Application of Flow Injection Technique in Pharmaceutical Analysis. Part II.: Other spectroscopic methods and electroanalytical detection

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Abstract

A review dealing with applications of flow injection technique (FIA) in drug analysis is presented. Comments on the application of automated FIA systems in the quality control of pharmaceuticals and in pharmaceutical research are involved in the Part I. Part II comprises papers using fluorescence, AAS/AES, MS, FT-IR spectroscopy, amperometry, potentiometry, voltammetry and gravimetry as detection techniques.

Keywords: Flow injection analysis; automation; drugs; pharmaceutical analysis.

1. Introduction

In Part I [1] the versatility of flow injection analysis (FIA) [2] was mentioned and the development of FIA in the field of quality control of pharmaceuticals together with a wide applicability for automation in analytical control laboratories were pointed out. The review (Parts I and II) covers period between 1997 and 2000. The previous reviews have been published by our group [3-5] and other authors [6-8]. The most recent monograph has appeared in 1996 [9].

All papers cited in this review are sorted by detection techniques and all main parameters concerning the drug assayed, sample matrix, techniques or reagents used, linear calibration range, detection limit and sample throughput are tabulated.

The Part I covered papers employing spectrophotometry and chemiluminescence as detection techniques most commonly used in FIA assays of pharmaceuticals. The Part II deals with other spectroscopic detection methods that were employed in FIA systems, such as fluorescence, AAS/AES, MS, FT-IR spectroscopies, and electroanalytical techniques represented by amperometry, potentiometry and voltammetry.

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In addition gravimetric detection belongs to methods that were successfully implemented in FIA systems developed for the measurement of pharmaceutically important substances in solutions, pharmaceutical formulations or human body fluids.

2. Comments on detection techniques in the FIA systems

Generally, the most widely spread detection technique used in FIA is spectrophotometry in visible spectral region and this holds also for FIA quantification of pharmaceuticals. Spectrophotometry enables to detect and quantify many drugs and related substances by using simple or even rather complicated derivatisation routines leading to the formation of coloured reaction products "on-line" in the flow system. Selectivity of such detection is often improved by integrating extraction (including solid-phase extraction) or dialysis modules in the FIA manifolds.

In the recent three years chemiluminescence became the second most frequently used detection method in FIA of drugs. Thanks to the selectivity and sensitivity of relatively small number of known chemiluminescence reactions employing appropriate enhancers or sensitizers a number of important

Table 1: FIA assays with fluorescence detection

drug	matrix	λ (nm)	reagents/ technique	linear range (mg/l)	detection limit (mg/l)	ST (h^{-1})	ref.
adrenaline	solution	510 (330)	oxidation O_2	0.05 – 15 20 – 40	0.02	107	10
anthracyclines **daunorubicin** **doxorubicin** **epirubicin**	pharmaceuticals urine	615 (393)	Eu^{3+} optosensor	 30 – 800 nM 20 – 800 nM 20 – 800 nM	 16 nM 10 nM 10 nM	12	11
ascorbic acid	pharmaceuticals	464 (340)	thionine blue	0.14 – 8.8		80	12
ascorbic acid (L-)	pharmaceuticals	530 (430)	phenylenediamine	0.02 – 2	0.014		13
ascorbic acid (L-)	tablets	419 (227)	Tl^{3+}	0.14 – 2.8 µM		45	14
benzodiazepines **diazepam** **nitrazepam** **oxazepam**	tablets	 370 (241) 389 (283) 418 (237)		 10 – 125 10 – 150 25 – 150	 5 5 10	60	15
L-carnitine	pharmaceuticals	460 (340)	NADH	1 – 100 µM			16
chlorpromazine	injections, tablets	374 (253)	UV irradiation	10 – 600 µg/l	5.4 µg/l	90	17
fluticasone	solution		o-phtalaldehyde				18
folic acid	tablets			0.1 – 40		28	19
hydrazides **iproniazid** **isoniazid**	solutions pharmaceuticals	395 (320)	H_2O_2	14 10	0.008 0.005	24	20
lisurid	tablets		dissolution				21
naproxen	pharmaceuticals	353 (329)		10 – 100			22
noradrenaline	solution	512 (397)	Fe^{3+}	0.5 – 75		84	23
oxytetracycline	tablets		optical fibre sensor	0.2 – 200 µM			24
paracetamol	pharmaceuticals	426 (360)				60	25
phenothiazines	pharmaceuticals urine	424 (350)		0.5 – 480	0.06		26
phenytoin	serum	514 (470)	immunoreaction	2.5 – 40			27
pyridoxine	pharmaceuticals	385 (295)	solid phase	5 – 1800 ng/ml	0.33 ng		28
quinine	pharmaceuticals	451 (350)	beta-cyclodextrin	3 nM – 20 mM	0.2 ng/ml		29
reserpine	tablets	490 (386)	acetone	0.01 – 0.75	0.45	90	30
salicylamide **salicylic acid**	pharmaceuticals	415 (260)		0.01 – 0.32 0.04 – 1		35 45	31
selenium	food supplements			0.3 – 1300 ng/ml	0.4 ng/ml		32
sulphamethazine	tablets	345 (287)	sulphite	20 – 2000 µg/l	3.5 µg/l		33
thiamine	tablets injections	440 (370)	irradiation Hg lamp	0.01 – 10	0.11 µg/l	100	34
thiamine	pharmaceuticals		o-phtalaldehyde	0.2 – 6 ng/ml	0.1 ng/ml		35
tryptophan	pharmaceuticals			0.1 – 50	25 ng/ml		36
vitamin B_1, B_2, B_6	tablets		fibre optic sensor				37
vitamin K_1	pharmaceuticals	412 (305)	irradiation	1 – 100 µM	0.4 µM		38
vitamin K_3	pharmaceuticals	425 (325)		0.1 – 18	0.005	70	39

