Publications Template

#	Research Title	Field	Abstract	Year of Publication Publishing	Publishing Link "URL"
1	**GRAPHICAL SPECTROPHOTOMETRIC DETERMINATION OF IONIZATION CONSTANTS OF ENTACAPONE AND ITS QUANTIFICATION IN TABLETS AND PLASMA**	Analytical Chemistry	Two graphical spectrophotometric techniques have been developed for the determination of ionization constants of entacapone (ENP). The first one depends on plotting the relationships between absorbance values at three λmax against different pH values. Consequently, the pKa values are corresponding to the pH of drug solution at the inflection points in these plots. The second one is based on plotting the derivative spectrophotometric titration curves and interpolating pKa at D1/2. Both techniques have been successfully applied to evaluate two ionization constants of ENP. On the other hand, three selective, sensitive and validated spectrophotometric methods have been developed for the determination of drug	2012	https://www.wjpps.com/Wjpps_controller/abstract_id/136

			in bulk powder and tablets. Method A depends on measuring the first derivative spectrophotometric peak-to-peak amplitudes of ENP in methanolic solution at 368–405 nm. This method is highly sensitive, so it allows the determination of ENP in human plasma, where linear correlation was achieved in the range 0.9-2.4 µg mL-1. Method B is pH-induced difference spectrophotometry (ΔA) and its first derivative (ΔD1). This method involves measurement of analytical signal values of drug alkaline solution against its acidic solution from peak to peak at (299-365) nm and (266-340) nm for ΔA and ΔD1, respectively. Method C is based on the oxidative coupling reaction with 3-methylbenzothiazolin-2-one hydrazone (MBTH) in the presence of Ceric ammonium sulphate, Ce (IV), as an oxidant to produce deep-green colored species measurable at 535 nm. All the proposed methods were validated in compliance		

			with ICH guidelines.		
2	**Validated High-Performance Thin-Layer Chromatographic Method for the Evaluation of Oseltamivir Pharmaceutical Formulations Counterfeited with Ascorbic Acid Compared with a Colorimetric Method**	Analytical Chemistry	Aselective high-performance thin-layer chromatographic (HPTLC) method has been established for the quantitative determination of oseltamivir phosphate (OST) without interference of ascorbic acid (ASC) added to some of its counterfeit pharmaceutical formulations. Chromatographic separation was performed on precoated silica gel 60 GF_{254} plates with methanol–water–ammonia 6:4:0.05 (v/v) as mobile phase at ambient temperature. The developed plates were scanned and quantified at 254 nm. Experimental conditions such as band size, mobile phase volume,	2013	https://link.springer.com/article/10.1556/JPC.26.2013.5.7

			chamber saturation time, migration of solvent front, etc. were critically studied, and the optimum conditions were selected. Asatisfactory resolution was obtained with R_F 0.70 and 0.83 for OST and ASC, respectively. Also, HPTLC-band detection method has been established for rapid qualitative assay of OST using ninhydrin spray. On the other hand, a colorimetric method has been established using bromocresol green (BCG) as rapid, accurate, and selective comparative method. Both methods were validated for linearity, accuracy, precision, selectivity, and		

			specificity. The calibration plots were linear between 5.00 and 14.00 µg band^{-1} and 6.00–18.00 µgmL^{-1} for the HPTLC and colorimetric methods, respectively. The detection limits were 1.80 µg band^{-1} and 2.00 µg mL^{-1}, for the HPTLC and colorimetric methods, respectively. The simplicity of the proposed methods suggest its application in quality control analysis of OST in its capsules and granules for oral suspension.		
3	**Validated spectrophotometric methods for the evaluation of oseltamivir counterfeit pharmaceutical capsules**	Analytical Chemistry	Four rapid, reliable and economical spectrophotometric methods have been established for the quantitative determination of Oseltamivir phosphate (OST) without the interference of ascorbic acid (ASC) found in	2014	https://www.sciencedirect.com/science/article/pii/S1110093114000027

			some of its counterfeit capsules. The first method involves the use of derivative spectrophotometry with the zero-crossing technique where OST was easily determined using its ^{1}D (Dk= 3) at 219 nm. The second method is based on a first-order derivative ratio spectrophotometry (^{1}DD, Dk= 5) where 218 nm was selected for its quantification, while the third method applies a more advanced spectrophotometric method based on the ratio difference spectrophotometry (RD) in which the difference in absorbance ratio was measured between 217 and 210 nm. In the fourth method, difference spectrophotometric method (DA) is applied by subtracting		

