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Review Article

A Comprehensive Review on Developed Pharmaceutical Analysis Methods by Iranian Analysts in 2018

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Abstract

This article summarizes the publishing activities including bioanalytical and pharmaceutical analyses researches carried out in Iran in 2018 in order to connect academic researchers to those in industry, medical care units and hospitals. A wide spectrum of analytical methods has been used to determine and/or evaluate drug levels in the biological samples, based on physical, chemical and biochemical principles. We have compiled a concise survey of the literature covering 125 reports and tabulated the relevant analytical parameters. Chromatographic and electrochemical methods were found to be the technique of choice for many workers and almost 83% studies were performed by using these methods. This is the first annual review of the literature searching in SCOPUS database for published bioanalytical and pharmaceutical analysis researches in Iran.

Introduction

Iran (also known as Persia in the West) has a very long history of civilization and the Iranian scientists played a significant role in the scientific findings in the past. Geographically, Iran is located in the Silk Road connecting Asia to Europe and had scientific interactions with the ancient civilizations of Greece, India, and China beside the economic and cultural interactions. Great scientists have emerged from Iran who contributed substantially in expanding medicine, chemistry, physics, mathematics, astronomy, philosophy etc. They played an essential role to keep alive the scientific approaches in the Dark Ages and handing over these approaches to the pioneers of Renaissance.

The history of higher education in Iran goes back to centuries before the birth of Jesus, as examples schools of Nisibis, Sarouyeh, Reishahr. The first educational complex in the form of university was Academy of Gundeshapur or Jundishapur in Ahvaz in the 3rd century CE and was attracted scientists and scholars from Rome, China, and India. Various books were written in Pahlavi script in Jundishapur. After spread of Islam to Iran in the mid 7th century CE, the Islamic world entered to the Golden age of learning. The Abbasi caliphate invited the scientists (from different origins including Iranian, Jeweish, Syrian, Greek etc.) to the new capital of Islamic empire, Baghdad. Many Pahlavi books were translated into Arabic. A number of Iranian scientists are well-known in the West includes; Razes (865-925 CE), Majusi which was called as Haly Abbas in the Western literature (930-994 CE) and Avicenna (980-1037 CE). The Rab'-e-Rashidi (Rabi Rashidi) complex in Tabriz (Tauris) was the next comprehensive university established ~ 700 years ago which consist of various faculties, libraries, production units for medicine and also farms for herbals production. The updated and Western style higher education institution was found by Amir Kabir in the form of Dar ul-Funun in Tehran on 1851 which is continued the academic jobs as the University of Tehran later on.[1-4]

The first paper with Iran affiliation under subject of chemistry indexed in Scopus data base goes to back to 1840 and was published by Zinin[5] in German language. Figure 1 depicts the number of publications per year indexed in Web of science since 1983 and searched using "AFFILCOUNTRY (Iran)". Then the reported publications are limited to the subject of "Chemistry" and the sub-category of "Analytical chemistry" and "Pharmaceutical analysis". Concerning the number of publications, there is an obvious increase in the all research activities in Iran after year 2000 and continuously increasing patterns in the number of articles was observed for articles published by Iranian researchers in the field of chemistry and analytical chemistry. Until now, the maximum numbers of articles

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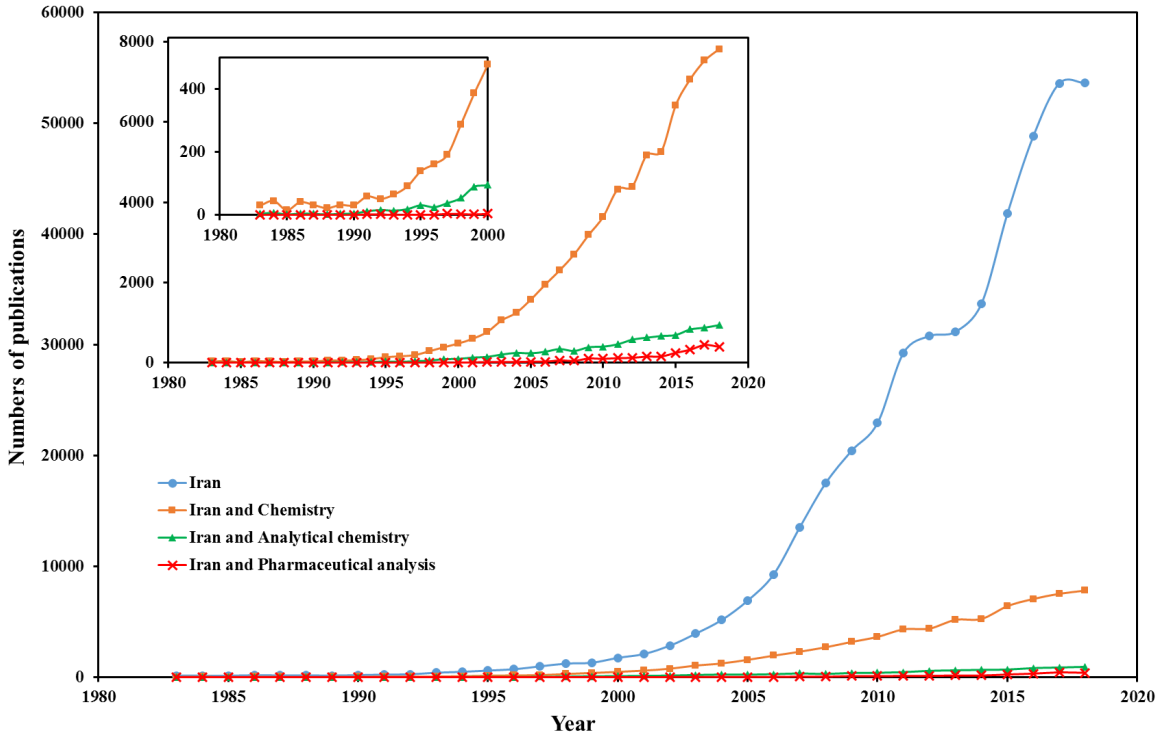

Figure 1. The number of publications of Iranian researchers indexed in Web of science since 1983.

published per year (i.e. in 2018) was 53624 which 7819 of them are in chemistry subject and 939 of chemistry articles is related to analytical chemistry. From citation viewpoint, the highest number of citations for the articles until now is related to Assadi et al.[6] published in Journal of Chromatography A with 2339 citations.

Pharmaceutical research encompasses a wide array of researches, extending from "basic research" to "clinical research" conducted to expand knowledge in the field of drug manufacturing, drugs and pharmaceutical analysis including therapeutic drug monitoring (TDM) and or medication concentrations control in the biological fluids. Analytical chemistry as an inseparable part of pharmaceutical analysis employs various instruments and methods to identify, and quantify pharmaceutical compounds in different stages of drug development and clinical application.[7] Bioanalytical methods as subset of analytical methods can be used for the quantification of drugs, their metabolites and also biomarkers in various biological matrices.[8] The applicability of bioanalytical methods in the pharmaceutical research for analysis of the biological samples became the mainstay of pharmaceutical analysis development. The main aim in the pharmaceutical analysis is selecting the appropriate methodology to drug monitoring, and developing valid analytical schemes that are performed with suitable quality.[9] So far, various analytical methods including spectrophotometric, fluorometric methods, high-pressure liquid chromatography (HPLC) in combination with different detection methods, such as UV detection, fluorescence detection (FD), or mass spectrometry (MS), gas chromatography (GC) in combination with flame ionization detector (FID) and MS and capillary electrophoresis were developed for analysis of pharmaceutical compounds in the biological samples. As many different types of analytical methods are reported for pharmaceutical analysis, the selection of an appropriate assay for a given drug will be easy if the respective merits of each method are documented.

To the best of our knowledge, this is the first annual review to summarize Iran's published pharmaceutical analysis studies which covers the bioanalytical researches conducted in Iran for determination of pharmaceuticals in various biological samples in 2018. In order to find the reported studies, SCOPUS database search are limited to year of 2018 with the keyterms of: "drug determination, pharmaceutical determination, drug analysis, pharmaceutical analysis, drugs level in biological samples and pharmaceuticals level in biological samples". A total of 125 studies were identified for the given keywords. Since this review is limited to the determination methods of drug/pharmaceutical compounds in the biological samples, other sample matrices such as food and environmental samples are excluded and the literature search was restricted to the biological samples only.

Reported Analytical Methods for Determination of Drug/pharmaceutical Compounds in the Biological Samples

According to methodological aspects, the selected literature on the determination of pharmaceutical compounds in the biological samples were classified into several categories.

Optical methods

Spectrophotometry

Spectrophotometric methods are the commonly used methods for analysis due to their fast, available and

simple instrumentation. The reported spectrophotometric studies in Iran in 2018 are summarized here and the analytical properties of each method are given in Table 1. Barkat Rezaei et al.[10] developed a poly (vinyl alcohol)/ chitosan (PVA/CS) nanofiber decorated with Ag ions as a smart spectrophotometric probe for determination of azathioprine in spiked serum samples. In the absence of azathioprine, Ag ions reduce by ascorbic acid and result in the formation of decorated silver nanoparticles (AgNPs) on to the surface of nanofibers. The presence of azathioprine during reduction process leads to a decrease in the formation of AgNPs on to the surface, consequently decreasing plasmon resonance intensity of nanoparticles. Hashemi et al.[11] reported a combination of dispersive liquid-liquid microextraction (DLLME) method and surface plasmon of silver nanoparticles for determination of captopril in spiked serum and urine samples. The presence of captopril affected the AgNPs synthesis and results in enhancement in plasmon absorbance intensity of nanoparticle. Shahrouei et al.[12] also employed a gold nanoparticle's aggregation for direct determination of ceftriaxone in spiked urine and serum samples. The suggested mechanism for aggregation of nanoparticles in the presence of ceftriaxone molecules are given in Figure 2. Farahmand et al.[13] developed a spectrophotometric method for determination of atenolol, propanolol and carvedilol in plasma and urine samples after extraction with an air assisted liquid–liquid microextraction (AA-LLME) by applying the solidification of a floating organic droplet method for simultaneous extraction of drugs. Hamid et al.[14] used a surfactant ion pair-switchable solvent dispersive liquid–liquid microextraction (SIP-SS-DLLME) for extraction/pre-concentration of phenazopyridine prior to spectrophotometry measurement in spiked urine and plasma samples. Protonated triethylamine bicarbonate and Aliquat 336 are used as a protonated switchable solvent and ion-pair agent in this study, respectively.

Nezhadali et al.[15] used a solid phase extraction (SPE) method based on molecularly imprinted polymers (MIPs) prior to spectrophotometry determination for fluoxetine. The MIPs were synthesized using pyrrole as a functional monomer and fluoxetine as a template molecule. Mehrabifar et al.[16] described a spectrophotometric method for the determination of penicillamine based on analyte derivatization with 6-dichlorophenolindophenol as the chromogenic agent. They used Fe_3O_4 nanoparticles for pre-concentration of penicillamine from spiked serum samples. Kamari et al.[17] used a Fe_3O_4@SiO_2-MIP for magnetic SPE of amitriptyline in spiked plasma and urine samples, Sadat Alaei[18] used a photoresponsive molecularly imprinted polymers conjugated hyperbranched polymers based on functionalized magnetic nanoparticles (PMIP@HBPM) for determination of azathioprine in spiked urine and plasma prior to analyte determination and Amraei et al.[19] reported a direct spectrophotometric determination of cefixime in spiked urine and plasma using parallel factor analysis (PARAFAC) and partial least squares (PLS). As mentioned in all reports, the spiked biological samples were used to demonstrate the application of the methods on real samples. Due to relatively poor selectivity of spectrophotometric methods and presence of drug's metabolites with very similar spectroscopic properties, application of the developed methods on real samples and also reporting the selectivity and also interference results are highly recommended.

