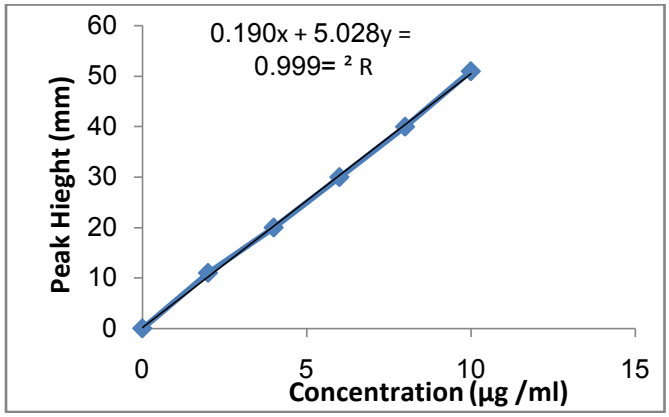

Figure 4 :Least squares calibration graph

Table 3: Method accuracy from recovery assays for the studied compounds analyses.

Concentration Range (µg mL^{-1})	Recovery ± r.s.d %		Recovered conc. (µg mL^{-1})		%RSD	
	MMP_S	MMP_{EXT}	MMP_S	MMP_{EXT}	MMP_S	MMP_{EXT}
2	**100.0**±0.14	**95.0**±0.13	2.00	1.90	0.24	0.14
4	**97.5**±0.12	**93.3**±0.12	3.90	3.80	0.19	0.04
6	**100.0**±0.12	**100.0**±0.12	6.00	6.00	0. 10	0.15
8	**97.5**±0.11	0.11**98.7**±	7.80	7.90	0.64	0.52
10.0	**100.0**±0.13	**98.0**±0.11	10.0	9.80	0.01	0.13

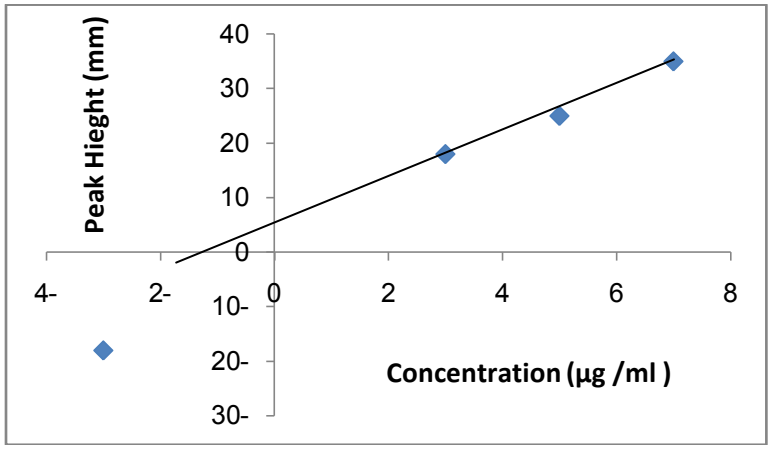

Figure 5:standard addition method

Table 4: Intra and inter-day precision and accuracy of MMP_S analysts (n=3) home-made UV-IC

Added ($mg\ l^{-1}$)	Intra-day			Inter-day		
	Found	%Rec	%RSD	Found	%Rec	%RSD
2.0	2.00	100.0	0.24	2.0	100.0	0.25
4.0	3.90	97.5	0.19	3.9	97.5	0.04
6.0	6.00	100.0	0.10	5. 8	96.6	0.09
8.0	7.80	97.5	0.64	7.8	97.5	0.65
10.0	10.0	100.0	0.01	9.9	99.0	0.01

1- Effect of concentration eluent on the separation and retention time :-

A Series of experiments were establish to find the Optimum eluent concentration . Figure 2 shows the chromatogram standard obtaind by inject 0.5 mg/l (MMP) on a C_{18} column into the eluent with concentration of (10% CH_3CN, 40% C_2H_5OH,50% $DI.H_2O$ + 30mM CH_3SO_3H). The (MMP) peak is well resolved in less than 5 min from the void volume. The baseline dip at approximately 1 min is due to dissolved Oxygen from the previous injection and mixed effect between Ethanol and acetonitril in the eluent [21]. One peak appearance in chart ,the mean cause of separation is a properties column and type eluent ,the peak refers to (MMP_{EXT}) Methamphetamine which is mean studies , after extract and purity material that is other compound extraction within mean peak ,this peaks unknown didn't important now in my studies. Also figure 2 shows one peak appearance which indicated that the C18 column and type concentration of eluent suitable for MMP separation . so this concentration was used in subsequent work .

2- Effected Column Temperature on the separation at (MMP) Active components:-

The effect column temperature in the range 25-45C^o on separation of MMP was evaluated .As expected increasing the column temperature decrease the Retention time and led to good baseline for the separation chromatogram due to difficulty of maintain temperature stability in the home – made IC system So 25^{o}c was chosen in the future work . Under the condition establish a calibration graph for MMP was obtained .It is linear in the rang (2-10) µg / ml Typical calibration results are shown in figure 4.The linear graph has a regression coefficient of (0.999) for five points . Table 3 reports data from the calibration as calculated by the SPSS CD software.

Sample Analysis :-

Chromatograms of a (MMP) sample as well as comparison of Peak and retention time allows the identification of (MMP), recoveries for standard sample ranged from 93.3-100.0% suggesting that the analysis method is accurate [23]. The results are shown in Table 3 .

Accuracy :-

To evaluated the accuracy of the home-made IC system .A recovery experiments were performed on MMP_{EX} and a representative samples by using a standard additions method for all these determinations to avoid the interferences effect Figure 5 and Table 3 reports the data obtained which were calculated by using SPSS software . The average recoveries were in acceptable range (99.3-100.0 %) which clearly indicated that method can be used successfully for determination MMP In order to establish the validity of the home – made IC system . The same batch of representative samples were analysed by the IC system and classical method or matching the results with Criminal Evidence Dept. – Criminal lab .)

A good agreement between the results clearly indicated that the made-home IC system can be used for several applications . Also the Intra-day and Inter-day precision and accuracy of MMPst analysis Table 4 listed these results which clearly indicated that IC-UV method can be used for accurate and precision determination of MMPst and MMPext The matrix of extracted plant does not effect this determination. [24].

Conclusion :-

This work describes an IC method that baseline separate one active components in Hashish narcotic plant using extraction method before separation (MMP) using C_{18} column 4.6×250 mm, 5µm and mixture eluent (10%CH_3CN + 40% C_2H_5OH + 50% DI. Water 18.2 Ωcm^{-1} + 30mM CH_3SO_3H) as mobile phase. This method can be used for the quality control at Hashish plant a common medicinal plant in many states. It is superior to the IC method that measures only one purported active components with spirited method that require long separation times and have insufficient peaks.

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