



# QUALITATIVE ANALYSIS FOR ILLICIT DRUGS AND NPS (NEW PSYCHOACTIVE SUBSTANCES) USING GC-MS

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This research aims to analyze 500 samples for illicit drugs and NPS (New Psychoactive Substances) using GC-MS (Gas Chromatography Mass Spectrometry) in East Java Indonesia. Sample analysis was carried out by GC-MS with HP-5 column, injection port temperature of 250°C. Column temperature was maintained at 100°C for 2 minutes. It was then raised from10°C/minute to 280°C and maintained for 5 minutes Based on the results, it was detected 244 (48.8 %) Methamphetamine (MA); 30 (6 %) 3,4-Methylenedioxymethamphetamine (MDMA); 44 (8.8 %) 3,4-Methylenedioxyethylamphetamine (MDA); 56 (11.2 %) 3,4-Methylenedioxyethylamphetamine (MDA); 56 (11.2 %) 3,4-Methylenedioxyethylamphetamine (MDEA); 106 (21.2 %) AB-Fubinaca; 5 (1.0 %) Tramadol; 5 (1.0 %) Ketamine; and 10 (2.0 %) Mephedrone. The identification of illicit drugs and NPS in confiscated materials was successfully achieved via GC-MS. The availability of GC-MS libraries is of great assistance in the identification of new drugs. Alternatively, the study of characteristic molecule fragments combined with the determination of their accurate masses can be a useful approach to identify unknown samples that were not previously analyzed.

Keywords: Qualitative Analysis, illicit drugs, NPS, GC-MS

#### **INTRODUCTION**

illicit drugs market are a troubling phenomenon in International and National scope, especially in, East Java, which remains as the second highest in Indonesia after Jakarta.<sup>1</sup> Similarly, the detected of New Psychoactive Substances (NPS) will increase its level in not only the national community, but international community are also likely to pose a more significant social problem in the future.<sup>2-5</sup> NPS (New Psychoactive Substances) are compounds that affect the central nervous system and make potential addiction. However, its use has not been regulated in the United Nations Single Convention on Narcotic Drugs of 1961, the 1971 United Nations Convention on Psychotropic Substances. There are some

groups of NPS, synthetic cannabinoids derivatives, cathinones derivatives, ketamine derivatives, phenethylamines derivatives, piperazines derivatives, and plant-based substances which are deliberately developed to deceive the law.<sup>6-8</sup>

During the past decades, NPS has been introduced and traded through various distribution modes, including the internet. This has increased the demand and number of users throughout the world, thereby making it a serious threat to humanity.<sup>4,8,9</sup>

The existence of NPS in Indonesia became publicized in 2013 at the time an Indonesian actor used drugs containing 3,4methylendioxymethcathinone. In 2015, new and unregistered evidence containing mephedrone/4-MMC was found and in early

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2017. "blue sapphires" containing 4chloromethcathinona/4-CMC were found in several cafes in in East Java. In 2016 and early 2017, the public was confronted by the trading of AB-Chimanaca and AB-Fubinaca in gorilla tobacco, which is a type of synthetic cannabinoids. Based on the World Drugs Report 2014, UNODC noted a 50% increase in the synthetic cannabinoids trend compared to other new substances.<sup>1</sup> In 2020 until 2022, AB-Fubinaca detected in Indonesia were increase. many cases reported people used it as illicit drugs.10

GC-MS is a method that is often used to detect the presence of NPS in powders, crystals, tablets, and tobacco samples. GC-MS is still able to detected 500 samples of NPS with high specificity, selectivity, accuracy and precision. These samples contain of synthetic derivatives cannabinoids (AB-Fubinaca); cathinones derivatives (Mephedrone); phenethylamines derivatives (amphetamine; methylamphetamine (MA); 3.4-Methylenedioxymethamphetamine (MDMA); 3,4-Methylenedioxyethylamphetamine (MDA); Phencyclidine/Ketamine derivatives, and semi opiate (tramadol). This article study showed chromatogram and mass spectra of 500 samples of NPS from some dosage form such as tablets, capsules, powders,; crystal powders, and some of simplicia.

#### EXPERIMENTAL MATERIAL AND METHODS

All reagents and solvents such as ethyl acetate was purchased from standard commercial suppliers from Merck pro analysis grade, with samples obtained from the Surabaya Forensic Laboratory in the form of powder, crystals, tablets, capsules, crude material.

#### Sample preparation

100 mg of the sample was dissolved in 10.0 ml ethyl acetate, and then centrifuged at 1000 rpm. It was filtered by 0.2 microns, and 1-2  $\mu$ l was injected into the GC-MS instrument.

