

The development and validation of HPLC
methods for the detection of drug and detergent
traces on laboratory glassware in a
pharmaceutical laboratory

P M Mothobi

The development and validation of HPLC methods for the detection of drug and detergent traces on laboratory glassware in a pharmaceutical laboratory

Pride Mmakeletso Mothobi

(B.Sc. (Hons) Env.Sci)

Dissertation submitted in fulfillment of the requirements for the degree ***Magister Scientiae (Pharmaceutics)*** at the Potchefstroom Campus of the North-West University.

Supervisor: Dr J.C Wessels

Co-Supervisor: Dr M.E Aucamp

Potchefstroom

2012

"The LORD is my shepherd, I shall not want"

Psalm 23:1

ACKNOWLEDGEMENTS

I would like to express my sincere appreciation to the following people and institutions for their contributions in enabling me to complete this study:

To the following people at the **RIIP[®]/CENQAM[®]**, North-West University (Potchefstroom Campus)

Dr. Erna Swanepoel, for allowing me to conduct this study. For authorising the use of the **RIIP[®]/CENQAM[®]** resources whenever needed. Know that I am greatfull for this opportunity wherever I am.

Dr. Marique Aucamp, for her invaluable assistance, patience and guidance personally and in all aspects of this study. "You took it by the horns";

Dr. Anita Wessels, for her technical solid approach and assistance in ensuring completion of this study;

Portia, Selina and the late **Mpho**, for their analytical assistance;

Tanie Elsa, Ouma (Rianda) and Tanie Zetta, for their words of encouragement;

To the following persons of the School for Environmental Sciences and Development, Microbiology, North-West University (Potchefstroom Campus)

Dr. Retha van der Walt, for her unconditional love, friendship, and for believing in me;

Prof. Carlos Bezuidenhout, for the solid scientific foundation he laid in my undergraduate and post graduate studies.

To my father (**Khomotso**) and my two brothers (**Itumeleng and Onthatile**), thank you for your love and support always.

TABLE OF CONTENTS

ABSTRACT	vi
UITTREKSEL.....	viii
LIST OF FIGURES.....	x
LIST OF TABLES	xv
ABBREVIATIONS.....	xviii
AIM AND OBJECTIVES	xix
CHAPTER 1: INTRODUCTION.....	1
1.1 GENERAL INTRODUCTION.....	1
1.2 HYPOTHESIS	3
CHAPTER 2: LITERATURE REVIEW	5
2.1 OVERVIEW OF THE STUDY LABORATORY.....	5
2.2 CHROMATOGRAPHY	7
2.3 HPLC METHOD DEVELOPMENT	8
2.4 PROCEDURE FOR DEVELOPING AN HPLC METHOD.....	9
2.4.1 Analytes/compound of interest	9
2.4.2 Selection of the chromatographic mode	11
2.4.3 Selection of the mobile phase	14
2.4.4 Choosing a column	15
2.4.5 Isocratic vs. Gradient analysis	16
2.4.6 Choosing an HPLC detector	17

2.5	METHOD VALIDATION.....	19
2.6	CONCLUSION.....	23
CHAPTER 3:	EQUIPMENT, MATERIALS AND METHODS	24
3.1	INTRODUCTION	24
3.2	GLASSWARE CLEANING PROCEDURES	24
3.2.1	Automated glassware cleaning procedure	24
3.2.1.1	Cleaning procedure	25
3.2.1.2	Possible drawbacks of the current in-house automated glassware cleaning procedure.....	25
3.2.2	Manual glassware cleaning procedure	26
3.2.2.1	Detergents and non-corrosive cleaning agents	26
3.2.2.2	Pre-rinsing/ prewashing of glassware	28
3.2.2.3	General manual cleaning/ washing of glassware	28
3.2.2.4	Drying glassware	28
3.2.2.5	Visual inspection	28
3.2.2.6	Possible drawbacks of the current in-house manual glassware cleaning procedure	28
3.3	PHYSICAL PROPERTIES	29
3.3.1	Colour, solubility & state of matter observations	29
3.3.2	Density analysis	30
3.4	UV-SPECTROPHOTOMETRIC ANALYSIS	30