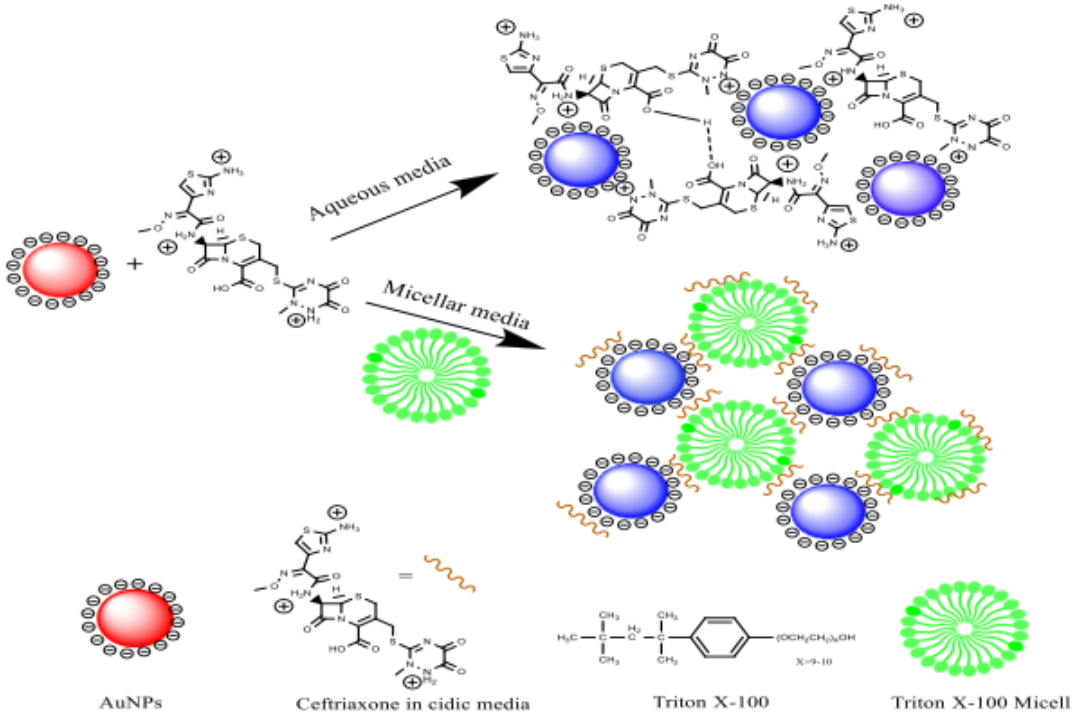

Figure 2. Suggested mechanism for the nanoparticles aggregation in the presence of ceftriaxone molecules in aqueous and micellar media. Adapted with permission from Shahrouei et al.[12] Copyright (2018) Elsevier.

Spectrofluorometery

Fluorimetric methods are usually more selective and sensitive than absorbance methods. This is due to this fact that fluorescence emission is detected against a low background isolated from the excitation source. Soleimani et al.[20] reported a validated spectrofluorometery method for determination of rizatriptan in urine samples after its extraction with the aid of a Fe_3O_4@SiO_2-MIP. In their work, adsorption equilibrium behaviors were studied and the results showed that rizatriptan adsorption on the nanosorbent is monolayer and is compatible with Langmuir assumptions. Some similar works were performed for determination of ofloxacin in plasma and urine samples after extraction by Fe_3O_4 @$nSiO_2$ @$mSiO_2$ –NH_2 nanosorbent.[21] Other similar works were performed for determination of furosemide in serum samples after extraction by a DLLME with methanol and chloroform solvent mixture[22] and for determination of ofloxacin in spiked plasma samples after extraction by using $CoFe_2O_4$ nanoparticles grafted multi-walled carbon nanotubes (MWCNTs).[23]

Eskandari et al.[24] used a fluorometric probe based on the MIP capped terbium metal-organic frameworks (MIP@TbMOF-76) for the measurement of cefixime in urine samples. The synthesized composite had an intense fluorescence emission related to the terbium emission which undergo a decrease in the presence of cefixime. The cefixime extraction procedure was performed by Fe_3O_4 /GO before analysis. Amjadi et al.[25] synthesized a dual-emission molecularly imprinted mesoporous silica embedded with carbon dots and CdTe quantum dots (mMIP@CDs/CdTe QDs) and used it for determination of celecoxib in spiked serum samples. Detection mechanism is quenching of green emissive QDs in the presence of celecoxib. Other similar works were performed for determination of atropine by using a MIP capped GQDs in spiked plasma samples,[26] and for determination of tetracycline using fluorescent nano-sensor based on oxidized starch polysaccharide biopolymer-capped CdTe/ZnS quantum dots.[27]

Rahbar et al.[28] synthesized a graphitic carbon nitride nanosheet (g-C_3N_4) as nano fluoroprobe and used for determination of metformin in spiked serum samples. Nanosheets were synthesized by direct pyrolysis of melamine at 600 °C. Cu (II) is used as a quencher for g-C_3N_4 nanosheets; addition of metformin to the solutions leads to fluorescence restoring proportional to its concentration which indicating that metformin molecule as a strong ligand participates in complex formation reaction with Cu (II) ions. A similar work was performed by Amin et al.[29] for determination of zoledronic acid in serum samples in the presence of a nitrogen doped carbon dots (N-CDs) - Fe^{3+} as a label free fluorescence platform. An illustration of fluorescence sensing strategy for zoledronic acid detection are shown in Figure 3. Najafi et al.[30] used a thioglycolic acid (TGA)-capped Au: CdTe quantum dots (QDs) for determination of gemcitabine in plasma and urine samples. The response of probe is result of quenching in fluorescence of nanoprobe due to the formation of a TGA-capped Au: CdTe QDs– gemcitabine comple and Bahrami et al.[31] use a combined unfolded principal component analysis and artificial neural network (UPCA-AN) for determination of ibuprofen in serum by three-dimensional excitation–emission matrix fluorescence spectroscopy. Analytical performance of each reported study are summarized at Table 1.

Resonance light scattering method

Resonance light scattering (RLS) is an optical method based on elastic light-scattering and occurs when an incident beam in energy is close to an absorption band.[32] Maleki et al.[33] synthesized a polyacrylonitrile nano fibers decorated with magnetic carbon dots (MCDs@NFs) nanocomposite for determination of famotidine in spiked serum samples. Possible mechanism for RLS enhancement is this fact that the RLS properties is related to morphology of the particles and the molecular volume. So, the higher RLS intensity observed for nanocomposite in the presence of famotidine is due to the bigger molecular volume after assembling of famotidine on the nanoparticle's surface.

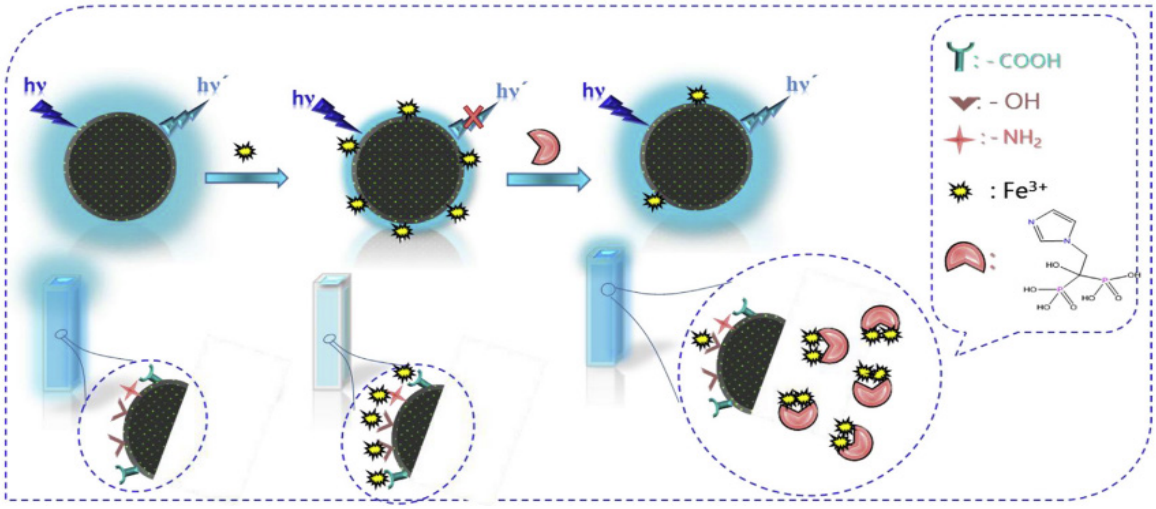

Figure 3. A schematic mechanism for zoledronic acid detection with N-CDs/Fe^{3+} system. Adapted with permission from Amin et al.[29] Copyright (2018) Elsevier.

Table 1. Analytical details of optical methods used for pharmaceutical analysis in biological samples conducted in Iran (year 2018).

Method	Analyte	Sample	Linear range	Detection limit	Remarks	Ref.
Spectrophotometry	Azathioprine	Serum	0.14 – 2.88 μmol L^{-1}	0.09 μmol L^{-1}	PVA/CS nanofiber decorated with Ag-NPs as a smart probe	10
Spectrophotometry	Captopril	Serum Urine	0.77 – 22.5 nmol L^{-1}	0.51 nmol L^{-1}	-	11
Spectrophotometry	Ceftriaxone	Urine Serum	5 – 300 ng mL^{-1}	3 ng mL^{-1}	Gold nanoparticles as a nanoprobe	12
Spectrophotometry	Atenolol Propanolol Carvedilol	Urine Plasma	0.3 – 6 μg mL^{-1} 0.3 – 1.4 μg mL^{-1} 0.3 – 2 μg mL^{-1}	0.09 μg mL^{-1} 0.08 μg mL^{-1} 0.1 μg mL^{-1}	Using floating organic droplet solidification for simultaneous extraction of drugs	13
Spectrophotometry	Phenazopyridine	Urine Plasma	5 – 180 μg L^{-1}	0.88 μg L^{-1}	Using a SIP-SS-DLLME method for analyte extraction	14
Spectrophotometry	Fluoxetine	Serum Plasma	10^{-7} –10^{-8} mol L^{-1}	6.56×10^{-8} mol L^{-1}	Using a MIP based on pyrrole for SPE of fluoxetine	15
Spectrophotometry	Penicillamine	Serum	50 – 3000 μg L^{-1}	-	Using Fe_3O_4 NPs for analyte pre-extraction	16
Spectrophotometry	Amitriptyline	Plasma Urine	0.05 – 250 mg L^{-1}	0.01 mg L^{-1}	Using a Fe_3O_4@SiO_2-MIP for analyte extraction	17
Spectrophotometry	Azathioprine	Urine Plasma	0.1 – 80 μg mL^{-1}	1.77 ng mL^{-1}	Using of PMIP@HBPM for analyte extraction	18
Spectrophotometry	Cefixime	Urine Plasma	0.5 – 9.0 μg mL^{-1}		Using PARAFAC and PLS	19
Spectrofluorometery	Rizatriptan	Urine	2.5 – 200 ng mL^{-1}	1.1 ng mL^{-1}	Using a Fe_3O_4@SiO_2-MIP for analyte extraction	20
Spectrofluorometery	Ofloxacin	Urine Plasma	1 – 500 μg L^{-1}	0.21 μg L^{-1}	Using Fe_3O_4 @nSiO_2 @mSiO_2 –NH_2 for analyte extraction	21
Spectrofluorometery	Furosemide	Serum	0.3 – 20 μg mL^{-1}	0.12 μg mL^{-1}	Using DLLME with methanol and chloroform solvent mixture for analyte extraction	22
Spectrofluorometery	Ofloxacin	Plasma	100 – 750 ng mL^{-1}	23 ng mL^{-1}	Using $CoFe_2O_4$ /MWCNTs for analyte extraction	23
Spectrofluorometery	Cefixime	Urine	0.8 – 90 ng mL^{-1}	0.34 ng mL^{-1}	Using Fe_3O_4 /GO for SPE of cefixime and MIP@TbMOF-76 as a fluorometric probe	24
Spectrofluorometery	Celecoxib	Serum	0.08 – 0.90 μmol L^{-1}	57 nmol L^{-1}	Using mMIP@CDs/CdTe QDs as a nanoprobe	25
Spectrofluorometery	Atropine	Plasma	0.5 – 300 ng mL^{-1}	0.22 ng mL^{-1}	Using MIP capped GQDs as a nanoprobe	26
Spectrofluorometery	Tetracycline	Urine Serum	9.14 – 7230 nmol L^{-1}	2.74 nmol L^{-1}	Using oxidized starch polysaccharide biopolymer-capped CdTe/ZnS quantum dots as nanoprobe	27
Spectrofluorometery	Metformin	Serum	0.01 – 20 μmol L^{-1}	3 nmol L^{-1}	Using of g-C_3N_4/Cu(II) as a nano fluoroprobe	28
Spectrofluorometery	Zoledronic acid	Serum	0.1 – 10 μmol L^{-1}	0.04 μmol L^{-1}	Using a N-CDs - Fe^{3+} system as a fluorescence platform	29
Spectrofluorometery	Gemcitabine	Plasma Urine	0.3 – 100 μmol L^{-1}	0.1 μmol L^{-1}	TGA-capped Au: CdTe QDs as a nanoprobe	30
Spectrofluorometery	Ibuprofen	Serum	0.043 – 0.43 μmol L^{-1}	0.519 nmol L^{-1}	Using UPCA-AN method	31
RLS	Famotidine	Serum	0.15 – 50 μmol L^{-1}	0.04 μmol L^{-1}	Using MCDs@NFs nanocomposite as a sensor	33

Electrochemical methods

Electrochemical methods are regarded as more sensitive method in compared with optical methods. In electrochemical techniques, the potential, current, or charge in an electrochemical cell serves as the analytical signal. Almost all electrochemical studies performed in Iran in 2018 are summarized in Table 2. As can be seen from Table 2, different types of electrochemical techniques

Table 2. Analytical details of electrochemical methods used for pharmaceutical analysis in biological samples conducted in Iran (year 2018).