			absorbance at 252 from that at 263 nm where the difference in absorbance was zero for ASC. The proposed methods were validated for linearity, accuracy, precision and selectivity. Synthetic mixtures of different proportions and commercial capsules were assayed by the proposed methods and the results revealed good accuracy and repeatability of the developed methods.		
4	**HPTLC and Spectrophotometric Estimation of Febuxostat and Diclofenac Potassium in Their Combined Tablets**	Analytical Chemistry	An accurate, precise, rapid, specific and economic high-performance thin-layer chromatographic (HPTLC) method has been developed for the simultaneous quantitative determination of febuxostat (FEB) and diclofenac	2016	**https://pubmed.ncbi.nlm.nih.gov/27406127/**

			potassium (DIC). The chromatographic separation was performed on precoated silica gel 60 GF_{254} plates with chloroform–methanol 7:3 (v/v) as the mobile phase. The developed plates were scanned and quantified at 289 nm. Experimental conditions including band size, mobile phase composition and chamber-saturation timewere critically studied, and the optimum conditions were selected. A satisfactory resolution (R_s = 2.67) with R_F 0.48 and 0.69 and high sensitivity with limits of detection of 4 and 7 ng/band for FEB and DIC, respectively, were		

			obtained. In addition, derivative ratio and ratio difference spectrophotometric methods were established for the analysis of such a mixture. All methodswere validated as per the ICH guidelines. In the HPTLC method, the calibration plots were linear between 0.01–0.55 and 0.02–0.60 µg/band, for FEB and DIC, respectively. For the spectrophotometric methods, the calibration graphs were linear between 2–14 and 4–18 µg/mL for FEB and DIC, respectively. The simplicity and specificity of the proposed methods suggest their application in quality control analysis of FEB and DIC in their		

			rawmaterials and tablets. A comparison of the proposed methods with the existing methods is presented.		
5	**High-performance thin-layer chromatographic methods for the determination of febuxostat and febuxostat/diclofenac combination in human plasma**	Analytical Chemistry	Two simple, sensitive and specific high-performance thin-layer chromatographic (HPTLC) methods were developed for the determination of febuxostat (FEB) individually, and simultaneously with diclofenac (DIC) in human plasma. Method A presents the first HPTLC-ultraviolet attempt for FEB determination in human plasma. FEB was separated from endogenous plasma components (at hR_F=70) with ethyl acetate-methanol-water (9:2:1, v/v) mixture as mobile phase and quantified by densitometry at its λ_{max} (315 nm). Method B is considered the first attempt for the simultaneous determination of FEB and DIC in human plasma. A mixture of petroleum ether-chloroform-ethyl acetate-formic acid (7.5:1:2.5:0.25, v/v) was used as the mobile phase. The two drugs were separated at hR_F of 39	2018	https://pubmed.ncbi.nlm.nih.gov/29660667/

			and 60 for FEB and DIC, respectively. FEB and DIC were quantified by densitometry at their isoabsorptive point (289 nm). FEB calibration plots were linear between 0.1 and 7 μgmL^{-1} in both methods A and B. In method B, DIC showed linear response in the range of 0.08–8 μgmL^{-1}. Sample preparation was performed by liquid-liquid extraction using diethyl ether. Both methods did not record any interference from plasma matrix, the studied drugs' metabolites or their decomposition products. They were successfully applied for the determination of the studied drugs in healthy male volunteers after oral administration of FEB or FEB/DIC dosage forms. FEB plasma concentration increased significantly when given with DIC. The proposed methods provided very simple, rapid and cheap approaches that might be attractive for the future pharmacokinetic and bioavailability studies of FEB and/or DIC.		