3.5	HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC) ANALYSIS	31
3.5.1	Instrument used for method development	31
3.5.2	HPLC method chromatographic conditions	32
3.5.3	Preparation of standard solutions	33
3.5.4	Preparation of sample solutions	35
3.5.5	Conclusion	37
CHAPTER 4:	RESULTS AND DISCUSSION	38
4.1	DENSITY ANALYSES	38
4.2	SPECTROPHOTOMETRIC ANALYSES	39
4.2.1	Discussion and conclusion of spectrophotometric analyses	41
4.3	HPLC METHOD DEVELOPMENT ANALYSES	42
4.3.1	Analyte	42
4.3.2	Chromatographic mode and column choice	42
4.3.3	Mobile phase selection	43
4.3.4	Conclusion	53
4.4	INVESTIGATION OF THE EFFICIENCY OF THE CURRENT IN-HOUSE GLASSWARE CLEANING PROTOCOLS	53
4.4.1	Chromatographic conditions	53
4.4.2	Results of the developed HPLC method operational limits	54
4.4.3	HPLC method operational limit results and discussion	60
4.3.3.1	System suitability	60

4.3.3.2	HPLC method limits	62
4.3.3.3	Conclusion	64
4.4.4	Cleaning samples	64
4.4.4.1	Rinse vs. swabbing procedures	64
4.4.4.1.1	Conclusion	68
4.4.4.2	Evaluation of the current in-house cleaning procedure results	68
4.4.4.3	Calculation of the detergent contaminants percentage recovery	74
4.4.4.4	Discussion and conclusion	75
4.5	SUMMARY AND CONCLUSION	75
CHAPTER 5: CLEANING VALIDATION USING HPLC FOR ANALYSIS		77
5.1	INTRODUCTION	77
5.2	VALIDATION	77
5.2.1	Scope	78
5.2.2	Chromatographic conditions	78
5.2.3	Standard preparation	79
5.2.4	Results and discussion	84
5.2.4.1	Validation test procedure and acceptance criteria	84
5.3	SUMMARY AND CONCLUSION	101
5.4	RESEARCH RECOMMENDATIONS	101

REFERENCES.....	103
-----------------	-----

ABSTRACT

Pharmaceutical contract testing laboratories carry a responsibility to ensure that medicine made available for consumption by patients is of the approved quality for their intended health use. Glassware is an essential tool in testing of pharmaceutical products. Glassware used in most pharmaceutical contract testing laboratories is non-dedicated hence proper glassware cleaning procedures are essential. Contract testing laboratories need to perform glassware cleaning validation studies to verify that glassware used in the testing of medicines is adequately cleaned from one product to the next and to ensure that the cleaning procedures themselves do not contribute any unwanted residues to the glassware.

The aim of this study was to develop and validate HPLC methods for the detection of drug and detergent residues recovered from glassware in a pharmaceutical contract testing laboratory. The objectives of the study were to:

- i. Develop and validate an HPLC method to detect selected glassware cleaning detergents;
- ii. Investigate the efficacy of the current in-house glassware cleaning protocol (manual and automatic cleaning);
- iii. Investigate the efficacy of cleaning detergents on glassware exposed to drugs;
- iv. Develop an efficient glassware cleaning protocol;
- v. Validate a glassware cleaning protocol for a pharmaceutical laboratory.

Cleaned laboratory volumetric flasks of varying sizes were randomly used as samples. Glassware washed with the automatic laboratory glass-washer and manually washed glassware was subjected to the rinsing and swabbing sampling procedures. A standard addition and recovery procedure was also employed to prove that the cleaning procedure works and that the glassware is indeed clean after being hand washed or automatically washed with the glassware washer.

The HPLC method was validated on an LC Agilent® 1100 DAD series system using a µBondapak C₁₈ (300 mm x 3.9 mm, 10 µm). Acetonitrile: buffer containing 0.02 M hexanesulfonic acid sodium salt with the pH adjusted to 3.0 with phosphoric acid in the ratio 25:75 was used as mobile phase with the flow rate set at 1.0ml/min. UV detection set at 220 nm and the injection volume at 25 µl.

The regression line plot obtained was linear over a concentration range from 5000 µg/ml to 15 000 µg/ml for Ekon D concentrate® and a concentration range from 9700 µg/ml to 39 000 µg/ml for LaboClean FT concentrate®. The correlation coefficient of 0.993 was obtained for Ekon D concentrate® and 0.999 for LaboClean FT concentrate®. The detection limit and quantitation limit were 1568 µg/ml and 5228 µg/ml for Ekon D concentrate®, and 917 µg/ml and 3059 µg/ml for LaboClean FT concentrate®. The relative standard deviation (%RSD) obtained for both detergents were below 7.0%. The mean recovery of the method was 99.5%.