Method	Analyte	Sample	Linear range	Detection limit	Remarks	Ref.
DPV	Levodopa Carbidopa Methyldopa Benserazide Tolcapone Entacapone	Serum	10 – 320 μmol L^{-1} 0.5 – 600 μmol L^{-1} 2 – 380 μmol L^{-1} 1 – 36 μmol L^{-1} 0.1 – 178 μmol L^{-1} 2 – 85 μmol L^{-1}	-	Ag/AgCl as reference electrode, a Pt wire as auxiliary electrode and a gold electrode as a working electrode.	35
Voltammetry	Sulfasalazine	Serum	0.009 – 1.6 μmol L^{-1}	0.0017 μmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode, NiO NPS modified CPE as working electrode	36
Voltammetry	Fluconazole	Serum Urine	0.01 – 400 μmol L^{-1}	3.5 nmol L^{-1}	A yolk shell Fe_3O_4@PA-Ni@Pd/Chitosan nanocomposite -modified CILE as working electrode	37
Voltammetry	Trazosin	Serum, Urine	2 – 250 μmol L^{-1}	0.3 μmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and MIP/AuNPs/SPCE as working electrode	38
DPV and Square wave voltammetry (SWV)	Alprazolam Chlordiazepoxide Clonazepam Diazepam Oxazepam	Serum	0.05 – 0.42 μmol L^{-1} 0.28 – 0.65 μmol L^{-1} 0.075 – 0.4 μmol L^{-1} 0.02 – 1.0 μmol L^{-1} 0.063 – 1.39 μmol L^{-1}	-	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and poly (DA-CS)-AuNPs– modified GCE as working electrode	39
SWV	Raloxifene	Serum	0.03 – 520 μmol L^{-1}	7.0 nmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and CPE/NiO/SWCNTs/ 1B4MPTFB as working electrode	40
SWV Amperometry	Epinephrine	Serum	0.36 – 380 μmol L^{-1}	0.3 nmol L^{-1}	Ag/AgCl as a reference electrode a pt wire as auxiliary electrode and GQD-CS modified CPE electrode as working electrode	41
DPV	Nifedipine Dehydronifedipine	Urine	0.1 – 100 μmol L^{-1}	0.015 μmol L^{-1} 0.017 μmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and MWCNT modified GCE as working electrode	42
Voltammetry	Epirubicin	Serum	0.02 – 700 μmol L^{-1}	7.0 nmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and Fe_3O_4 -SWCNTs/ MOCTICl/ CPE as working electrode	43
DPV	Didanosine	Urine	0.02 – 50 μmol L^{-1}	8 nmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and PGE/PPy/rGO as working electrode	44
SWV	Midazolam	Urine	0.5 – 1000 nmol L^{-1}	0.177 nmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and MIP nanoparticles modified CPE as working electrode	45
SWV	Trimipramine	Urine Serum	0.1 – 25 nmol L^{-1}	0.045 nmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and CPE modified with MWCNTs and a nano-structured MIP as working electrode	46
SWV	Mephedrone	Plasma Urine	1 – 100 nmol L^{-1}	0.8 nmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and MIP/polytyramine/f-MWCNT@ AuNPs nanocomposite/GCE as working electrode	47
CV	Tramadol	Urine	3.5 nmol L^{-1} – 0.01 mol L^{-1}	2.04 nmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and GNS@Ag NPs/ MIPNPs/[BMP] Tf2N RTIL/CPE as working electrode	48
CV	Dopamine	Serum	0.8 – 800 μmol L^{-1}	0.62 μmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and MWCNT/$TOAI_3$ /CIL as working electrode	49
DPV	Methimazole	Serum	5.2 – 50 μmol L^{-1}	2 μmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and CuNPs-P-L-Arginine/MWCNTs/GC as working electrode	50
DPV	Meloxicam	Plasma	9.0 – 8500 nmol L^{-1}	1.008 nmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and AuNPs-ChCl-GO/CPE as working electrode	51

Table 2 Continued.

DPV	Celecoxib	Plasma	9.6 – 7400 nmol L^{-1}	6.58 nmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and AgNPs-ChCl-GO /CPE as working electrode	52
Differential pulse anodic stripping voltammetry (DPASV)	Acyclovir	Serum Urine	0.03 – 1.0 µmol L^{-1}	0.01 µmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and GCE modified CS-MWCNTs + TiO_2 NPs with as working electrode	53
Linear sweep voltammetry (LSV)	Isoxsuprine	Serum	0.04 – 5.0 µmol L^{-1} 0.2 – 5.0 µmol L^{-1}	0.012 µmol L^{-1} 0.06 µmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and GCE / MWCNTs / Ag NPs as working electrode	54
DPV	Theophylline	Urine	1.0 – 700 µmol L^{-1}	0.2 µmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and GQDs/SPE as working electrode	55
DPV	Methyldopa	Urine	0.5 – 800 µmol L^{-1}	0.23 µmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and Cu/TiO_2 /GCE as working electrode	56
DPV	Epinephrine Paracetamol Tryptophan	Serum Urine	7 – 560 µmol L^{-1} 5 – 580 µmol L^{-1} 4 – 560 µmol L^{-1}	0.45 µmol L^{-1} 0.27 µmol L^{-1} 0.38 µmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and Ni-LFONRCPE as working electrode	57
Voltammetry	Favoxatehydrochloride Tolterodine tartrate	Urine	7.8 – 1200 µmol L^{-1} 0.76 – 2200 µmol L^{-1}	0.086 µmol L^{-1} 0.29 µmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and FMCPE as working electrode	58
DPV	Meclizine	Urine	0.33 – 29.13 µmol L^{-1}	80 nmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and bare GSPE as working electrode	59
amperometry	L-Carnitine	Serum	24 – 312 µmol L^{-1}	7.08 µmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and anodized Ni/Cu alloy electrode as working electrode	60
DPV	Rituxan	Serum	7 – 300 µmol L^{-1}	0.56 µmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and PA-MAM/RGO/PGE as working electrode	61
DPV	Azithromycin	Serum	0.3 – 920 nmol L^{-1}	0.1 nmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and MIP/GNU/GO/GCE as working electrode	62
Fast Fourier transform SWV	Estradiol valerate	Blood	0.1 – 10000 ng mL^{-1}	0.01 ng mL^{-1}	CPE modified with $Tb_2(CO_3)_3$ nanoparticles as working electrode	63
DPV	Methimazole	Serum	0.007 – 6 mmol L^{-1}	3 µmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and MIP- PGE as working electrode	64
DPV	Diclofenac sodium	Urine	0.23 – 12.95 µmol L^{-1}	0.12 µmol L^{-1}	A saturated calomel electrode as a reference electrode, a Pt wire as auxiliary electrode and PPGE as working electrode	65
DPV	Repaglinide	Serum Urine	0.005 – 1 µmol L^{-1}	1.8 nmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and MIP-GCE as working electrode	66
LSV	Tizanidine	Serum	0.01 – 1.0 µmol L^{-1} 1.0 – 10 µmol L^{-1}	3 nmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and OPPY/CNT/GCE as working electrode	67
DPV	Propylthiouracil	Urine	0.05 – 5 µg mL^{-1}	0.02 µg mL^{-1}	Using a copper nanoparticles-decorated hollow fibers for analyte extraction	68
DPV	Nitrazepam	Urine	0.03 – 20 ng mL^{-1} 20 – 450 ng mL^{-1}	9 ng L^{-1} 0.03 ng mL^{-1}	Using a HLLME procedure for extraction of nitrazepam. Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and GCE as working electrode	69
Adsorptive stripping voltammetry	Warfarin Maycophenolic acid	Serum Urine	0.05 – 150 µmol L^{-1} 0.5 – 200 µmol L^{-1}	0.02 µmol L^{-1} 0.03 µmol L^{-1}	A saturated calomel electrode as a reference electrode, a Pt wire as auxiliary electrode and modified CPE by b-CD/MWCNTs/Co_3O_4 NPs/CPE as working electrode	70

Table 2 Continued.

Adsorptive stripping differential pulse voltammetry (ASDPV)	Metformin	Urine	0.1 – 80 µmol L^{-1}	14 nmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and CPE modified with γ-Fe_2O_3@HAp/Cu (II) as working electrode	71
ASDPV	Ceftizoxime	Plasma	0.001 – 1 nmol L^{-1}	0.00035 nmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and HGNPs/rGO/PGE as working electrode	72
DPASV	Docetaxel	Urine Serum	0.3 – 3.3 µmol L^{-1}	90 nmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and Au-MWCNTs/GCE as working electrode	73
DPV	Dopamine Acetaminophen Xanthine	Urine Serum	0.3 – 60 µmol L^{-1} 0.4 – 40 µmol L^{-1} 0.4 – 70 µmol L^{-1}	20 nmol L^{-1} 30 nmol L^{-1} 50 nmol L^{-1}	Ag/AgCl as a reference electrode a pt wire as auxiliary electrode and GCE/PDA-MWCNTs as working electrode	74
DPV	Acetaminophen Codeine	Urine Plasma	0.01 – 1.5 µmol L^{-1} 0.06 – 10 µmol L^{-1}	0.007 µmol L^{-1} 0.01 µmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and CuO-$CuFe_2O_4$ nanoparticles /CPE as working electrode	75
DPV	Acetaminophen Pramipexole Carbamazepine	Plasma	2.5 – 110 µmol L^{-1} 0.6 – 105 µmol L^{-1} 6.0 – 97 µmol L^{-1}	0.58 µmol L^{-1} 0.38 µmol L^{-1} 1.04 µmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and ZSM-5 nanozeolite and TiO_2 NPs modified CPE as working electrode	76
DPV	Dopamine	Serum	25 – 3000 pg mL^{-1}	2 pg mL^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and the modified Au electrode with spindle-shaped gold nanostructure as working electrode	77
SWV	Diclofenac Morphine Mefenamic acid	serum	0.04 – 1200 µmol L^{-1} 0.9 – 400 µmol L^{-1} 1.0 – 600 µmol L^{-1}	0.008 µmol L^{-1} 0.4 µmol L^{-1} 15 µmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and NiO-SWCNTs/DDPM/CPE as working electrode	78
DPV	Diclofenac	Urine	5 – 80 mg L^{-1}	1.1 mg L^{-1}	A saturated calomel electrode as a reference electrode, a Pt wire as auxiliary electrode and MIP-CPE as working electrode	79
SWV	Flutamide	Urine	0.05 – 200 nmol L^{-1}	14 pmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and CPE modified with CuO/GO/PANI as working electrode	80
DPV/CV	Flutamide	Plasma	0.1 – 110 µmol L^{-1}	0.029 µmol L^{-1}	Ag/AgCl as a reference electrode a Pt wire as auxiliary electrode and HF/ HBP-GO/PGE as working electrode	81

e.g. differential pulse voltammetry (DPV), adsorptive stripping voltammetry, square wave voltammetry (SWV), amperometry, adsorptive stripping differential pulse voltammetry (ASDPV), differential pulse anodic stripping voltammetry (DPASV), cyclic voltammetry (CV) and linear sweep voltammetry (LSV) were used for pharmaceutical analysis. The common characteristic of all voltammetric methods is the application of a potential to an electrode and the monitoring of the resulting current flowing through the electrochemical cell.[34] Among of these methods, SWV and DPV can offer better sensitivity with higher signal-to-noise ratios, and can be employed as a powerful techniques for determination of trace levels of compounds. So, mostly these methods were used for determination of pharmaceutical compounds in biological samples.