#### Method of GC-MS analysis

Sample analysis was carried out by GC-MS with HP-5 column, injection port temperature of 250°C. Column temperature was maintained at 100°C for 2 minutes. It was then raised from10°C/minute to 280°C and maintained for 5 minutes. Data analysis was performed using GC-MS chromatogram approach which was determined by Library MS data from NIST or UNODC with fragmentation analysis from MS.

#### **RESULTS AND DISCUSSION**

#### Results

GC-MS are a reproducible method to quickly and accurately analyse illicit drugs and NPS (new psychoactive substances) on samples of tablets, capsules, and powder, crude material.11 LC-MS method is commonly used for routine analysis of NPS and illicit drugs in biological fluid analysis such as urine and blood specimens.<sup>11-13</sup> Narcotics Schedules I-V are compounds that are classified as thermostable such as heroin, lysergic acid diethylamide (LSD), marijuana (cannabis), methaqualone, and 3.4peyote, methylenedioxymethamphetamine that is called ecstasy. Therefore, they could be analysed using GC-MS.14-16.

Some crime scene evidence for illicit drugs such as powders, tablets, capsules, and crude material consist of synthetic derivatives cannabinoids (AB-Fubinaca); cathinones derivatives (Mephedrone); phenethylamines derivatives (amphetamine; methylamphetamine (MA); 3.4-Methylenedioxymethamphetamine (MDMA); 3,4-Methylenedioxyethylamphetamine (MDA); Phencyclidine/Ketamine derivatives an and semi opiate (tramadol) could be detected with high specificity and selectivity using GC-MS instrument. Validation method for analysing illicit drugs are necessary based on criteria acceptance of International Council for Harmonisation (ICH) consisting of specificity, selectivity, accuracy and precision, LOO (Limit of Quantification) and LOD (Limit of Detection). Illicit drugs and NPS (New psychoactive Substances) are major compounds, therefore, LOO (limit of Quantification) and LOD (Limit of Detection) are not necessary because they does not match the analysis of traces elements (heavy metals) or organic contamination compounds such as contamination of organic solvents, and pesticides.17

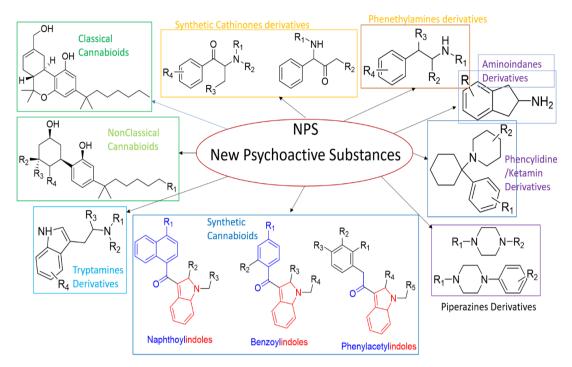


Fig. 1: The groups of New Psychoactive Substances (NPS).

The result of qualitative analysis on 500 samples consisting of tablets, capsules, powder, and crude material is shown in **Fig. 2**. Based on the results, it was detected 244 (48.8%) Methamphetamine (MA); 30 (6 %) 3,4-Methylenedioxymethamphetamine (MDMA);

44 (8.8%) 3,4-Methylenedioxyethylamphetamine (MDA) ;56 (11.2%) 3,4-Methylenedioxyethylamphetamine (MDEA); 106 (21.2) AB-fubinaca ; 5 (1.0%) tramadol; 5 (1.0%) ketamine; 10(2.0%) mephedrone that is shown in **Table 1**.



Fig. 2: The samples of Drugs of Abused and NPS.

| Samples | %<br>Samples | Compound                                       | TR<br>(Retention<br>Time) | Similarity<br>Index |
|---------|--------------|--|---------------------------|---------------------|
| 244     | 48.8         | Methamphetamine<br>(MA)                        | 5.7                       | 100%                |
| 30      | 6            | 3,4-Methylenedioxymethamphetamine<br>(MDMA)    | 10.92                     | 100%                |
| 44      | 8.8          | $H_2N$ $O$ | 7.05                      | 100%                |
| 56      | 11.2         | 3,4-Methylenedioxyethylamphetamine<br>(MDEA)   | 17.5                      | 100%                |
| 106     | 21.2         | AB-Fubinaca                                    | 27.5                      | 100%                |
| 5       | 1            | OH<br>N<br>Tramadol                            | 24.01                     | 99%                 |
| 5       | 1            | O NH<br>Cl<br>Ketamine                         | 23.12                     | 98%                 |
| 10      | 2            | NH<br>O<br>Mephedrone                          | 17.5                      | 99%                 |
| 500     | 100 %        | Total samples                                  |                           |                     |

Table 1: The result of Qualitative analysis on Drugs of Abuse and NPS Using GS-MS Instrument.