In the results obtained detergent traces were recovered from approximately 16% of the total sampled hand washed glassware and in 13% of the hand washed glassware, drug contaminants were also recovered. From the machine washed sampled glassware 10% was contaminated with drug residues and none of the sampled machine washed glassware flasks were contaminated with soap residues.

The HPLC method developed for the detection of detergent and drug traces recovered from laboratory glassware was a success. The automated glassware cleaning procedure was more efficient in the cleaning of laboratory glassware when compared to the manual cleaning procedure. Observation shows that the current in-house glassware cleaning protocol is efficient; however, the SOP is not followed properly. The developed HPLC method was proved to meet all the performance expectations and acceptance criteria for cleaning validation purposes. The aim of this study to develop and validate the HPLC method for the detection of drug and detergent traces recovered from laboratory glassware for a pharmaceutical contract testing laboratory was met.

UITTREKSEL

Farmaseutiese toetslaboratoriums dra 'n verantwoordelikheid deur toe te sien dat die medisyne wat aan pasiënte beskikbaar gestel word vir 'n bepaalde siektetoestand van aanvaarbare kwaliteit is. Glasware is onontbeerlike toerusting tydens die toets van farmaseutiese produkte. In die meeste laboratoriums word glasware nie toegewys aan 'n bepaalde analitiese proses nie en dit is dus noodsaaklik dat die skoonmaakproses van hoogstaande gehalte is. Dit is dus nodig dat kontraklaboratoriums die skoonmaakprosesse wat gebruik om glasware mee te was sal valideer om sodoende te verseker dat die produk waarvoor dit gebruik was, behoorlik verwyder is. Verder is dit ook noodsaaklik dat vasgestel word dat die skoonmaakmiddels nie bydra tot onnodige residue op die glas nie.

Die doelstellings vir hierdie studie was om Hoë Druk Vloeistof Chromatografie (HPLC) metodes te ontwikkel en te valideer waarmee geneesmiddel- en skoonmaakmiddelresidue wat moontlik in glasware agtergelaat kon word, te kan analiseer. Die doelwitte van die studie was dus om:

- i. 'n HPLC metode te ontwikkel en te valideer waarmee sekere skoonmaakmiddels op glasware geanaliseer kon word;
- ii. Die effektiwiteit van die skoonmaakproses in die kontraklaboratorium (handwas en outomaties) vas te stel;
- iii. Die effektiwiteit van die skoonmaakmiddels om geneesmiddels van glasware te verwyder;
- iv. 'n Effektiewe skoonmaakproses te ontwikkel indien nodig, en
- v. Die validering van die skoonmaak protokol.

Skoon volumetriese flesse in verskillende groottes is ewekansig gekies vir die toetse. Beide glasware wat met die hand gewas is en wat met die outomatiese wasser gewas is, is met gedistilleerde water gespoel en/of smere met katoenstokkies is geneem, om monsters vir analyses te bekom. 'n Standaard herwinningsprosedure is ook gebruik om te verseker dat die glasware inderdaad skoon was nadat dit gewas is.

Die HPLC metode is op 'n LC Agilent® 1100 DAD sisteem ontwikkel en valideer. 'n µBondapak C₁₈ (300 mm x 3.9 mm, 10 µm) kolom is gebruik. Asetonitriël en 0.02M heksaansulfoonsuur buffer met 'n pH van 3.0 (aangepas met fosforsuur) in 'n verhouding 25:75 is as mobiele fase gebruik. Die vloeisnelheid was 1.0ml/min en UV deteksie by 220nm is gebruik. Die inspuitvolume was 25 µl.

Tydens die validasie van die analitiese metode vir die skoonmaakmiddels is die volgende resultate verkry. Vir Ekon D concentrate® is 'n lyn in die konsentrasiegebied 5000 µg/ml tot 15 000 µg/ml opgestel en vir LaboClean FT concentrate® 9700 µg/ml tot 39 000 µg/ml. Die korrelasie koëffisiënt vir Ekon D concentrate® was 0.993 en vir LaboClean FT concentrate® was dit 0.999. Die detekselimiete was onderskeidelik 1568 µg/ml en 917 µg/ml en die kwanitifiseringslimiete was onderskeidelik 5225 µg/ml en 3059 µg/ml. The persentasie relatiewe standaard afwyking (%RSD) vir beide middels was laer as 7.0%. Die gemiddelde herwinning van die metode was 99.5%.