Mohammadi et al.[35] developed a DPV method by using an unmodified gold electrode for simultaneous determination of levodopa, carbidopa, methyldopa, benserazide, tolcapone and entacapone in the presence of dopamine as uncalibrated interference in spiked serum samples. Amani-Beni et al.[36] validated a voltammetric method for determination of sulfasalazine by using NiO nanoparticles modified carbon paste electrode (CPE) in spiked serum samples. Some other electrochemical works were developed and validated for determination of fluconazole in serum and urine samples by using a yolk shell Fe_3O_4@PA-Ni@Pd/Chitosan nanocomposite -modified carbon ionic liquid electrode (CILE),[37] for determination of terazosin by using MIP with disposable gold nanoparticles modified screen printed carbon electrode (MIP/AuNPs/SPCE),[38] for simultaneous determination

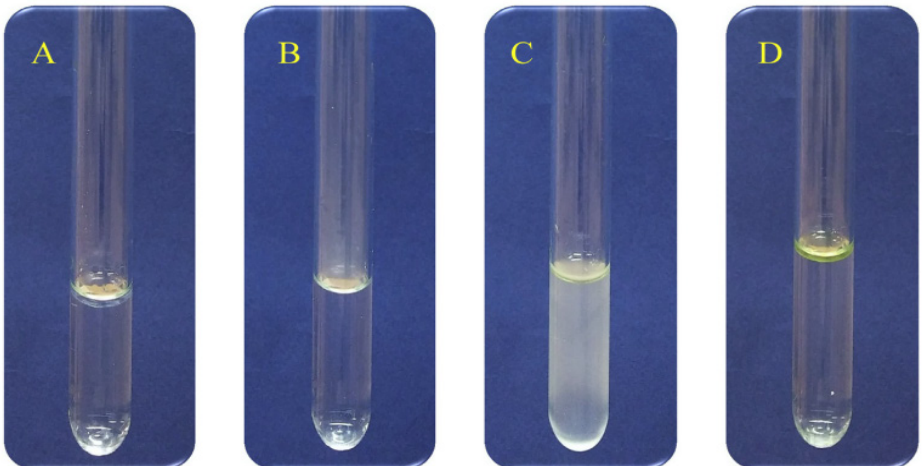

Figure 4. Illustration of the phase behavior of water and N,N−dipropylamine in HLLME of nitrazepam (a) primary separated water and N,N−dipropylamine phases (b) homogeneous solution of water and N,N−dipropylamine after the addition of HCl (c) cloudy mixture of water and N,N−dipropylamine after the addition of NaOH (d) phase separation. Adapted with permission from Shahraki et al.[69] Copyright (2018) Elsevier.

of alprazolam, chlordiazepoxide, clonazepam, diazepam, and oxazepam in spiked serum samples by using a poly(dopamine-chitosan) (DA-CS)-AuNPs organic-inorganic hybrid – modified glassy carbon electrode (GCE),[39] for determination of raloxifene in spiked serum samples by using CPE modified with NiO/SWCNTs and 1-butyl-4-methylpyridinium tetra-fluoroborate (1B4MPTFB),[40] for determination of epinephrine in serum by using graphene quantum dots - chitosan (GQD-CS) modified CPE electrode,[41] for determination of nifedipine and dehydronifedipine by using MWCNT modified glassy carbon electrode (GCE),[42] for determination of epirubicin in spiked serum samples by using both 3D Fe_3O_4 decorated single wall carbon nanotubes (Fe_3O_4 -SWCNTs) nanocomposite and 1-methyl-3-octylimidazlium chloride (MOCTICl) as amplifier into CPE,[43] for determination of didanosine in spiked serum samples by using pencil graphite electrode modified with polypyrrole and reduced graphene oxide (PGE/PPy/rGO),[44] for determination of midazolam in spiked urine samples by using MIP nanoparticles modified CPE,[45] for determination of trimipramine in spiked urine and serum samples by using CPE modified with MWCNTs and a nano-structured MIP,[46] for determination of mephedrone in spiked urine and plasma samples by using MIP/polytyramine/f-MWCNT@ AuNPs nanocomposite/GCE,[47] for determination of tramadol by using CPE modified with nanographene/ tramadol-MIP/ionic liquid (GNS@Ag NPs/ MIPNPs/ [BMP]Tf2N RTIL) with measured concentration between 45.2 – 51.0 µmol L^{-1} in urine sample of patients receiving tramadol,[48] for determination of dopamine in spiked serum samples by using a MWCNT/ionic liquid paste electrode / tetra-n-octylammonium triiodide (MWCNT/ $TOAI_3$ /CIL),[49] for determination of methimazole in serum samples by using CuNPs-P-L-Argenine/MWCNTs/GCE,[50] for determination of meloxicam in the spiked plasma samples by using CPE modified with gold nanoparticles modified choline chloride functionalized graphene oxide (AuNPs-ChCl-GO),[51] for detection of celecoxib in the spiked plasma samples by using CPE modified with AgNPs modified ChCl-GO,[52] for determination of acyclovir in spiked serum and urine samples by using GCE modified with a polymeric chitosan film decorated with MWCNTs + TiO_2 NPs,[53] for determination of isoxsuprine in spiked serum samples by using GCE modified with MWCNTs decorated with Ag NPs,[54] for determination of theophylline in spiked urine samples by using GQD modified SPE,[55] for determination of methyldopa in spiked urine samples by using a GCE modified with Cu/TiO_2 nanocomposite,[56] for simultaneous determination of epinephrine, paracetamol and tryptophan in spiked urine and serum samples by using modified CPE with Ni-doped Lewatit FO36 nano ion exchange resin,[57] for determination of favoxate hydrochloride and tolterodine tartrate in spiked urine samples by using ferrocene modified carbon paste electrode (FMCPE),[58] for determination of meclizine in spiked urine samples by using bare graphite screen-printed electrode (GSPE),[59] for determination of L-carnitine in spiked serum samples by using anodized Ni/Cu alloy electrode,[60] for determination of rituxan in spiked serum samples by using PAMAM dendrimer/RGO nanocomposite modified PGE,[61] for determination of azithromycin in serum samples by using GCE modified with MIP / gold nanourchin (GNU) / GO,[62] for determination of estradiol valerate in spiked blood samples by using CPE modified with terbium carbonate ($Tb_2(CO_3)_3$) nanoparticles,[63] for determination of methimazole in the spiked serum samples by using MIP- PGE,[64] for determination of diclofenac sodium in spiked serum samples by using pretreated pencil graphite electrode (PPGE),[65] for determination of repaglinide in spiked serum and urine samples by using a MIP-GCE,[66] and for determination of tizanidine in spiked serum samples by using GCE modified with a thin film of MWCNTs coated with an electro-polymerized layer of titan yellow-doped overoxidized polypyrrole (OPPY).[67]

Tahmasebi et al.[68] reported a DPV method for determination of propylthiouracil in spiked urine samples after electromembrane extraction (EME) by copper nanoparticles- decorated hollow fibers and Shahraki et al.[69] used a homogeneous liquid–liquid microextraction (HLLME) method (Figure 4) before DPV measurement of nitrazepam in spiked urine samples by using GCE.

It should be noted that the stripping analyses have the lowest limits of detection of any of the commonly used electroanalytical techniques. Gholivand et al.[70] developed a ASV method for simultaneous determination of warfarin and mycophenolic acid by using CPE modified with b-cyclodextrin/multi-walled carbon nanotubes/cobalt oxide nanoparticles (b-CD/MWCNTs/Co_3O_4 NPs/CPE) as working electrode. LOD of reported method for warfarin and mycophenolic acid in serum and urine samples were 0.02 and 0.03 µmol L^{-1}, respectively. Mirzajani et al.[71] used a ASDPV for determination of metformin with LOD of 14 nmol L^{-1} by using CPE modified with γ-Fe_2O_3@ hydroxyapatite/Cu (II) nanocomposite (γ-Fe_2O_3@HAp/ Cu (II)). The average found amount in urine samples 6 h after taking a 500 mg metformin hydrochloride tablet was reported to be of 6.7 µmol L^{-1}. A similar work was also performed by Azadmehr et al.[72] for determination of ceftizoxime using PGE modified by hollow gold nanoparticles (HGNPs)/rGO in plasma samples with a very low LOD of 0.35 pmol L^{-1}. Najari et al.[73] developed a DPASV method for determination of docetaxel by using Au-MWCNTs/GCE as working electrode in spiked serum and urine samples with LOQ and LOD of 0.3 µmol L^{-1} and 90 nmol L^{-1}.

In some reports, an identical pharmaceutical compound is measured with different methods or in different samples which some of these along with analytical details are given here.

Shahbakhsh et al.[74] used a DPV method for determination of acetaminophen in the spiked serum and urine samples by using modified GCE with polydopamine (PDA)-MWCNTs. LOD of 30 nmol L^{-1} was reported for this study. While Hasanpour et al.[75] detected acetaminophen with same method in the same samples by using modified CPE with copper ferrite - copper oxide (CuO-$CuFe_2O_4$) nanoparticles with LOD of 7 nmol L^{-1}. Another DPV method reported for acetaminophen determination in plasma samples was developed by Hassaninejad-Darzi et al.[76] by using ZSM-5 nanozeolite and TiO_2 nanoparticles modified CPE as working electrode. They reported a LOD of 580 nmol L^{-1} for acetaminophen.

Shahbakhsh et al.[74] also measured dopamine in the spiked serum and urine samples with LOQ and LOD of 0.6 µmol L^{-1} and 20 nmol L^{-1}, respectively. While Taheri et al.[77] used a DPV method for dopamine determination by using the modified Au electrode with spindle-shaped gold nanostructure in serum sample with LOQ and LOD of 0.16 nmol L^{-1} and 13 pmol L^{-1}, respectively. They reported a dopamine serum level of 0.16 nmol L^{-1} for healthy subjects and 0.35 – 1.23 nmol L^{-1} for patient subjects.

Akbarian et al.[78] used a SWV method by using CPE modified with NiO-SWCNTs as conductive mediator and 2, 4-dimethyl-N/-[1-(2, 3-dihydroxy phenyl) methylidene] aniline (DDPM) as electro-catalyst (NiO-SWCNTs/DDPM/CPE) for determination of diclofenac in spiked serum samples in the presence of morphine and mefenamic acid. Mostafavi et al.[79] used a DPV method by using a diclofenac - MIP–CPE for diclofenac determination in urine samples. The reported LOD for SWV and DPV method by mentioned working electrode are 0.008 µmol L^{-1} and 3.7 µmol L^{-1}. Other same drug measured with different methods is flutamide. Afzali et al.[80] used a SWV method by using CPE modified with CuO NPs/GO/polyaniline (PANI) as working electrode for determination of flutamide in spiked urine samples with LOD of 14 pmol L^{-1} and Rezaeifar et al.[81] develoed a DPV method by using a sensitive electrochemical sensor based on hyperbranchedpolyglycerol functionalized- graphene oxide developed, using ionic liquid mediated hollow fiber-pencil graphite electrode (HF/ HBP-GO/PGE) for determination of flutamide in spiked plasma samples with LOD of 0.029 µmol L^{-1}.

Chromatographic methods

Chromatographic methods which are physical methods for the separation of mixture based on the concept of partition coefficient, have always been a more specialized approach than optical and electrochemical methods. In our researches, it is found that more than half of the studies in 2018 were performed by these methods. It can be related to high selectivity and specificity of chromatographic methods in compared with other methods. The reported works are classified in the following sections and details of each study are given in Table 3.