6	**A novel HPLC-DAD method for simultaneous determination of febuxostat and diclofenac in biological samples: pharmacokinetic outcomes**	Analytical Chemistry	**Aim:** To develop a simple HPLC-DAD method for simultaneous determination of febuxostat (FEB) and diclofenac (DIC) in biological samples to assess pharmacokinetic outcomes of their coadministration. **Methodology & results:** Sample preparation was performed by liquid–liquid extraction. Drugs analysis was done on C18 column usingmethanol-formic acid pH 2.1 (76:24, v/v) as mobile phase and time-programmed UV detection. Lower limits of quantitation for FEB and DIC were 10 and 20 ng/ml, respectively. Baseline pharmacokinetics were similar to published data on either drug alone.	2019	https://pubmed.ncbi.nlm.nih.gov/30475064/

			Coadministration led to more than twofold increase in FEB C_{max} and AUC together with a reduced hepatic uptake in rats. **Conclusion:** DIC interfered with initial distribution and terminal clearance of FEB potentially due to reduced FEB hepatic uptake.		
7	**A novel voltammetry offline coupled MALDI/TOF MS characterization of electrochemical reaction products and the voltammetric determination of febuxostat in human plasma**	Analytical Chemistry	A simple offline coupling voltammetry-MALDI/TOF MS procedure is presented for studying electrochemical reactions. It was utilized for the characterization of the electro-reduction products of febuxostat in methanolic acetate buffer (0.1 M, pH 5). The MS analysis reveals that the carboxylic and nitrile groups are the electro-reducible groups at −0.9338 and −1.5503 V with the conversion to aldehydic and amino groups, respectively. The developed voltammetric method was validated and applied successfully for the drug determination	2019	https://www.sciencedirect.com/science/article/abs/pii/S0039914018311330

			in pharmaceutical tablets and real plasma samples within the linearity ranges 0.03–2 and 0.4–5 µg mL−1, respectively.		
8	**Simultaneous determination of methocarbamol and aspirin in presence of their pharmacopeialrelated substances in combined tablets using novel HPLC-DAD method**	Analytical Chemistry	Objective and Significance: Methocarbamol (MET) and aspirin (ASP) are widely used as a muscle relaxant combination. The USP reports guaifenesin (GUA) and salicylic acid (SAL) as related substances and hydrolytic products of MET and ASP, respectively. This work aimed at developing and validating a simple and sensitive RP-HPLC method for the determination of both drugs as well as their related substances (at their pharmacopeial	2019	https://www.tandfonline.com/doi/abs/10.1080/03639045.2018.1535603

			limits) in their bulk powders, laboratory prepared mixtures, and MET-ASP combined tablets. Methods and Results: Chromatographic separation was achieved in less than 9 min with the required resolution, peak symmetry, and accuracy on C_{18} column using isocratic elution system of diluted acetic acid (pH 3.2): acetonitrile at the ratio of 79: 21, v/v, at a flow rate of 1 mL/min. Detection was achieved with photodiode array at 233nm for MET, GUA, and SAL and at 273nm for ASP. The developed method has been validated as per		

			ICH guidelines and the calibration plots were linear over the concentration ranges of 2–150, 0.4–30, 25–450, and 0.2–27 lg/mL for MET, GUA, ASP, and SAL, respectively. Conclusion: The optimized method proved to be specific, robust and precise for the quality control of the studied drugs in pharmaceutical preparations to ascertain that their related substances are not exceeding the permitted pharmacopeial limits.		
9	**Simultaneous micro-determination of eplerenone and torsemide in their combined tablets using**	Analytical Chemistry	Sudden death is common in patients with Congestive heart failure, occurring at a rate of six to nine times that of the general population. Day by	2020	https://www.sciencedirect.com/science/article/abs/pii/S0026265X20300977

	HPTLC-Dual wavelength spectrodensitometric and spectrophotometric methods		Day, new combined therapies of more advanced drug generations are involved in treatment. Recently, the Eplerenone/ Torsemide binary therapy is displacing the older Spironolactone/ Frusemide treatment trend of heart disease because it proved better tolerability and potency. This necessitates fast development of analytical methods that are capable of simultaneous determination of such important drug mixture. In this study, validated analytical methods have been established for simultaneous quantitation of eplerenone (EPL) and torsemide (TOR). One of the methods represents the first highperformance thin-layer chromatographic (HPTLC) attempt for EPL-TOR simultaneous		