Die resultate wat verkry is dui daarop dat ongeveer 16% van die totale aantal monsters vir glasware wat met die hand gewas is, steeds seep residue bevat het en 13% daarvan steeds geneesmiddel residue bevat het. Vir glasware wat met die wasser gewas is, was daar 10% wat steeds geneesmiddel residue bevat het en geen daarvan het seep residue bevat nie.

Die HPLC metode wat vir die studie ontwikkel is kon suksesvol gebruik word om die analyses van die monsters te doen. Die metode is suksesvol gevalideer en dit het aan al die kriteria voldoen. Die outomatiese wasser is meer effektief om glasware skoon te was. Tydens oëbeërsing is vasgestel dat die handwas metode ook meer effektief sal wees indien die voorgestelde prosedure noukeuriger gevolg word. Die doelwitte van hierdie studie is dus suksesvol behaal.

LIST OF FIGURES

Figure 2.1	Schematic representation to selecting HPLC chromatographic mode	11
Figure 3.1	Schematic representation summary of the automated glassware cleaning procedure	26
Figure 3.2	Schematic representation summary of the manual glassware cleaning procedure	27
Figure 4.1	UV spectrum obtained for Ekon D concentrate® at concentration 2 mg/ml	39
Figure 4.2	UV spectrum obtained for LaboClean FT concentrate® at concentration 26 mg/ml	40
Figure 4.3	UV spectrum obtained for Contrad concentrate® at concentration 2 mg/ml	41
Figure 4.4	Chromatogram obtained with Ekon D concentrate® at a concentration of 5% v/v using mobile phase 1, with chromatograms (a) and (b) obtained at wavelengths 205 nm and 220 nm respectively	43
Figure 4.5	Chromatogram obtained with Laboclean FT. concentrate® at a concentration of 5% v/v using mobile phase 1, with chromatograms (a) and (b) obtained at wavelengths 205 nm and 220 nm respectively ...	44
Figure 4.6	Chromatogram obtained with Contrad concentrate® at a concentration of 5% v/v using mobile phase 1, with chromatograms (a) and (b) obtained at wavelengths 205 nm and 220 nm respectively	45
Figure 4.7	Chromatogram obtained with Ekon D concentrate® at a concentration of 5% v/v using mobile phase 2, with chromatograms (a) and (b) obtained at wavelengths 205 nm and 220 nm respectively	46

Figure 4.8	Chromatogram obtained with LaboClean FT concentrate [®] at a concentration of 5% v/v using mobile phase 2, with chromatograms (a) and (b) obtained at wavelengths 205 nm and 220 nm respectively ...	47
Figure 4.9	Chromatogram obtained with Contrad concentrate [®] at a concentration of 5% v/v using mobile phase 2, with chromatograms (a) and (b) obtained at wavelengths 205 nm and 220 nm respectively ...	47
Figure 4.10	Chromatogram obtained with Ekon D concentrate [®] at a concentration of 5% v/v using mobile phase 3, with chromatograms (a) and (b) obtained at wavelengths 205 nm and 220 nm respectively	48
Figure 4.11	Chromatogram obtained with Laboclean FT. concentrate [®] at a concentration of 5% v/v using mobile phase 3, with chromatograms (a) and (b) obtained at wavelengths 205 nm and 220 nm respectively	49
Figure 4.12	Chromatogram obtained with Contrad concentrate [®] at a concentration of 5% v/v using mobile phase 3, with chromatograms (a) and (b) obtained at wavelengths 205 nm and 220 nm respectively	49
Figure 4.13	Chromatogram obtained with Ekon D concentrate [®] at a concentration of 1% v/v using mobile phase 4, with chromatograms (a) and (b) obtained at wavelengths 205 nm and 220 nm respectively	51
Figure 4.14	Chromatogram obtained with LaboClean FT. concentrate [®] at a concentration of 1% v/v using mobile phase 4, with chromatograms (a) and (b) obtained at wavelengths 205 nm and 220 nm respectively ...	51
Figure 4.15	Chromatogram obtained with Contrad concentrate [®] at a concentration of 1% v/v using mobile phase 4, with chromatograms (a), (b) and (c) obtained at wavelengths 205 nm, 212 nm and 220 nm respectively	52
Figure 4.16	Linear plot obtained for Ekon D concentrate [®] peak 1 area response for the determination of the developed HPLC method operation limits	55