High-performance liquid chromatography (HPLC)-UV detector

Rezaee et al.[82] validated a HPLC-UV method for determination of thiopental in spiked plasma samples without any preconcentration procedures. Haghbin et al.[83] used a HPLC-UV method for determination of morphine in plasma and cerebrospinal fluid of patients addicted to opiates undergoing surgery. Ramezani et al.[84] developed a micellar HPLC-UV method for isocratic isolation of some cardiovascular drugs including losartan, hydrochlorothiazide and triamterene with different polarities in spiked plasma samples. They used a green mobile phase additive i.e. deep eutectic solvent (DES) to improve the chromatographic behavior of the drugs. As a new type of solvent, DESs have been attracted great attention because of its excellent physical and chemical properties. They can also be used as an extractor solvent in various sample preparation procedures. Rajabi et al.[85] used an air agitated-emulsification microextraction (AA-EME)-HPLC-UV for simultaneous extraction/determination of amphetamine and methamphetamine by using a DES comprising choline chloride and phenylethanol (ChCl:

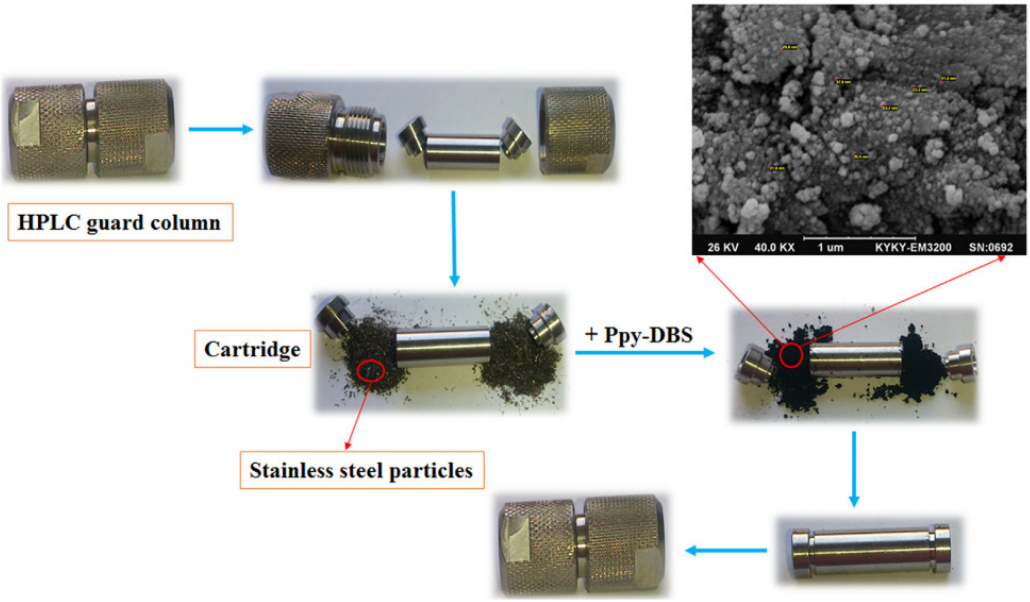

Figure 5. Schematic diagram of the packed column preparation for the packed EC-IT-SPME system. Adapted with permission from Asiabi et al.[94] Copyright (2018) Elsevier.

Ph-EtOH). A similar work was performed by Ghoochani et al.[86] for simultaneous determination of escitalopram, desipramine and imipramine in spiked plasma samples. Khataei et al.[87] used a three-phase hollow fiber liquid phase microextraction (HF-LPME) based on two immiscible organic phases by using DES for determination of dydrogesterone and cyproterone acetate in spiked plasma and urine samples. However, other extractor solvents (other than DESs) had also been used before HPLC methods in sample preparation step. For example: Rezaee Moghadam et al.[88] used a DLLME coupled with HPLC-UV method for simultaneous determination of enrofloxacin and ciprofloxacin in tissue samples. Mirparizi et al.[89] used a tandem dispersive liquid–liquid microextraction (TDLLME) followed by HPLC-UV for determination of rivastigmine and donepezil in spiked plasma samples. Similar works were performed for determination of duloxetine in plasma after extraction by using 1-butyl-3-methylimidazolium hexafluoroborate-based ultrasound-assisted in situ solvent formation microextraction (IL-UA-ISFME) procedure,[90] and for determination of sodium closantel in spiked blood and urine samples after extraction by a UA-DLLME.[91] Amiri Pebdani et al.[92] used a low-density solvent such as 1-undecanol as the extraction solvent for dispersive liquid-phase microextraction based on sequential injection solidified floating organic drop (DLPME-SI-SFOD) of phenobarbital and phenytoin from spiked urine and plasma samples prior to determination by HPLC-UV method. Haghnazari et al.[93] reported a continuous liquid–phase microextraction (CLPME) with toluene as an extraction solvent lighter than water for determination of amitriptyline and clomipramine in urine samples.

As has been already described a sample preparation step is often required before chromatographic analysis. In addition to LLME methods, solid phase extraction (SPE) methods have also been used to separate the analytes of interest from possible interferences of biological samples. Asiabi et al.[94] used an electrochemically controlled in-tube solid phase microextraction (EC-IT-SPME) method (modified with nanostructured polypyrrole film), followed by HPLC-UV for simultaneous determination of diclofenac and mefenamic acid in spiked samples of urine and plasma. Schematic diagram of the packed column preparation for the packed EC-IT-SPME system are shown in Figure 5. The stainless steel particles were placed in the column and used as the working electrode. Then, a nanostructured polypyrrole-dodecyl benzene sulfonate (Ppy-DBS) was coated on the inner surface of a stainless steel tube and the surface of the stainless steel particles using cyclic voltammetry in an aqueous solution of 0.2 mol L^{-1} pyrrole and 0.01 mol L^{-1} SDBS as the supporting electrolyte. After the electrochemical deposition, the packed column coated with the Ppy-DBS film was washed and dried for the online microextraction. Golzari Aqda et al.[95] used a cellulose acetate fibers for on–line micro solid–phase extraction (MSPE) of some nonsteroidal anti–inflammatory drugs such as naproxen, diclofenac and mefenamic acid and their determinations by HPLC-UV method in plasma and urine samples. Ghani et al.[96] reported a HPLC-UV method for determination of diclofenac in urine and plasma samples after its microextraction by a Mg-Al-layered double hydroxide (LDH)- GO mixed-matrix membrane. Similar works were performed for determination of L-cysteine in spiked plasma and urine samples after extraction by using zinc organic polymer (Zn-MOP) as a sorbent,[97] for simultaneous determination of losartan and valsartan in spiked plasma samples after extraction with stir bar sorptive extraction (SBSE) based on acrylate monolithic polymer,[98] for determination of vincristine in spiked plasma and urine samples by using a solvent bar microextraction (SBME) with water at pH 10.7 as donor phase, 1-octanol as the

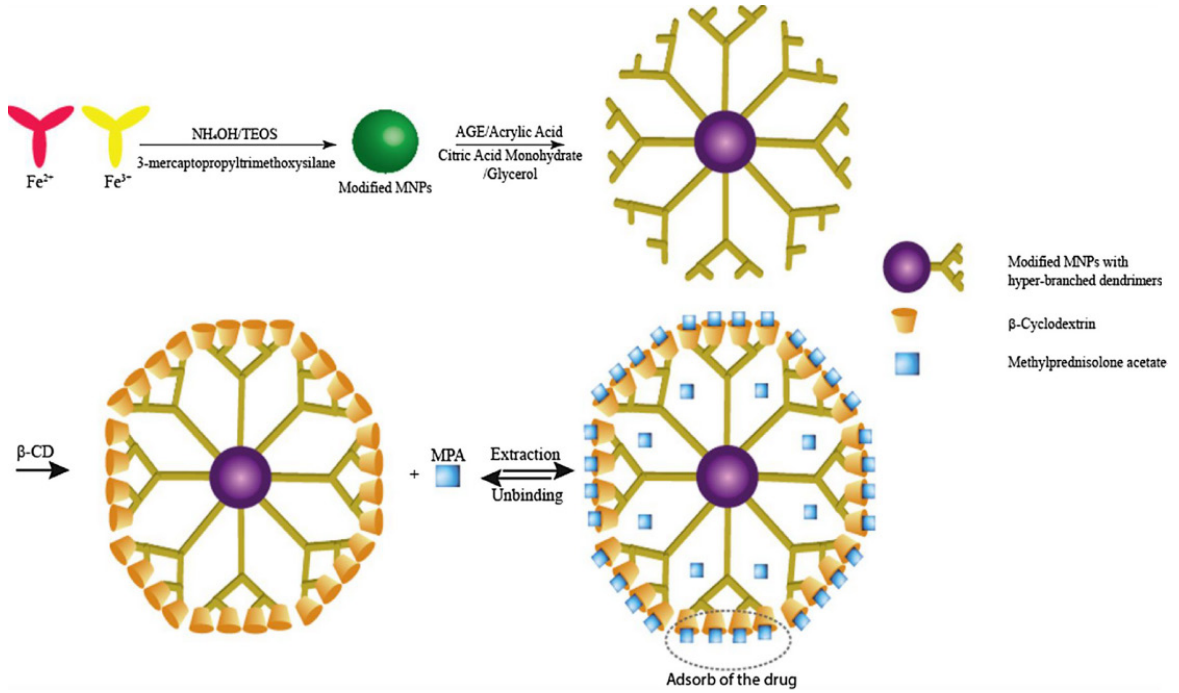

Figure 6. Illustration of synthesis and grafting process of AA@MNPs-D/β-CD. Adapted with permission from Baimani et al.[109] Copyright (2018) Elsevier.

supported liquid membrane and an aqueous receiving phase (pH =3.1),[99] for simultaneous determination of oxazepam, flurazepam, and diazepam in hair, nail and blood samples after extraction by using a vortex assisted dispersive solid phase extraction approach based on crab shell powder as micro-sorbent,[100] for determination of rizatriptan in spiked plasma and urine samples after extraction by using SBME technique,[101] for determination of diclofenac, ibuprofen, and mefenamic acid in spiked plasma and urine samples after extraction with centrifugeless ultrasound-assisted dispersive micro solid-phase extraction with Zn-Al-LDH-MWCNT nanohybrid as a nanosorbent coupled with salting-out ultrasound-assisted liquid-liquid microextraction based on solidification of a floating organic droplet,[102] for determination of zolpidem in spiked plasma and serum samples after an EME procedure,[103] for determination of phenazopyridine after a extraction procedure by coupling EME and packing a sorbent (a cation exchanger) with reported value of 33 μg L^{-1} and 237.3 μg L^{-1} for phenazopyridine in urine sample of patients treated with it respectively after 9 and 12 hour,[104] for determination of diclofenac in spiked plasma and urine samples after extraction by HF-LPME,[105] for determination of valproic acid in spiked plasma samples after extraction by using HF-EME method,[106] and for determination of exemestane, letrozole and paclitaxel in spiked urine samples after extraction by using three-phase HF-LPME based on two immiscible organic solvents (acetonitrile and n-dodecane).[107]

Magnetic solid phase extraction (MSPE) as an alternative method for SPE have mostly been used for sample preparation before HPLC-UV. Safari et al.[108] synthesized a magnetic framework composites (Fe_3O_4@TMU-10) microspheres for simultaneous MSPE of some tricyclic antidepressants including amitriptyline and imipramine in plasma and urine samples prior to HPLC-UV determination. The value for imipramine concentration in the investigated urine samples was reported to be 55 μg L^{-1}. A similar work was reported by Baimani et al.[109] for determination of methylprednisolone acetate after extraction with an allyl glycidyl ether/acrylic acid grafted on modified magnetite nanoparticles with functionalized dendrimer conjugated β-CD (AA@MNPs-D/β-CD) from spiked plasma and urine samples (see Figure 6). Similar works were performed for simultaneous extraction of morphine and codeine in the serum and blood samples after extraction by using magnetite (Fe_3O_4)/rGO/silver nano-composite (Ag NC) (with reported level of 0.610 – 1.075 μg L^{-1} for morphine and 0.746 – 1.124 μg L^{-1} for codeine in blood samples of addicted person to opium),[110] for determination of baclofen in spiked urine samples after extraction with superparamagnetic molecularly imprinted biopolymer (SMIBP),[111] for simultaneous determination of dasatinib, erlotinib, and nilotinib in spiked plasma, serum and urine samples after extraction by using bio-inspired magnetic sorbent doped melamine-phytate supermolecular aggregate (Fe_3O_4/ MPA) applied for UA-DMSPE,[112] for determination of buprenorphine and norbuprenorphine in spiked plasma samples after extraction by poly (para-phenylenediamine)-modified Fe_3O_4 nanoparticles (P*p*PDA/Fe_3O_4),[113] for determination of rivaroxaban in spiked plasma and urine samples after extraction with a high generation thermo-sensitive dendrimer of magnetic nanoparticles/ poly(N-isopropylacrylamide)/ G10 4-amino-2,3-dimethyl-1phenyl-3-pyrazoline-5-one (MNPs/PNIPAAm/G10-ADMPhP) nanocomposite,[114] for

determination of chlordiazepoxide in spiked urine samples after extraction with a magnetic MIP,[115] for determination of letrozole in spiked plasma and urine after extraction with magnetic nanoparticles (MNPs)/ polyethylene glycol (PEG)ylated dendrimer,[116] and for determination of amphetamine in spiked urine samples after extraction by using a magnetic block copolymer (poly ethylene glycol-b-poly (N,N-dimethylaminoethylmethacrylate-co-maleic acid) (magnetic PEG-b-P(DMAEMA-co-MA)).[117]