pharmaceuticals could be assayed by FIA technique and the number of papers dealing with FIA – chemiluminescence is surprisingly surpassing that of FIA – fluorimetric applications in the recent three years.

In Table 1 substances analysed with fluorescence detection in the FIA systems are listed. Fluorimetry provides excellent sensitivity and selectivity for FIA assays of low concentrations of drugs making the method especially suitable for performing automated content uniformity tests, drug liberation studies and clinical assays of low concentrations of drugs or their metabolites in blood or urine.

Table 2: FIA assays with AAS/AES detection

drug	matrix	λ (nm)	reagents/ technique	linear range (mg/l)	detection limit (mg/l)	ST (h^{-1})	ref.
ascorbic acid (L-)	tablets	248	Fe^{3+}	0.5 – 25	0.2	90	40
ascorbic acid	tablets, injections		Cr^{3+}	0.3 – 60			41
bismuth	pharmaceuticals urine	223.1		0 – 100 ng/ml	0.32 ng/ml	150	42
bismuth	tablets	223.1					43
lead	food supplements				0.03 µg/l		44
sodium potassium calcium magnesium	parenteral solutions						45
vitamin B_6	tablets, injections		MnO_2				46
zinc copper	multivitamin tablets						47
zinc	multivitamin tablets			1 – 20	0.998	18	48

Table 3: FIA assays with mass spectrometric detection

drug	matrix	reagents/ technique	linear range (mg/l)	detection limit (mg/l)	ST (h^{-1})	ref.
benzalkonium	pharmaceuticals	ion-spray	5 – 100 ng/ml	1.2 ng/ml		49
lead	pharmaceuticals	ICP				50
nifedipine	human plasma	N_2	0.5 – 100 ng/ml	0.5 ng/ml		51
topiramate	human plasma	electro spray, N_2	1 – 30			52

Table 4: FIA assays with FT-IR spectroscopic detection

drug	matrix	ν (cm^{-1})	reagents/ technique	linear range (mg/l)	detection limit (mg/l)	ST (h^{-1})	ref.
ketoprofen		1900 – 1500	stopped flow	0 – 10 mg/ml	0.02 – 0.04 mg/ml	42	53
paracetamol	pharmaceuticals	1254.1	Fe^{3+}	0.5 – 50 mM			54
propyphenazone caffeine	tablets	1595 1712			0.06 0.45		55

Other spectroscopic detection techniques used in the FIA of drugs are AAS/AES, FT-IR and MS (see Tables 2 – 4). They are not widely spread most probably because of the need of rather costly instrumentation and necessity to design and build special flow cells that are usually commercially unavailable. Nevertheless, here the FIA concept is often favourably utilised for on-line processing of samples prior to their determination.

Tables 5, 6 and 7 show FIA papers using electrochemical techniques such as amperometry, potentiometry and voltammetry for the determination of different drugs. Enzyme or immunoreagent-based biosensors and amperometric detectors involving chemically modified electrodes are successfully used for the analysis of biologically active substances including pharmaceuticals in the last decade.

Gravimetry with piezoelectric detectors (see Table 8) seems to become a modern detection tool in FIA enabling the assay of several drugs with relatively high sample throughput.