AB-Fubinaca contains cigarette or tobacco or simplicia. Samples were detected at 27.5 minutes (time retention) followed by other phytochemicals such as eugenol and caffeine with chromatogram profile that is shown ion Fig. 3, and while their mass profile spectra that is shown in Fig. 4. AB-Fubinaca is an NPS from synthetic cannabinoids derivatives laced on plant material that has been encountered by law enforcement in recent years. These products laced with synthetic cannabinoids which are smoked for their psychoactive effects.<sup>18</sup> Based on reported data UNODC 2022 and National Narcotics Board, there were a signification increased of illicit drugs from and NPS from synthetic cannabinoids derivatives such as AB-Fubinaca. AB-Fubinaca was previously reported in a patent by Pfizer in 2009. There are no commercial or medical uses for this substance. AB-Fubinaca has a high affinity binding as agonist to the CB1 receptor.<sup>5,18</sup> AB-Fubinaca is a synthetic cannabinoid that is recently encountered on the designer drug market and has been found laced on plant material and marketed under the guise of herbal incense products.

Phenethylamines derivatives such as Methamphetamine (MA); Methylenedioxymethamphetamine (MDMA); Methylenedioxyethylamphetamine (MDA); and Methylenedioxyethylamphetamine (MDEA) are commonly detected for illicit drugs. Methamphetamine (MA) is a compound that dominates illicit drugs and often found in tablet like dosage forms candies, not only Methamphetamine (MA), but also

Methylenedioxymethamphetamine (MDMA) (ecstasy); Methylenedioxyethylamphetamine (MDA); and Methylenedioxyethylamphetamine (MDEA) that is shown in **Fig. 5 and Fig. 6**. MA; MDMA; MDA dan MDEA are narcotic schedule that have strong activity as analgesic nevertheless these have strong addiction side effect. Because of the strong addiction side effect, MA and MDMA were never used as medicine on clinical practices.

Ketamine and tramadol are medicines on clinical practices. Ketamine has strong analgesic and general anaesthesia with moderate addiction side effect. Ketamine is a class of Phencyclidine derivatives which are often used as illicit drugs. Tramadol is a medicine that has strong analgesic with mild addiction side effect. Tramadol is classified as semi-opioid medicine that is often used as illicit drugs. Tramadol is often detected in tablets or caplet dosage form and ketamine is often detected in white crystal powders dosage form that is shown in **Fig. 7**.

Mephedrone (4-MMC) was also found in the drug market. Mephedrone is a group of synthetic cathinone derivatives. Mass profile spectra of Mephedrone can be shown in **Fig. 8**. Mephedrone exhibits high abuse liability. It has earlier onset and shorter duration of effects probably related to its short elimination of halflife compared to MDMA, and this, could explain a more compulsive pattern of use as described by the user's illicit drugs users.<sup>19</sup>

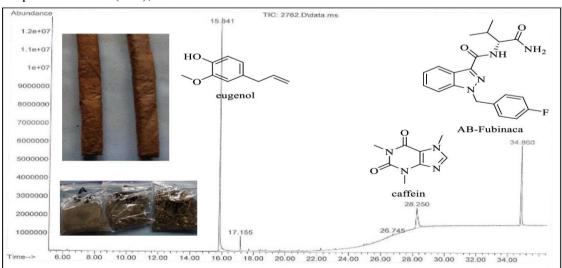


Fig. 3: Chromatogram profile of cigarette or tobacco or simplicia samples.