HPLC- diode array detector (DAD)

DAD as a multi-wavelength detector is used for obtaining spectral profiles of molecular mixtures or chromatographically separated samples providing a facility to check peak purity. The spectrum can be employed to show the optimal wavelength for the detection within one run. Ghorbani et al.[118] used an ultrasonic assisted magnetic dispersive solid phase microextraction method coupled with HPLC-DAD for determination of some serotonin–norepinephrine reuptake inhibitor drugs such as duloxetine, venlafaxine and atomoxetine in urine samples. They used a magnetic p-phenylenediamine functionalized reduced graphene oxide quantum dots@ Ni nanocomposites (MrGOQDs–PD@ Ni) as a nanosorbent for preconcentration of investigated analytes. Jalilian et al.[119] developed a similar method for determination of nortriptyline, cetirizine, naproxen, diclofenac and ibuprofen after simultaneous extraction with MWCNT/ MNP@poly(2-aminopyrimidine) composite (MWCNTs/ Fe_3O_4@PAPy). Alahyari et al.[120] used a DLLME coupled with HPLC-DAD method with chloroform and acetone as extraction and disperser solvents for determination of some opioids such as morphine, codeine and methadone in urine sample. The concentration of morphine, codeine and methadone in three postmortem urine samples are reported to be in the range of 2.5 – 17.8 μg mL^{-1}, 0.9 – 5.3 μg mL^{-1}, 1.9 – 10.4 μg mL^{-1}, respectively. Rezaei et al.[121] developed a HPLC-DAD method for simultaneous determination of prednisolone and methylprednisolone and mycophenolic acid in spiked plasma samples. They used a fast-elution protocol and smart methodology based on multivariate curve resolution-alternating least square (MCR-ALS) modeling for this analysis. A similar work was performed for determination of three immunosuppressant drugs including tacrolimus, everolimus and cyclosporine A in whole blood samples using intelligent chemometrics resolving of coeluting peaks in the presence of blood interferences.[122]

HPLC-fluorescence detector (FD)

Fluorescence detectors are the most sensitive and low cost among the existing HPLC detectors. Typically, fluorescence sensitivity is 10 -1000 times higher than that of the UV detector for strong UV absorbing materials. Some of the chromatographic studies are performed by HPLC-FD methods. For example: Rastkari et al.[123] developed a HPLC-FD method for determination of lisinopril.

They used a magnetic polydimethylsiloxane/OH-functionalized multiwalled carbon nanotubes nanocomposite (PDMS/MWCNT-OH-NC) for extraction of lisinopril from spiked plasma samples and 4-fluoro-7-nitro-2,1,3-benzoxadiazole for precolumn derivatization.

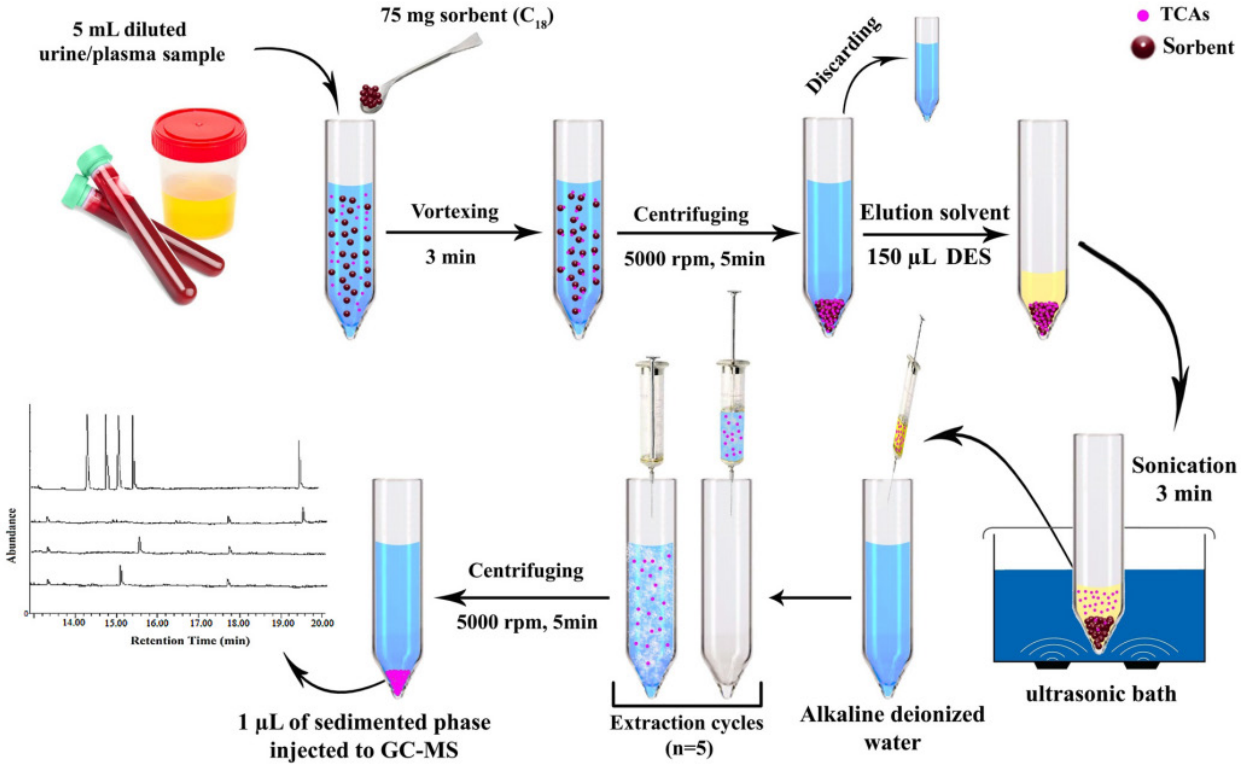

Figure 7. Schematic illustration of the developed DSPE-DES-AALLME preconcentration procedure. Adapted with permission from Mohebbi et al.[109] Copyright (2018) Elsevier.

Table 3. Analytical details of chromatographic methods used for pharmaceutical analysis in biological samples conducted in Iran (year 2018).

Method	Analyte	Sample	Linear range	Detection limit	Remarks	Ref.
HPLC-UV	Thiopental	Plasma	-	0.001 μg mL^{-1}	Mixture of acetonitrile: methanol: potassium di-hydrogen phosphate buffer (10 mM, pH 2.7) (40:10:50) as mobile phase, Drug monitoring at 280 nm,	82
HPLC-UV	Morphine	Plasma Cerebro-spinal Fluid	-	-	-	83
Micellar HPLC-UV	Losartan Hydro-chlorothiazide Triamterene	Plasma	7 – 20 μg mL^{-1} 0.5 – 20 μg mL^{-1} 0.5 – 17 μg mL^{-1}	0.6 μg mL^{-1} 0.04 μg mL^{-1} 0.08 μg mL^{-1}	0.12 mol L^{-1} SDS, 5% acetonitrile, 4% DES, and 2% acetic acid as mobile phase, monitoring at 254 nm	84
HPLC-UV	Amphetamine Methamphetamine	Plasma	15 – 2000 ng mL^{-1} 8 – 3000 ng mL^{-1}	5.0 ng mL^{-1} 2.0 ng mL^{-1}	0.05 mol L^{-1} phosphate buffer solution (pH 5.5) and acetonitrile (20:80) as mobile phase, Drug monitoring at 210 nm. Using AA-EME for analyte extraction	85
HPLC-UV	Escitalopram Desipramine Imipramine	Plasma	10 – 5000 ng mL^{-1} 15 – 8000 ng mL^{-1} 15 – 6000 ng mL^{-1}	3.0 ng mL^{-1} 4.5 ng mL^{-1} 4.0 ng mL^{-1}	Acetonitrile and 0.05 mol L^{-1} phosphate buffer solution (pH 3.0) as mobile phase, druge monitoring at 220 nm. Using AA-EME-LD-DES for analyte extraction.	86
HPLC-UV	Dydrogesterone Cyproterone acetate	Urine Plasma	1.0 – 500 μg L^{-1} 2.0 – 500 μg L^{-1} 2.0- 500 μg L^{-1} 4.0 – 500 μg L^{-1}	0.5 μg L^{-1} 1.0 μg L^{-1} 1.0 μg L^{-1} 2.0 μg L^{-1}	Water: acetonitrile (38:62) as mobile phase, analyte monitoring at 282 nm, Using HF-LPME with DES for analyte preconcentration.	87
HPLC-UV	Enrofloxacin Cipro-floxacin	Tissue	-	5.3 μg kg^{-1} 7.3 μg kg^{-1}	Acetonitrile and phosphoric acid buffer (0.01 M, pH 3) (25:75% v/v) as mobile phase, Drug monitoring at 278 nm, Using a DLLME method for analyte extraction	88
HPLC-UV	Rivastigmine Donepezil	Plasma	2 – 1100 ng mL^{-1} 3 -1100 ng mL^{-1}	0.5 ng mL^{-1} 1 ng mL^{-1}	A mixture of 0.05 mol L^{-1} phosphate buffer (pH 4.0) and acetonitrile (70:30) as mobile phase, Drug monitoring at 210 nm, Using TDLLME for analyte extraction	89
HPLC-UV	Duloxetine	Plasma	2.0 – 1500 μg L^{-1}	0.8 μg L^{-1}	A mixture of ammonium formate (10 mmol L^{-1}) and acetonitrile (40:60) with pH of 3.8 as mobile phase, Drug monitoring at 230 nm, Using IL-UA-ISFME for analyte extrcaction.	90
HPLC-UV	Sodium closantel	Blood Urine	10 – 3000 μg L^{-1}	1 μg L^{-1}	A mixture of acetonitrile, water and ammonium acetate buffer of pH 4.3 (45:45:10, v/v) as mobile phase, Drug monitoring at 240 nm.	91
HPLC-UV	Phenobarbital Phenytoin	Urine Plasma	1 – 300 μg L^{-1} 2 – 400 μg L^{-1}	0.35 μg L^{-1} 1.2 μg L^{-1}	40% acetonitrile and 60% water as mobile phase, Drug monitoring at 220 nm, Using DLPME-SI-SFOD for analyte extraction	92
HPLC-UV	Amitriptyline Clomipramine	Urine	1 – 800 μg L^{-1}	0.35 – 0.7 μg L^{-1}	A mixture of 60% buffer containing 50.0 mmol L^{-1} sodium dihydrogen phosphate with pH 4.2 containing 0.50 mmol L^{-1} sodium dodecyl sulfate (SDS) and 40% acetonitrile as mobile phase, Drug monitoring at 210 nm, Using CLPME for analyte extraction.	93
HPLC-UV	Diclofenac Mefenamic acid	Urine Plasma	1.3 -200 μg L^{-1} 1.1 – 200 μg L^{-1} 2.1 – 200 μg L^{-1} 1.8 – 200 μg L^{-1}	1.0 μg L^{-1} 0.6 μg L^{-1} 1.60 μg L^{-1} 1.10 μg L^{-1}	10 mmol L^{-1} phosphate buffer (pH= 4.5) and acetonitrile (40:60) as mobile phase, drig monitoring at 210 nm, Using in-tube SPE for extraction of analyte	94
HPLC-UV	Naproxen Diclofenac Mefenamic acid	Urine Plasma	4 – 1000 μg L^{-1} 6 – 1000 μg L^{-1} 4 – 1000 μg L^{-1} 6 – 1000 μg L^{-1} 8 – 1000 μg L^{-1} 8 – 1000 μg L^{-1}	1.0 μg L^{-1} 1.5 μg L^{-1} 1.1 μg L^{-1} 1.8 μg L^{-1} 2.4 μg L^{-1} 2.0 μg L^{-1}	MeOH-water (80:20 v/v, pH = 3) as mobile phase, Drug monitoring at 230 and 275 nm. Using cellulose acetate fibers for analyte extraction.	95

Table 3 Continued.