Table 5: FIA assays with amperometric detection

drug	matrix	reagents/ technique	linear range (mg/l)	detection limit (mg/l)	ST (h^{-1})	ref.
acetylsalicylic acid	pharmaceuticals				150	56
adrenaline	pharmaceuticals	Fe^{3+}	0.3 – 20	0.1	153	57
adrenaline	pharmaceuticals		0.1 μM – 0.5 mM	10 pM		58
adrenaline **L-dopa** **dopamine**	pharmaceuticals					59
ascorbic acid	solution	modified electrode		0.25 pM		60
ascorbic acid	tablets		0.7 μM – 1 mM	0.3 μM		61
ascorbic acid	pharmaceuticals	modified electrode	0.02 – 1 mM			62
ascorbic acid	tablets	biamperometry	0.01 – 2 mM	8 μM		63
ascorbic acid **paracetamol**	pharmaceuticals	biamperometry	0.3 – 20 0.4 – 25	0.02 0.02		64
ascorbic acid	pharmaceuticals	modified electrode	0.5 μM – 1 mM	0.47 μM		65
ascorbic acid **dopamine** **epinephrine** **dipyrone**	pharmaceuticals	array of microelectrodes				66
chloramine T	pharmaceuticals	biamperometry	up to 65	0.5	220	67
chloramphenicol	pharmaceuticals	biamperometry	up to 8	0.05	68	68
glucose	parenteral solutions	wall-jet cell	0.1 – 1 M		30	69
mercury	pharmaceuticals	biosensor	10 – 60 ppb		15	70
metronidazole	pharmaceuticals	biamperometry	0.2 – 8	0.008	50	71
naltrexone	pharmaceuticals		20 nM – 10 μM		90	72
norephedrine	drugs	wall jet cell	up to 3 μg	0.15 μg		73
penicillin G	solution					74
perindopril	solution	enzymatic biosensor	20 – 100 nM	10 nM	72	75
S-perindopril	solution	enzymatic biosensor	200 pM – 80 nM		65	76
streptomycin	solution	binding with BSA	0.001 – 1 mM	0.8 μM		77

Table 6: FIA assays with potentiometric detection

drug	matrix	reagents/ technique	linear range (mg/l)	detection limit (mg/l)	ST (h^{-1})	ref.
acetylsalicylic acid	pharmaceuticals	ISE tubular electrode	2.5 – 500 M			78
acetylsalicylic acid	pharmaceuticals	ISE	4 – 40 mM		28	79
bromide	human serum		9 – 24 mM		20	80
chlorpromazine	injections	PbO_2	0.01 – 2		20	81
cholic acid	pharmaceuticals	ISE, wall jet				82
dopamine	injections	periodate ISE	8 – 270		200	83
fluoride	pharmaceuticals	pervaporation	1.5 – 200			84
iron	vitamin formulations	ISE	0.032 – 10 mM	0.025 mM		85
penicillin	solution	penicillinase	1 – 100 mM		45	86
penicillins	pharmaceuticals	enzyme electrode	0.001 – 1 mM	20 µM		87
penicillin V, G	pharmaceuticals	biosensor	1000 – 10000 units/ml			88
pipazethate	pharmaceuticals	membrane electrode				89
promethazine	pharmaceuticals	ISE	0.5 µM – 0.1 mM	0.2 µM		90
urea	ointments	pH-enzyme electrode	1 – 13 mM			91
urea	pharmaceuticals	biosensor	1 – 15 mM			88

Table 7: FIA assays with voltammetric detection

drug	matrix	reagents/technique	linear range (mg/l)	detection limit (mg/l)	ST (h^{-1})	ref.
codeine	pharmaceuticals human plasma	chemically-modified electrode, square-wave voltammetry	0 – 32 µM	10 nM		92
lipase	pharmaceuticals	reduction of vitamin K_3	10 – 1500 units/l			93
nifedipine	pharmaceuticals	polarography	0.05 – 0.5 mM	0.015 mM	120	94

Table 8: FIA assays with gravimetric detection

drug	matrix	reagents/ technique	linear range (mg/l)	detection limit (mg/l)	ST (h^{-1})	ref.
adrenaline **dopa**	pharmaceuticals	piezoelectric detection	4 – 850 3.5 – 730	1.22 1.05	120	95
lignocaine	pharmaceuticals	piezoelectric detection, dodecyl-phenylsulfonate	0.01 – 2	8	120	96
noradrenaline	pharmaceuticals	piezoelectric detection	0.01 – 1.2			97
phenobarbiturate	pharmaceuticals	piezoelectric detection	up to 1 mg/ml	0.44		98
procaine	pharmaceuticals	piezoelectric detection, dodecyl-phenylsulfonate	0.02 – 2 mg/ml	0.01 mg/ml	120	99

ST – sample throughput

3. Conclusion

Flow injection analysis is a versatile tool that has contributed substantially to the development of automation in pharmaceutical analysis. The number of chemical drugs being determined by FIA is ever increasing and this technique together with sequential injection analysis (SIA) proved to be an invaluable means for carrying out automated routine analyses in pharmaceutical quality control and research laboratories.

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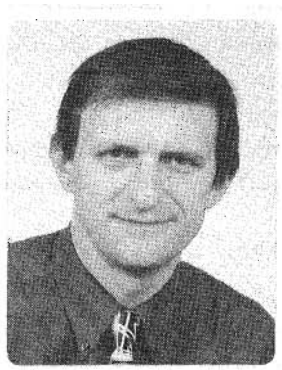

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