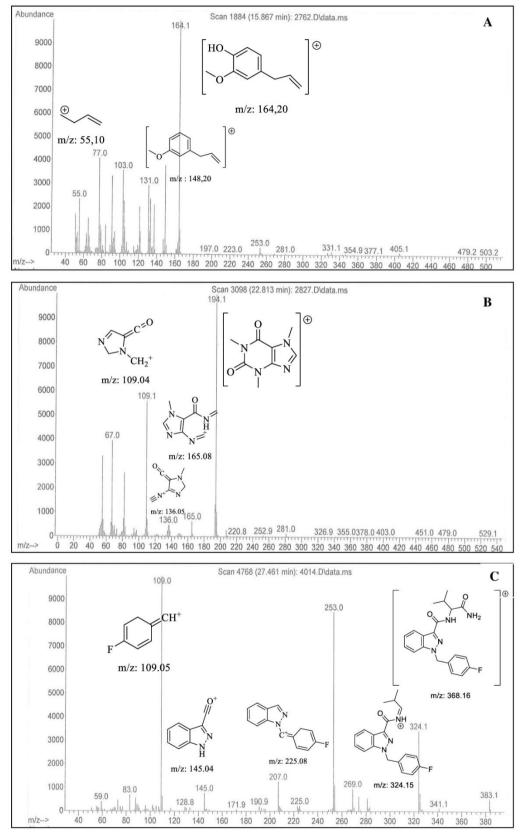
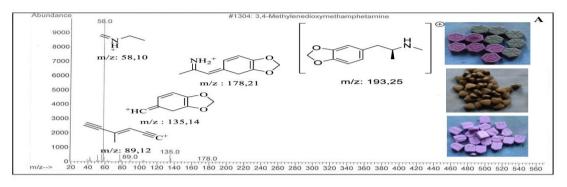


Fig. 4: Mass profile spectra of A.eugenol, B.caffeine . C.AB-Fubinaca.



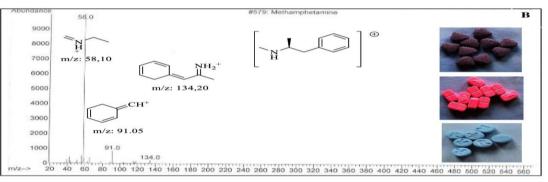
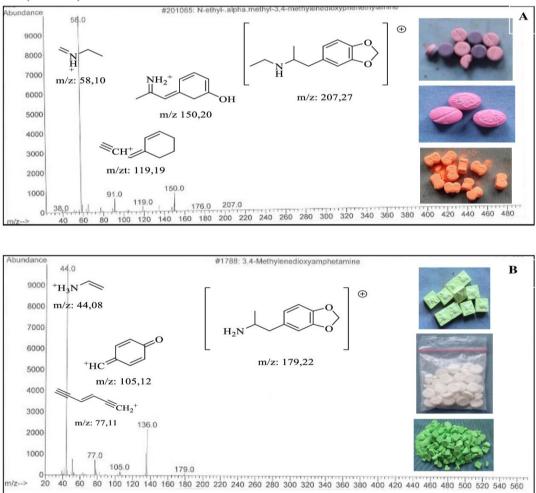


Fig.5: Mass profile spectra of A. Methamphetamine (MA) and B.Methylendioxymethamphetamine (MDMA).



**Fig. 6 :** Mass profile spectra of **A.** Methylenedioxyethylamphetamine (MDEA) and **B.** Methylenedioxyethylamphetamine (MDA).

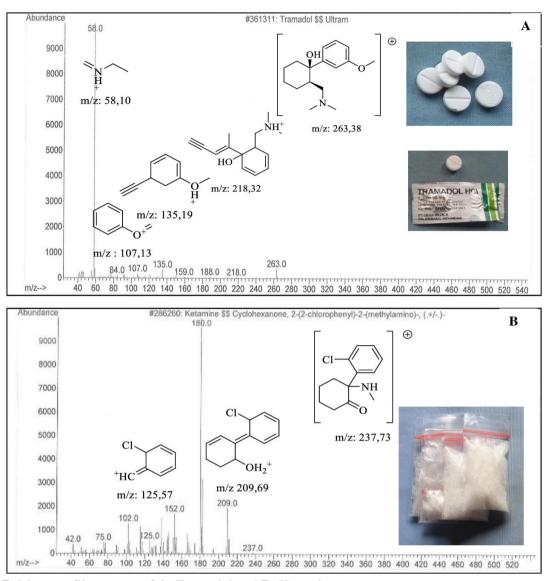


Fig. 7: Mass profile spectra of A. Tramadol and B. Ketamin.

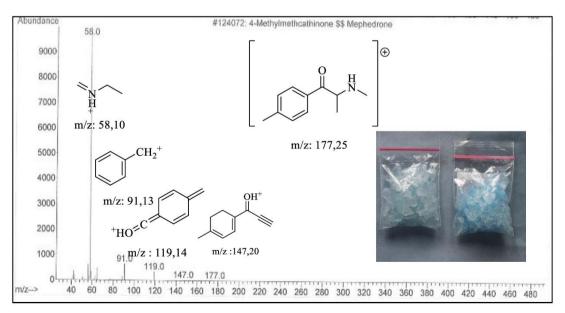


Fig. 8: Mass profile spectra of Mephedrone.