Figure 4.17	Linear plot obtained for Ekon D concentrate [®] peak 2 area response for the determination of the developed HPLC method operation limits	55
Figure 4.18	Linear plot obtained for LaboClean FT. concentrate [®] peak area response for the determination of the developed HPLC method operation limits ...	56
Figure 4.19	Typical chromatogram obtained for Ekon D concentrate [®] at a concentration of 10 mg/ml with the wavelength set at 220 nm	63
Figure 4.20	Typical chromatogram obtained for LaboClean FT concentrate [®] at a concentration of 13 mg/ml, with chromatograms (a) and (b) obtained at wavelengths 205 nm and 220 nm respectively	63
Figure 4.21	Chromatogram obtained from a swab sample of an API Standard volumetric flask, detected at wavelength 220 nm	65
Figure 4.22	Chromatogram obtained from a swab of an API Sample volumetric flask, detected at wavelength 220nm	66
Figure 4.23	Chromatogram obtained from a sterile swab solution, detected at wavelength 220nm	67
Figure 4.24	Chromatogram obtained from the API standard volumetric flask rinsate, detected at wavelength 220nm	67
Figure 4.25	Chromatogram obtained from the API sample volumetric flask rinsate, detected at wavelength 220nm	68
Figure 4.26	A typical chromatogram obtained from injecting the solvent at a wavelength of 220 nm	69
Figure 4.27	A typical chromatogram obtained from the hand washed glassware rinsate, with the wavelength set at 220 nm	70
Figure 4.28	A typical chromatogram obtained from the machine washed glassware rinsate, with the wavelength set at 220 nm	70

Figure 4.29	Depiction of the solvent associated peak in Ekon D concentrate [®] standard	71
Figure 4.30	Magnification of the solvent associated peak identified in Figures 4.27, 4.28, and 4.29	71
Figure 4.31	A typical chromatogram obtained from the API contaminated volumetric flask rinsate, detected at wavelength 220 nm	72
Figure 4.32	A typical chromatogram obtained from the API contaminated volumetric flask rinsate, detected at wavelength 220 nm	73
Figure 4.33	A typical chromatogram obtained from a detergent contaminated volumetric flask rinsate, detected at wavelength 220 nm	73
Figure 5.1	Linear plot obtained for Ekon D concentrate [®] peak 1 for HPLC method validation, conducted by the analyst developing the HPLC method	86
Figure 5.2	Linear plot obtained for Ekon D concentrate [®] peak 2 for HPLC method validation, conducted by the analyst developing the HPLC method	86
Figure 5.3	Linear plot obtained for the LaboClean FT concentrate [®] peak for HPLC method validation, conducted by the analyst developing the HPLC method	87
Figure 5.4	Chromatogram obtained for LaboClean FT concentrate [®] with the mobile phase buffer adjusted by 50%	89
Figure 5.5	Chromatogram obtained for Ekon D concentrate with mobile phase buffer adjusted by 50%	89
Figure 5.6	Chromatogram obtained for Ekon D concentrate [®] after adjusting the mobile phase pH to 2.5	90

Figure 5.7	Chromatogram obtained for LaboClean FT concentrate [®] after adjusting the mobile phase pH to 2.5	90
Figure 5.8	Chromatogram obtained for Ekon D concentrate [®] when using Luna C ₁₈ 250 x 4.6 mm, (5 µm) column	91
Figure 5.9	Chromatogram obtained for LaboClean FT concentrate [®] when using Luna C ₁₈ 250 x 4.6mm, (5 µm) column	91

LIST OF TABLES

Table 2.1	Properties of analyte to guide method development.....	9
Table 2.2	Practical analytical steps for cleaning validation	22
Table 3.1	Chromatographic conditions for the HPLC.....	32
Table 3.2	System suitability conditions	33
Table 3.3	Standard preparation of Ekon D concentrate®	34
Table 3.4	Standard preparation of LaboClean FT concentrate®	34
Table 3.5	Standard preparation of Contrad concentrate®	35
Table 4.1	Density results of Ekon D®, Laboclean FT® and Contrad concentrate® ...	38
Table 4.2	Standard preparation for the determination of the developed HPLC method operational limits	54
Table 4.3	Ekon D concentrate® peak 1 area response summary report obtained for the determination of operational limits of the developed HPLC method	57
Table 4.4	Ekon D concentrate® peak 2 area response summary report obtained for the determination of operational limits of the developed HPLC method	58
Table 4.5	LaboClean FT concentrate® peak area response summary report obtained for the determination of operational limits of the developed HPLC method	59
Table 4.6	Percentage recovery of detergent contaminants	74
Table 5.1	Chromatographic conditions for the HPLC system	79