			estimation. A mixture of ethyl acetate – methanol– chloroform (8:2:1, v/v/v) was applied as mobile phase. EPL and TOR were well resolved and scanned at 242 nm (at hR_F 77) and 288 nm (at hR_F 36), respectively. Other proposed methods include solvent-induced difference, derivative ratio and ratio subtraction (complemented with isosbetstic point) spectrophotometric methods that were adopted as faster, simpler and more economic alternatives for the routine analysis of the increasingly used binary EPL-TOR medication, especially in the developing countries. Both HPTLC and spectrophotometric methods successfully determined both drugs without interference		

			from one another. All methods were validated as per ICH guidelines. In the HPTLC method, calibration plots were linear between 50-600 and 35-800 ng/band for EPL and TOR, respectively. The spectrophotometric methods proved excellent linearity in the range of 3-25 and 3-30 μg/mL for EPL and TOR, respectively. Within all the proposed methods, ratio subtraction spectrophotometric method was the most sensitive one for EPL (LOD = 0.75 μg/mL). Regarding TOR, direct spectrophotometry achieved the lowest LOD (0.71 μg/mL). The high accuracy, simplicity and low cost of the proposed methods recommend their application in the industrial analysis of EPL and TOR combined dosage forms. A full comparison with the		

			reported techniques shows the privileges of the suggested analytical procedures.		
10	**Gradient HPLC-DAD method for quantification of novel oral anticoagulant "Edoxaban" in plasma: Its selective determination in presence of sixteen coadministered drugs**	Analytical Chemistry	As an anticoagulant, Edoxaban (EDX) is a high risk drug that may cause a life-threatening bleeding. Also, it is prescribed as a chronic therapy for atrial fibrillation and venous thromboembolism patients. They are special population that needs appropriate care and optimum dosing of EDX. Hence, its monitoring in the patient plasma is fundamental, especially in emergency and special circumstances. However, such patient mostly receives many drugs of different pharmacological classes, side by side with EDX. This study represents the first attempt to quantify EDX in	2020	https://www.sciencedirect.com/science/article/abs/pii/S1570023220305791

			plasma without interference of the plasma matrix or concomitant medications. An accurate RPHPLC-DAD method was developed for this purpose. It succeeded to monitor EDX level, selectively, without interference of plasma matrix or 16 of its frequently co-administered drugs. All drugs were extracted from plasma samples by protein precipitation followed by evaporation and concentration. EDX was well resolved from the co-administered drugs on C_8 column using linear gradient elution of methanol and phosphate buffer (pH 4), at a flow rate of 1 mL/min. EDX appeared at retention time 9.6 min and was quantified at its λ_{max} (290 nm). It exhibited a linear response over the concentration range		

			of 0.15-2.2 μg/mL plasma which covers the reported therapeutic concentration. The suggested method fulfilled the US FDA guidelines for bioanalytical method validation. The developed method is fully discussed in comparison with the reported techniques. An in vivo study was performed to ensure applicability of the method on real plasma samples without interference from plasma matrix, co-administered drugs or the expected metabolites. It presented a unique selectivity of the method that guarantees accurate laboratory monitoring of EDX in plasma in almost all combined treatments including such novel oral anticoagulant drug.		

11	**Development of hybrid spectrofluorimetric method for simultaneous determination of Valsartan and Sacubitril in LCZ696 tablets**	Analytical Chemistry	A hybrid Spectrofluorimetric method was developed for the simultaneous determination of binary mixtures, without prior separation steps. It coupled synchronous spectrofluorimetry with derivative ratio mathematical treatment. The method was applied successfully to quantify a new model binary mixture consisting of Valsartan (VAL) and Sacubitril (SAC). This mixture was recently approved by FDA as LCZ696. It added a great value in reducing morbidity and mortality in resistant heart failure (HF) patients. First derivative ratio synchronous fluorescence was measured at 258–295 (peak-to-peak) and 204 nm for VAL and SAC, respectively. ICH guidelines were fulfilled for the method validation. VAL and SAC showed linear responses in the range of 60–200 and 20–200 ng mL_{-1}, respectively. The proposed method was compared, in details, with the reported ones. Its high accuracy, selectivity, simplicity and affordable cost recommend method	2020	https://www.sciencedirect.com/science/article/abs/pii/S1386142521003243

			application in large-scale routine analysis of LCZ696 tablets. Moreover, reliable application of this new integrated spectrofluorimetric method suggests expansion of its application for various therapeutic combinations and different matrices.		