#### Conclusion

GC-MS is a method that can detect the presence of NPS with high specificity and selectivity. From 500 samples, it was detected 244 (48.8%) Methamphetamine (MA); 30 (6%) 3,4-Methylenedioxymethamphetamine

(MDMA); 44 (8.8%) 3,4-Methylenedioxyethylamphetamine (MDA) ; 56 (11.2%) 3,4-Methylenedioxyethylamphetamine (MDEA); 106 (21.2%) AB-fubinaca; 5 (1.0%) Tramadol; 5 (1.0%) ketamine; and 10 (2.0%)mephedrone

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تحليل الأدوية التي اسئ استخدامها مع مواد نفسية نشطة جديدة باستخدام التحليل الغازي الكروماتوجرافي مع طيف الكتلة

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يهدف هذا البحث إلى تحليل ٥٠٠ عينة للمخدرات غير المشروعة و المواد ذات التأثير النفسى الجديدة باستخدام التحليل الغازي الكروماتوجرافي مع طيف الكتلة في جاوة الشرقية بإندونيسيا. تم إجراء تحليل العينة بواسطة GC-MS مع عمود 5-HP، ودرجة حرارة منفذ الحقن ٢٥٠ درجة مئوية. تم الحفاظ على درجة حرارة العمود عند ١٠٠ درجة مئوية لمدة دقيقتين. ثم تم رفعها من ١٠ درجة مئوية / دقيقة إلى ٢٨٠ درجة مئوية والحفاظ عليها لمدة ٥ دقائق. وبناء على النتائج، تم الكشف عـن مئوية / دقيقة إلى ٢٨٠ درجة مئوية والحفاظ عليها لمدة ٥ دقائق. وبناء على النتائج، تم الكشف عـن إجراء تحدين / ٤٨٨٪) من الميثامفيتامين (MA)؛ ٣٠ (٦%) ٢٠٤–ميثيلين ديوكسي ميثامفيتامين (MDMA)؛ ٤٤ (٨,٨%) ٢٠٤–ميثيلين ديوكسي إيثيلامفيتامين (MDA)؛ ٣٥ (٢.١١,٢) ٢٥٤–ميثيلـ ين ديوكسي إيثيلامفيتامين (MDA)؛ ١٠٦ (٦%) أب–فوبيناكا؛ ٥ (١٠,١٠%) ٢٥١–ميثيلـ ين ديوكسي و ١٠ (٢,٠٢%) ميفيدرون. تم بنجاح التعرف على المخدرات غيـر المشـروعة و RNS في المـواد المصادرة عبر GC-MS. يعد توفر مكتبات GC-MS ممثابة مساعدة كبيرة في تحديد الأدوية الجديـدة. و دا (٢,٠٢%) ميفيدرون. تم بنجاح التعرف على المخدرات غيـر المشـروعة و RN في المـواد و دو المصادرة عبر GC-MS. يعد توفر مكتبات GC-MS ممثابة مساعدة كبيرة في تحديد الأدوية الجديـدة. وبدلاً من ذلك، يمكن أن تكون دراسة أجزاء الجزيء المميزة جنبًا إلى جنب مع تحديد كتلتهـا الدقيقـة طريقة مفيدة لتحديد العينات غير المعروفة التى لم يتم تحليلها مسبقا.





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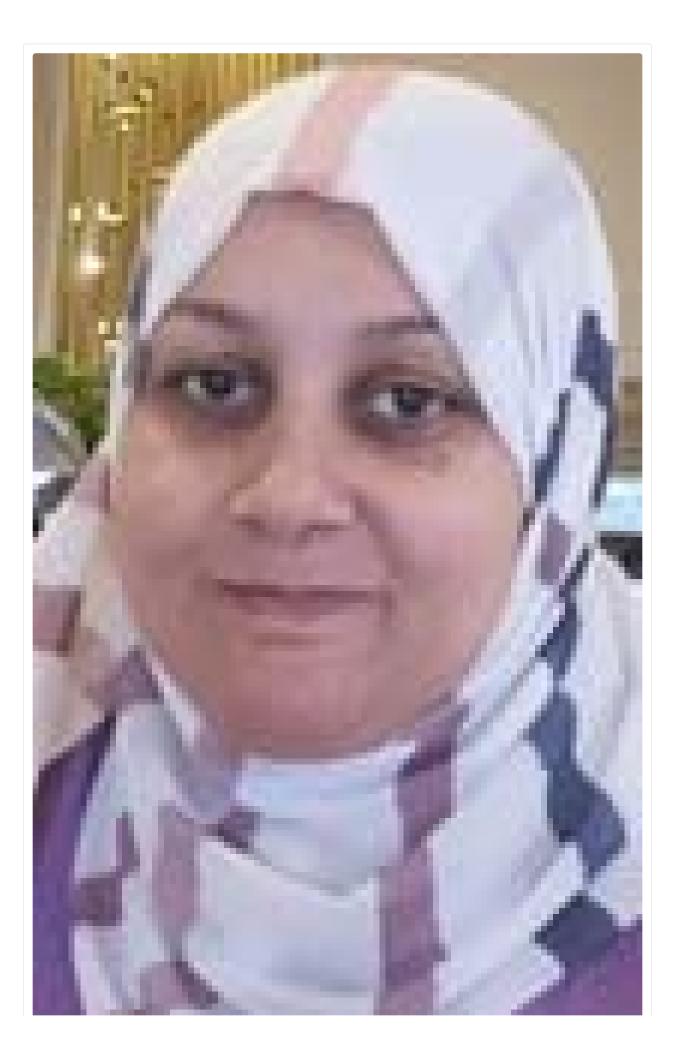