Table 5.2	Standard preparation of Ekon D concentrate®	80
Table 5.3	Standard preparation of LaboClean FT concentrate®	81
Table 5.4	Ekon D concentrate® peak 1 area response summary report obtained for the validation of the developed HPLC method, conducted by the analyst developing the HPLC method	82
Table 5.5	Ekon D concentrate® peak 2 area response summary report obtained for the validation of the developed HPLC method, conducted by the analyst developing the HPLC method	83
Table 5.6	LaboClean FT concentrate® peak area response summary report obtained for the validation of the developed HPLC method, conducted by the analyst developing the HPLC method	84
Table 5.7	Ekon D concentrate® peak 1 area response summary report obtained for method transfer of the developed HPLC method, conducted by a post graduate student using a Shimadzu® UFLC system	93
Table 5.8	Ekon D concentrate® peak 2 area response summary report obtained for method transfer of the developed HPLC method, conducted by a post graduate student using a Shimadzu® UFLC system	94
Table 5.9	LaboClean FT concentrate® peak area response summary report obtained for method transfer of the developed HPLC method, conducted by a post graduate student using a Shimadzu® UFLC system	95
Table 5.10	Ekon D concentrate® peak 1 area response summary report obtained for method validation of the developed HPLC method, conducted by an inexperienced analyst using a Shimadzu® UFLC system	96

Table 5.11	Ekon D concentrate [®] peak 2 area response summary report obtained for method validation of the developed HPLC method, conducted by an inexperienced analyst using a Shimadzu [®] UFLC system	97
Table 5.12	LaboClean FT concentrate [®] peak area response summary report obtained for method validation of the developed HPLC method, conducted by an inexperienced analyst using a Shimadzu [®] UFLC system	98

ABBREVIATIONS

API	Active pharmaceutical ingredient
cGMP	Current good manufacturing practices
FDA	The Food and Drug Administration, USA
GLP	Good laboratory practices
GMP	Good manufacturing practices
HPLC	High performance liquid chromatography
HSA	Hexane sulphonic acid
LC	Liquid chromatography
MS	Mass spectrometry
LOD	Limit of detection
LOQ	Limit of quantitation
PDA	Photodiode array
% RSD	Percentage relative standard deviation
SOP	Standard operating procedure
UFLC	Ultra fast liquid chromatography
UPLC	Ultra pressure liquid chromatography
USP	United States Pharmacopoeia
UV/Vis	Ultraviolet/visible

AIM AND OBJECTIVES

AIM

The aim of the study was to develop and validate HPLC methods for the detection of drug and detergent traces recovered from laboratory glassware in a pharmaceutical contract testing laboratory.

BACKGROUND

Pharmaceutical contract testing laboratories carry a responsibility to ensure that components, containers and closures, in-process materials, and finished drug products conform to the established standards and specifications (FDA, 2006). Data generated by contract testing laboratory is expected to be meaningful, thorough, timely, unbiased, well documented, and scientifically sound as it ensures quality control and compliance by pharmaceutical manufacturing companies (Ahuja, 2005).

Volumetric glassware forms part of nearly all critical analytical experiments in contract testing laboratories. Contamination of glassware may therefore compromise the reliability of the generated data. Unreliable data violates cGMP regulations that clearly mandate the reliability and accuracy of data generated in contract testing laboratories.

Limited information is available regarding the chemical composition of laboratory detergents. This issue is caused by competition amongst the detergent suppliers and the need to protect detergent formulations from being copied. The detergents efficacy on glassware exposed to drugs is a matter based on the user's discretion in cases where user instructions are not specified by the supplier.

Research is not clear in describing protocols relating to laboratory glassware care and maintenance in pharmaceutical environments. This puts contract testing laboratories at a risk of using laboratory glassware that might not be properly cleaned for generating valuable data.

OBJECTIVES

To achieve the aim of the study the following objectives were pursued:

1. Develop and validate an HPLC method to detect selected glassware cleaning detergents;
2. Investigate the efficacy of the current in-house glassware cleaning protocol;
3. Investigate the efficacy of cleaning detergents on glassware exposed to drugs;
4. Develop an efficient glassware cleaning protocol;
5. Validate a glassware cleaning protocol for a pharmaceutical laboratory.