HPLC-UV	Diclofenac	Urine Plasma	1 – 200 µg L^{-1} 2 – 200 µg L^{-1}	0.23 µg L^{-1} 0.57 µg L^{-1}	Mixture of acetonitrile-water (60:40 v/v) as mobile phase, Drug monitoring at 276 nm. Using LDH/GO mixed-matrix membrane for analyte extraction	96
HPLC-UV	L-cysteine	Plasma Urine	4 – 1000 µg L^{-1}	0.76 µg L^{-1}	Water-methanol (95:5, v/v; pH 7.0) as mobile phase, drug monitoring at 230 nm, Using Zn-MOP for analyte extraction.	97
HPLC-UV	Losartan Valsartan	Plasma	24 – 1000 ng mL^{-1} 91 – 1000 ng mL^{-1}	7 ng mL^{-1} 27 ng mL^{-1}	Mixture of acetonitrile and acetate buffer (pH 3.8; 10 mM) as mobile phase, drug monitoring at 220 nm, using SBSE for analyte extraction.	98
HPLC-UV	Vincristine	Plasma Urine	0.05 – 5 mg L^{-1}	0.015 mg L^{-1}	30% water-diethyl amine at a ratio of 59:1 (pH = 7.5, component A) and 70% methanol (component B), drug monitoring at 297 nm, Using SBME for analyte extraction.	99
HPLC-UV	Oxazepam Flurazepam Diazepam	Hair Nail Blood	0.79 – 20 µg mL^{-1} 0.04 – 10 µg mL^{-1} 0.04 – 10 µg mL^{-1} 3.15 – 20 µg mL^{-1} 2.11 – 20 µg mL^{-1} 1.07 – 20 µg mL^{-1} 0.3 – 15 µg mL^{-1} 3.38 – 15 µg mL^{-1} 3.8 – 15 µg mL^{-1}	0.23 µg mL^{-1} 0.011 µg mL^{-1} 0.012 µg mL^{-1} 0.94 µg mL^{-1} 0.61 µg mL^{-1} 0.31 µg mL^{-1} 0.1 µg mL^{-1} 1.1 µg mL^{-1} 1.2 µg mL^{-1}	Mixture of acetonitrile- methanol–pure water optimized on (80:10:10, v/v/v) as mobile phase, drug monitoring at 238 nm, Using crab shell powder as micro-sorbent for analyte extraction.	100
HPLC-UV	Rizatriptan	Plasma Urine	50 – 10000 ng mL^{-1}	15 ng mL^{-1}	10 mM sodium dihydrogen phosphate, methanol, and acetonitrile (40:40:20) as mobile phase, Drug monitoring at 226 nm, Using solvent bar microextraction for analyte extraction	101
HPLC-UV	Diclofenac Ibuprofen Mefenamic acid	Plasma Urine	0.8 – 2000 ng mL^{-1} 0.8 – 2500 ng mL^{-1} 0.5 – 2000 ng mL^{-1}	0.2 ng mL^{-1} 0.2 ng mL^{-1} 0.1 ng mL^{-1}	Acetonitrile and 0.05 mol L^{-1} phosphate buffer solution (pH 3.0) (65:35) as mobile phase, drug monitoring at 220 nm, Using UA-Dµ-SPE-S-UA-LLME-SFO for analyte extraction	102
HPLC-UV	Zolpidem	Urine Plasma	10 – 1000 ng mL	3 ng mL^{-1}	Mixture of methanol and 50 mmol L^{-1} ammonium acetate buffer containing 0.1 % v/v triethylamine at a pH = 3.7 as mobile phase, Drug monitoring at 300 nm, Using an EME method for analyte extraction	103
HPLC-UV	Phenazopyridine	Urine	10 – 1000 µg L^{-1}	0.2 µg L^{-1}	A mixture of acetonitrile and a 10 mmol L^{-1} acetate buffer with the pH of 5.5 (60:40, v/v), Drug monitoring in 395 nm, analyte extraction by coupling of EME and SPE procedure	104
HPLC-UV	Diclofenac	Plasma Urine	50 – 2000 ng mL^{-1}	2.8 ng mL^{-1}	Methanol- potassium hydrogen phosphate (pH 2.5) (70:30, v/v) as mobile phase, Drug monitoring at 254 nm, Using HF-LPME for analyte extraction	105
HPLC-UV	Valproic acid	Plasma	0.5 – 10 µg mL^{-1}	0.2 µg mL^{-1}	A mixture of acetonitrile and 80 mM sodium dihydrogen phosphate buffer (pH = 3.5) in the ratio 43:57 (v/v) as mobile phase, Drug monitoring at 210 nm, Using a HF-EME for analyte extraction.	106
HPLC-UV	Exemestane Letrozole Paclitaxel	Urine	1.8 – 200 µg L^{-1} 0.9 – 200 µg L^{-1} 1.2 – 200 µg L^{-1}	0.6 µg L^{-1} 0.3 µg L^{-1} 0.4 µg L^{-1}	A mixture of acetonitrile and ultra-pure water (50:50) as mobile phase, Drug monitoring at 240 nm, Using HF-LPME for analyte extraction.	107
MSPE-HPLC/UV	Amitriptyline Imipramine	Urine Plasma	8 – 800 ng mL^{-1}	5 ng mL^{-1}	10 mmol L^{-1} phosphate buffer (pH 4.0) containing 25 mmol L^{-1} $KClO_4$ and acetonitrile (65:35) as mobile phase. Monitoring at 220 nm. Using magnetic framework Fe_3O_4@ TMU-10 as a nanosorbent for analyte extraction	108
HPLC-UV	Methylprednisolone acetate	Plasma Urine	0.01 – 80 µg mL^{-1}	0.75 ng mL^{-1}	n-Butyl chloride, water-saturatedn-butyl chloride, tetrahydrofuran, methanol, and glacial acetic acid (95:95:14:7:6) as mobile phas, Drug monitoring at 254 nm, Using AA@MNPs-D/β-CD for analyte extraction	109
HPLC-UV	Morphine Codeine	Urine Blood	0.01 – 10 µg L^{-1}	0.0018 – 0.0021 µg L^{-1}	40% acetonitrile and 60% (v/v) phosphate buffer (0.01 mol L^{-1}, pH 6.5) as mobile phase, Drug monitoring at 285 nm, Using Fe_3O_4/rGO/Ag NC for analyte extraction	110

Table 3 Continued.

HPLC-UV	Baclofen	Urine	1 – 2500 µg L^{-1}	0.26 µg L^{-1}	Water-methanol mixture (47:53, v/v), Drug monitoring at 225 nm, Using SMIBP for analyte extraction	111
HPLC-UV	Dasatinib Erlotinib Nilotinib	Plasma Serum Urine	0.4 – 100 µg L^{-1} 0.3 – 120 µg L^{-1} 0.6 – 150 µg L^{-1}	0.15 µg L^{-1} 0.12 µg L^{-1} 0.2 µg L^{-1}	A mixture of 25 mmol L^{-1} potassium dihydrogenphosphate buffer, adjusted to pH 3.0 ± 0.1 and methanol (25:75, v/v) as mobile phase, Drug monitoring at 220 nm, Using Fe_3O_4/ MPA for analyte extraction.	112
HPLC-UV	Buprenorphine Norbuprenorphine	Plasma	3 – 150 ng mL^{-1} 1 – 120 ng mL^{-1}	0.8 ng mL^{-1} 0.3 ng mL^{-1}	Phosphate buffer (pH 3.4) and acetonitrile (50:50, v/v) as mobile phase, Drug monitoring at 280 nm, Using P*p*PDA/Fe_3O_4 for analyte extraction	113
HPLC-UV	Rivaroxaban	Urine Plasma	0.5-80 mg L^{-1}	3.06 µg L^{-1}	A mixture of 65 mL mobile A and 35 mL mobile B (mobile A was 5 mL acetic acid in 1000 mL water and mobile B was 70 mL acetonitrile in 30 mL mobile A. drug monitoring at 248 nm. Using MNPs/PNIPAAm/G10-ADMPhP nanocomposite for analyte extraction	114
HPLC-UV	Chlordiazepoxide	Urine	0.006 – 10 µg mL^{-1}	0.0014 µg mL^{-1}	Acetonitrile: water (50:50, v/v) as mobile phase, Drug monitoring at 254 nm, Using MMIP for analyte extraction.	115
HPLC-UV	Letrozole	Plasma Urine	0.1 – 20 µg mL^{-1}	0.92 ng mL^{-1}	A mixture of acetonitrile and HPLC grade water (30:70 v/v), Drug monitoring at 230 nm, Using MNPs/PEGylated dendrimer for analyte extraction.	116
HPLC-UV	Amphetamine	Urine	30 – 2000 ng mL^{-1}	8 ng mL^{-1}	A mixture of acetonitrile/phosphate buffer solution (10 mmol L^{-1}) at a ratio of 15/85 (V/V), Using magnetic PEG-b-P(DMAEMA-co-MA for analyte extraction.	117
HPLC-DAD	Duloxetine Venlafaxine Atomoxetine	Urine	2.9 – 560 ng mL^{-1} 2.3 – 580 ng mL^{-1} 3.4 – 560 ng mL^{-1}	1.0 ng mL^{-1} 0.7 ng mL^{-1} 1.1 ng mL^{-1}	20 mmol L^{-1} phosphate buffer solution (pH7.5), acetonitrile and methanol (65:12:23 v/v) as mobile phase, drug monitoring at 217 nm. Using MrGOQDs–PD@ Ni nanocopmosite as nanosorbent for analyte extraction	118
HPLC-DAD	Nortriptyline Cetirizine Naproxen Diclofenac Ibuprofen	Plasma Urine	0.25 – 1500 µg L^{-1}	0.07 – 3.5 µg L^{-1}	NaH_2PO_4 buffer (10 mmol L^{-1}, pH 2.5) and acetonitrile (45:55, v/v) as mobile phase, Drug monitoring at 210 – 230 nm, Using MWCNTs/Fe_3O_4@PAPy for analyte extraction.	119
HPLC-DAD	Morphine Codeine Methadone	Urine	0.5 – 100 µg mL^{-1}	25 µg L^{-1} 9 µg L^{-1} 10 µg L^{-1}	Acetonitrile (A) and 0.05 mol L^{-1} phosphate buffer at pH 2.3 (B) with gradient elution mode: 0–7 min, A% 10 and B % 90; 7–8 min, A% 20 and B% 80; 8–15 min, A% 20 and B% 80; 15–16 min, A% 37 and B% 63; 16–40 min, A% 37 and B% 63; 40–45 min, A% 10 and B% 90. Using a DLLME for analyte extraction.	120
HPLC-DAD	Prednisolone Methylprednisolone Mycophenolic acid	Plasma	2.8 – 400 µg L^{-1} 3.8 – 600 µg L^{-1} 0.1 – 4.5 µg L^{-1}	0.9 µg L^{-1} 1.3 µg L^{-1} 0.03 µg L^{-1}	A 60:40 (v/v) mixture of acetonitrile and water (0.02 mol L^{-1} KH_2PO_4 (pH=3.7) buffer solution as mobile phase, Drug monitoring at 210 – 400 nm.	121
HPLC-DAD	Tacrolimus, Everolimus Cyclosporine A	Blood	2.1 – 6.3 µg L^{-1} 2.4 – 200 µg L^{-1} 36 – 1250 µg L^{-1}	0.56 µg L^{-1} 0.08 µg L^{-1} 7.6 µg L^{-1}	90% acetonitrile and 10% phosphate buffer (pH = 3.5) as mobile phase, Drug monitoring at 210 – 400 nm.	122
HPLC-FD	Lisinopril	Plasma	3 – 1000 ng mL^{-1}	1 ng mL^{-1}	A mixture of methanol–sodium dihydrogen phosphate (pH 3.0; 0.005 mol L^{-1}; 75:25, v/v), Drug monitoring at ex/em 470/530 nm, Using PDMS/MWCNT-OH-NC as sorbent.	123
HPLC-FD	Bisphosphonates	Urine Serum	5 – 2500 µg L^{-1}	1.4 µg L^{-1}	A mixture of acetonitrile and water with the ratio of 10:90 (v/v), Drug monitoring at ex/em 335/470 nm, Using ZNPs as a DMSPE sorbent for analyte extraction.	124
HPLC-FD	Buprenorphine	Urine	1 – 1000 ng mL^{-1}	0.21 ng mL^{-1}	A mixture of acetate buffer (50 mM; pH 5): acetonitrile: triethylamine (52:47.95:0.05, v/v/v), Drug monitoring at ex/em: 210 / 345 nm. Using magnetic MIP for analyte extraction.	125
GC - FID	Imipramine Desipramine	Plasma	0.005 – 5 µg mL^{-1} 0.01 – 4 µg mL^{-1}	0.003 µg mL^{-1} 0.007 µg mL^{-1}	Temperature program: Detector temperature, 270 °C; splitless injection (1 µl) at 270 °C; initial oven temperature was 80 °C for 1 min and then increased to 280 °C with the rate of 30 °C min^{-1} and held for 5 min.	126

Table 3 Continued.