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Mumber of Articles: 45

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#### MACROLACTONES AND MACROLIDES FROM PLANT ENDOPHYTIC FUNGI, CHEMICAL SCAFFOLDS, BIOLOGICAL ACTIVITIES AND SPECTROSCOPY: A COMPREHENSIVE REVIEW

Pages 151-168 ehab Saad Elkhayat; Mohamed E Abouelela; Reda Ahmed Abdelhamid; Mohammad S Alorainy; Khaled A Shaaban

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#### DETERMINATION OF THE FLAVONOIDS OF BROCCHIA CINEREA FROM ALGERIA

Pages 169-177 Mohammed Tahar Ben Moussa; Said Nadji; Abdelhakim Bounab; Youcef Hadef View Article 🔀 PDF 604.56 K 😵 View on SCiNiTO

#### SCREENING OF YEAST ABILITY TO DECOLORIZATION AND COMPLETE BIODEGRADATION

#### OF MALACHITE GREEN TEXTILE DYE AND INVESTIGATION OF THEIR PHYTOTOXICITY

Pages 179-195

Nivien Allam; Eman Mostafa Mohamed; Somaya Mahmoud Nassar; Maysa Ahmed Ali

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## INVESTIGATING THE PROTECTIVE ACTIVITY OF POLYGONUM AVICULARE EXTRACTS AGAINST THE METHOTREXATE-INDUCED HEPATIC INJURY IN EXPERIMENTAL ANIMALS

Pages 197-206

Wiam Abdel-Nasser Hmidan; Adawia Kitaz; Samer Haj Kaddour; Mohammad Yaser Abajy

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### COMPARATIVE PROTEIN KINASE INHIBITORY ACTIVITY OF THREE ACACIA SPECIES GROWING IN NIGERIA

Pages 207-215

AUGUSTINE AJIBOGUN AHMADU; Bilqis Abiola Lawal; Aniefiok Sunday Udobre; Florence Tarfa; Adesegun Jubril Kashimawo; Patricia Odumosu

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Pages 233-248

Eman A El-Wakil; Heba Abdel-Hady; Eman A Morsi

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# QUALITATIVE ANALYSIS FOR ILLICIT DRUGS AND NPS (NEW PSYCHOACTIVE SUBSTANCES) USING GC-MS

Pages 249-259

Galih Satrio Putra; Sumari Sumari; Melanny Ika Sulistyowaty; Magdalena Sri Handajani; Farida Suhud; Dini Kesuma; Farida Anwari View Article 🛛 PDF 1.78 M 🛛 🚷 View on SCiNiTO

### FABRICATION OF ION SELECTIVE PVC MEMBRANE SENSOR FOR POTENTIOMETRIC DETERMINATION OF ESCITALOPRAM OXALATE IN ITS PURE FORM AND PHARMACEUTICAL TABLET

Pages 261-272 Ali Mohammed Atiyah; Kameran Shukur Hussein; Abdul Majeed Khorsheed Ahmed View Article 🖻 PDF 1.44 M 😪 View on SCiNiTO

# NEO-ADJUVANT VERSUS ADJUVANT USE OF BEVACIZUMAB IN THE MANAGEMENT OF ADVANCED OVARIAN CANCER

Pages 273-287

Nada Hassan Salah; hisham ahmed aboutaleb; mohammed alaa el din hassan; Mohammed abdelHakim Mekkawy; ola nabih abdel fattah

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### A COMPARATIVE STUDY OF THE EFFICACY AND SAFETY OF NEBULIZED VERSUS INTRAVENOUS MAGNESIUM SULFATE IN ADULTS WITH ACUTE ASTHMA EXACERBATIONS: A RANDOMIZED CONTROLLED STUDY

Pages 289-301

Maged Naguib; Nada A Saad; Mohammad Farouk Mohammad; Rasha M.Kharshoum; Raghda Hussein

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### COMPARING THE IMPACT OF LIDOCAINE SPRAY AND ICE SPRAY ON PAIN INTENSITY DURING THE CATHETERIZATION OF HEMODIALYSIS PATIENTS: A TRIPLE-BLIND CLINICAL TRIAL

Pages 303-310 Samira Foji; Hamid Roodsarabi; Fatemeh Torklalebag

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### ASSOCIATION OF SERUM VITAMIN D-BINDING PROTEIN WITH COMPONENTS OF METABOLIC SYNDROME IN TYPE 2 DIABETIC PATIENTS IN GORGAN

Pages 311-319

Abdoljalal Marjani; Safa Jalal Abdalsahib Alhasoon; Karrar Jaber Hasan Al-hajmee; Mojtaba Zare Ebrahimabad; Taghi Amiriani

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Pages 321-333

Debasmita Chatterjee; Krishnendu Paira; Satadal Das

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# INVESTIGATING THE LUNG CYTOTOXICITY OF KINETIN PROLONGED TREATMENT; IN VITRO AND IN VIVO STUDY

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#### ANTI-OBESITY ACTION OF GREEN TEA EXTRACT AND CURCUMIN: ROLE OF C1Q/TNF-RELATED PROTEIN-12 (CTRP-12) AND CASPASE-2.

Pages 345-362

Mohamed Hussein; Manal Mandour; Sary AbdElghaffar Nasr; Abdel-Raheim Meki; Michel E. Fakhry

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#### IMPACT OF LETROZOLE TREATMENT ON LIPID PROFILE IN POSTMENOPAUSAL WOMEN WITH HORMONE RECEPTOR-POSITIVE EARLY BREAST CANCER: A PROSPECTIVE STUDY

Pages 363-374

Ali Ghassan Ali; Remal Abdulaziz Asaad; Nader Mohammad Abedallaa

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### NOVEL THERAPEUTIC REGIMENS FOR URETHANE-INDUCED LUNG CANCER IN RATS: II CISPLATIN NANOPARTICLES COMBINED WITH CURCUMIN NANOPARTICLS ADJUVANT

Pages 375-394

Nahed A. Mohamed; Soad M.A. Faied; Naglaa T El Melegy; Hosaam E. Omer; Wael Sabry; Sary Kh. Abdel Ghaffar; Abdel Rahman Abdel qawy; Hala M. Mohamed

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#### NOVEL POTENTIAL DRUG INTERACTIONS WITH BISOPROLOL IN HOSPITALIZED ACUTE CORONARY SYNDROME PATIENTS

Pages 395-413

Sherouk Okda; Amira B. Kassem; Ahmed Mahmoud Elamrawy; Ahmad Salahuddin; sohila M Elonsy; Noha El-Bassiouny

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## ALTERATIONS OF NEUROBEHAVIORAL PERFORMANCE, BLOOD AND BRAIN CHOLINESTERASE ACTIVITIES AND CHOLESTEROL LEVELS BY REPEATED STATIN TREATMENTS IN MICE

Pages 415-425

Rawnaq Faris Al-Shalchi; Fouad Kasim Mohammad

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#### PREVALENCE OF OCCULT HEPATITIS B VIRUS INFECTION AMONG CHRONIC HEMODIALYSIS ADULT PATIENTS IN MINIA GOVERNORATE, EGYPT

Pages 427-435

Mustafa Hemeda; Rehab Mahmoud; Hala Rady Ahmed; Helal F. Hetta; Amr elzawily; Nancy G F wally

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Pages 437-448 BASIM SALEEM AHMED; HUSNI MHAMMAD

View Article 🛛 PDF 648.81 K 🛛 😵 View on SCiNiTO

#### T CELL EXHAUSTION: SUPREMACY IN LEUKEMIA AS FOR DISEASE AND THERAPY

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### THE ANTIMICROBIAL AND ANTIBIOFILM EFFECTS OF CITRUS LIMON AND CINNAMON CASSIA ESSENTIAL OILS AGAINST BIOFILM PRODUCING STAPHYLOCOCCI CAUSING

#### CHRONIC TONSILLITIS.

Pages 463-481

Elana Rafat Zaky; Nahla Mohammed Elsherbiny; Soad A. L. Bayoumi; Mohamed M Osman; amany adawy nafeh; Mohammed Saad Badary