GC - FID	Nicotine	Hair	0.01 – 30 μg g^{-1}	0.002 μg g^{-1}	Temperature program was started at 100 ºC (held 1 min) and then raised to 280 ºC at a rate 20 ºC min^{-1} and held constant for 2 min. Both injector and FID detector were set at 280 ºC. Using HS-SPME for analyte extraction.	127
GC - FID	valproic acid 3-heptanone	Plasma	0.25 – 100 mg L^{-1} 0.10 – 100 mg L^{-1}	0.065 mg L^{-1} 0.023 mg L^{-1}	Temperature program: The oven temperature was programmed from 70 °C held (for 2 min) to 200 °C at a rate of 15 °C min^{-1} and then increased to 300 °C at a rate of 20 °C min^{-1} for cleaning column. Using a DLLME for analyte extraction.	128
GC - FID	Sodium valproate	Plasma Urine	0.5 – 500 μg mL^{-1} 0.1 – 200 μg mL^{-1}	0.22 μg mL^{-1} 0.05 μg mL^{-1}	Temperature program: The oven temperature was programmed from 50 °C (held for 2 min) to 210 °C at a rate of 10 °C min^{-1} and held at 210 °C for 3 min; then, the temperature was raised with a rate of 15 °C min^{-1} to a final temperature of 290 °C that was held for 1 min. Using AA-LLME for analyte extraction.	129
GC - FID	Amitriptyline Imipramine Clomipramine	Plasma	0.003 – 1.5 μg mL^{-1} 0.006 – 1.5 μg mL^{-1} 0.01 – 1.5 μg mL^{-1}	0.001 μg mL^{-1} 0.002 μg mL^{-1} 0.003 μg mL^{-1}	Temperature program: initial temperature 70°C (held for 2 min), then increased to 300°C at a rate of 20°C min^{-1} and held for 2 min. Using LLLE coupled with DLLME method	130
GC - FID	Amitriptyline Doxepin	Plasma	10 – 3000 ng mL^{-1}	1.0 ng mL^{-1}	Temperature program: The GC oven was initially held at temperature of 100 °C for 1 min, programmed to 240 °C at 20 °C min^{-1}, further programmed to 260 °C at a rate of 2 °C min^{-1}, The injector and detector temperatures were 280 °C and 290 °C, respectively. Using AA-LLME for analyte extraction.	131
GC-FID	Clomipramine Imipramine	Urine	0.5 – 750 ng mL^{-1}	0.15 ng mL^{-1}	Temperature program: The initial column oven temperature was adjusted at 150 °C then increased by 30 ºC min^{-1} to 300 º C and hold for 1 min. The temperature of injection port and FID were 280 and 300 º C, respectively. The splitless mode of injection was used for 1 min and then split valve was opened at the split ratio of 10:1. Ultrapure nitrogen gas at 20 psi constant pressure was applied as carrier gas. Using EME coupled with EA-LLME for analyte extraction.	132
GC- MS	Amitriptyline Nortriptyline Clomipramine	Urine Plasma	27 - 5000000 ng L^{-1} 108 – 5000000 ng L^{-1}	8 – 15 ng L^{-1} 32 – 60 ng L^{-1}	Temperature program: The column oven temperature was initially held at 100 º C for 1 min, programmed at a rate of 50 ºC min^{-1} to 190 º C, then programmed at a rate of 5 ºC min^{-1} to 225 ºC (held for 3 min), followed by a rate of 20 ºC min^{-1} to 300 ºC and held for 5 min. MS operational conditions were: electron ionization at 70 eV, ionic source temperature: 250 ºC; transfer line temperature: 260 ºC; mass range: m/z 30 – 400; acquisition rate: 20 Hz. Detector voltage: -1700 V. Using DSPE coupled with DES-based AA-LLME for analyte extraction.	133
GC-MS	Methamphetamine	Urine	5 – 1500 μg L^{-1}	1.5 μg L^{-1}	Temperature program: The column temperature program was as follows: 2 min at 50 °C, raised up to 250 °C at a rate of 20 °C min^{-1}, and kept at 250 °C for 3 min. A split injection mode with split ratio of 1:5 was selected for the introduction of 1 μL of the extracts.The injector was kept at 260 °C. The electron impact ionization energy was 70 eV and the transfer line temperature was 180 ºC. Chromatograms were recorded in full-scan mode (40–550 m/z). Using HLPME with DPA for analyte extraction.	134
CE-DAD	Pregabalin	Serum	1.5 – 100 μg mL^{-1}	0.8 μg mL^{-1}	Each new capillary have been rinsed with NaOH (1.0 mol L^{-1}) for 30 min, deionized water for 20 min and background electrolyte (10 mmol L^{-1} 5-ASA, 1 mmol L^{-1} CTAB and 4 % (w/v) tri-sodium citrate) for 30 min, sequentially. Drug monitoring at 215 nm.	135

Table 3 Continued.

GC-MS	Ketoprofen Naproxen Diclofenac Ibuprofen	Urine Plasma	1 – 400 μg L^{-1} 1 – 400 μg L^{-1} 2 – 400 μg L^{-1} 2 – 400 μg L^{-1} 2.5 – 400 μg L^{-1} 2 – 400 μg L^{-1} 5 – 400 μg L^{-1} 5 – 400 μg L^{-1}	-	Temperature program: The GC oven was initially held at 70 °C for 2 min, then ramped to 280 °C at 25 °C min^{-1}, and finally held for 15 min. Chromatograms were recorded in the range of m/z 160 – 380. Using a DLLME associated with back extraction for analyte extraction.	[136]

Rahbar et al.[124] used a similar method for determination of bisphosphonates in spiked urine and serum samples after extraction by using zirconia nanoparticles (ZNPs) as a DMSPE. The extracted analyte derivatized by o-phthalaldehyde in the presence of 2-mercaptoethanol at basic medium to form fluorescent species. In other study, buprenorphine in urine samples was determined by this method after extraction by magnetic MIP, magnetite cores surrounded by polyamidoamine.[125]

Gas chromatography (GC)

GC is a technique of preference for separation of volatile compounds. FID and MS are the commonly used detectors for this method. By using a GC – FID; Ahmadi et al.[126] developed a method for simultaneous determination of imipramine and desipramine after extraction from spiked plasma samples by using $Fe_3O_4/SiO_2/C_8/NH_2$ MNPs. Ghiasvand et al.[127] determined nicotine in hair of smoker subjects after extraction by using sulfonated graphene – polyaniline nanocomposite coated fiber as a headspace (HS)- SPME sorbent. The reported concentration in two hair sample taken from smoker subjects is about 1.12 – 6.80 μg g^{-1}. Feriduni et al.[128] determined valproic acid and its main metabolite (3-heptanone) in plasma samples after extraction by a DLLME method with chloroform (as extractor) and acetonitrile (as dispersant). Abbaspour et al.[129] also determined sodium valproate in plasma and urine samples by same method after extraction with an air assisted (AA)-LLME by using chloroform as extractor. They reported a sodium valproate concentration in the ranges of 9.9 – 34.7 μg mL^{-1} and 0.5- 6.7 μg mL^{-1} for analyzed patient's plasma and urine samples, respectivly. Farajzadeh et al.[130] reported a GC-FID method for determination of amitriptyline, imipramine, and clomipramine in plasma samples after extraction by liquid–liquid–liquid extraction (LLLE) (acetonitrile as extraction solvent, n-hexane as clean up and co–extraction solvent and sodium sulfate as phase separating agent) followed by DLLME method by 1,2–dibromoethane as preconcentration solvent. The measured concentration in plasma taken from subjects treated with these drugs was 147 μg mL^{-1} for imipramine, 100 – 243 μg mL^{-1} for clomipramine and 95 – 191 μg mL^{-1} for amitriptyline. Mofazzeli et al.[131] developed a GC-FID method for determination of trace amounts of amitriptyline and doxepin in plasma samples. They used an AA-LLME by using toluene as extraction solvent prior to measurement. Nojavan et al.[132] validated a method for simultaneous quantification of clomipramine and imipramine in spiked urine samples. They used a tandem sample preparation method of EME combined with electro-assisted liquid-liquid micro-extraction (EA-LLME) for extraction and preconcentration of analytes. Another sample preparation combinational method was performed by Mohebbi et al.[133] for determination of amitriptyline, nortriptyline and clomipramine. The extraction procedure is schematically shown in Figure 7. As can be seen a DSPE method with C_{18} sorbent combined with DES (prepared from choline chloride and 4-chlorophenol)–based AA-LLME method was used for extraction of amitriptyline, nortriptyline and clomipramine prior to GC-MS determination. Other studies performed by using GC-MS method including determination of methamphetamine in urine samples after extraction by a homogeneous liquid–phase microextraction (HLPME) with the aim of dipropylamine (DPA) as a solvent with switchable hydrophilicity[134] and determination of ketoprofen, naproxen, diclofenac and ibuprofen in plasma and urine samples after extraction by DLLME method by a mixture of acetone (disperser solvent) and n-dodecane/TOPO (extraction solvent 95:5, v/v) associated with a back extraction.[135]

Capillary electrophoresis (CE)

CE is liquid separation technique that separates ions based on their electrophoretic mobility with the use of an applied voltage. Main advantage of this method high separation efficiency, high resolution, short analysis time, low consumption of sample and solvent, and low sensitivity to sample matrix.[137] Sargazi et al.[135] developed a micellar electrokinetic chromatographic method with indirect UV detection for pregabalin determination in spiked serum samples. They used 5-aminosalicylic acid (5-ASA) as probe for indirect UV detection. A decrease proportional with the concentration of pregabalin in the background absorbance related to 5-ASA used to the quantification of pregabalin in serum samples.

Conclusion

The present review depicts the reported analytical methods which had developed and validated for the determination of pharmaceutical compounds in the biological fluids by Iranian analysts in year 2018. The literature are classified based on applied methodology and instrumentation and analytical properties of each report were tabulated. According to this review it has been concluded that most of the used methods for pharmaceutical analysis are of chromatographic methods coupled with various detectors and electrochemical methods, as these methods provides best reliability, and sensitivity in compared with optical methods. A brief study on the journals of investigated articles show that mean value for their impact factors is 3.2 ± 1.6 and Journal of Chromatography A (impact factor: 3.716), Analytica Chimica Acta (impact factor: 5.123) possess the first and second ranks with considering the number of articles published in 2018 and Journal of Molecular Liquids (impact factor: 4.513)/ Sensors and Actuators B (impact factor: 5.667)/ Journal of Electroanalytical Chemistry (impact factor: 3.235)/ Talanta (impact factor: 4.244)/ Journal of the Iranian Chemical Society (impact factor: 1.593) are in the third ranks.

Conflicts of Interests

There are no conflicts of interest to declare.

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