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### EFFECT OF CHITOSAN OR N-ACETYL CYSTEINE COMBINATIONS WITH SOME ANTIBIOTICS ON BIOFILM FORMATION ON INTRAUTERINE DEVICES

Pages 483-497

Heba Ahmed Mohamed; Gamal Fadl Gad; Gehad Mostafa Elheiny; Ahmed Reda Eladway

View Article 🛛 PDF 1.52 M 🛛 🚷 View on SCiNiTO

# BIOSYNTHESIS OF ZINC OXIDE NANOPARTICLES USING ALOE VERA LEAVES EXTRACT AND THEIR ANTIBACTERIAL IMPACT

Pages 499-517 Magdy Abu-Gharbia; Jehan Salem; Gehad Al-Arabi

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## CIRCULATING MIRNA-122, MIRNA-155, AND MIRNA- 200 AS PREDICTORS FOR HEPATOCELLULAR CARCINOMA OCCURRENCE IN EGYPTIAN PATIENTS WITH HEPATITIS C RELATED LIVER CIRRHOSIS TREATED WITH DIRECT ACTING ANTIVIRAL DRUGS

Pages 519-532

Hanan M Nafeh; Alshaimaa Mohammed Rafat; Sahar Hassany; Abdelmajeed M. Moussa; Helal F. Hetta

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### IS THE ANTIMICROBIAL RESISTANCE OF ESCHERICHIA COLI. PATHOGEN IN CHILDREN WITH DIARRHEAL INFECTION STILL A CONCERN IN INDONESIA? : AN UPDATED REVIEW

Pages 533-553

Omnia Amir Osman Abdelrazig; Fadilah Fadilah; Badriul Hegar; Yulia Rosa Saharman

View Article 🕒 PDF 984.94 K 🛛 🚷 View on SCiNiTO

## EVALUATION OF BIOFIRE FILMARRAY PNEUMONIA PANEL IN DIAGNOSIS OF PNEUMONIA IN PEDIATRIC PATIENTS IN INTENSIVE CARE UNIT IN COMPARISON TO VITEK 2 COMPACT SYSTEM AND ROUTINE CULTURE METHODS

Pages 555-569

Amal Mohamed Hosni; Alaa Kadry Moawad; Hanan Hares AbdElateef; Mohamed Zakaria Abd El Rahman; Azaa Ahmed El-Tayeb; Noha Omer Sayed Khalil

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Pages 571-584

Ali A Ahmed; Omnia Hasan Bakr Badawy; Mohamed El Mokhtar; Omer A Ahmed; Marwa Sabet; A M Zahran; Salwa Seif Eldin

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# SNAPSHOT ON A POSSIBLE THERAPEUTIC EFFECT OF ANTI-DIABETIC DRUGS IN GASTRIC ULCER

Pages 585-595

Samah Omar Hassan; Asmaa H Matouk; Aliaa Anter; Gehan Heeba

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### EMPAGLIFLOZIN MITIGATES ISCHEMIA/REPERFUSION-INDUCED LIVER INJURY IN RATS: MODULATION OF NF-KB, SMAD-4, VEGF, AND FIBRINOGEN PROTEIN EXPRESSIONS

Pages 597-612

DeiaaEldeen Elsayed Abouzed; Heba Mohsen Mahmoud; Amira Murad; Ramadan Hemada Hemada

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## THERAPEUTIC EFFICIENCY OF RANIBIZUMAB AND 4-METHYL CATECHOL ON THE PROGRESSION OF STREPTOZOTOCIN-INDUCED DIABETIC RETINOPATHY IN RATS: INVOLVING VASCULAR ENDOTHELIAL GROWTH FACTOR-A AND NERVE GROWTH FACTOR

Pages 613-627

Eman Elsayed; Amira El-Saadany; Walaa Elwan; Amany Abdin; Fleur Abd Elmonem

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Pages 655-667

Shimaa M. M Elkhyat; Nageh Ahmed Elmahdy; Mayada E Elhusseiny; Abdel aziz Awad Zidan

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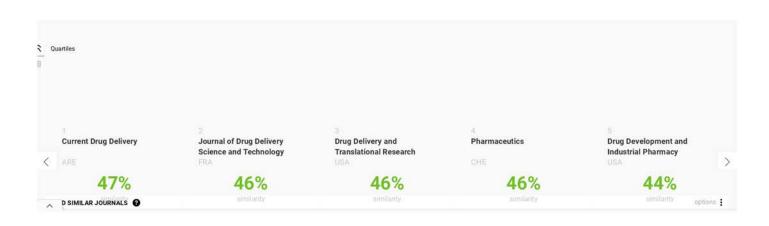
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