

DECLARATION BY THE CANDIDATE (Para 12B)

I declare that the thesis entitled “**Sample preparation and analysis of environmental markers of chemical warfare agents by means of tandem mass spectrometry**” is my own work conducted under the supervision of **Dr. D. K. Dubey**, Head, VERTOX Laboratory, Defence Research and Development Establishment, Gwalior and **Prof. (Dr.) Rajeev Jain**, Professor, School of Studies in Chemistry, Jiwaji University, Gwalior, at the VERTOX Laboratory, Defence Research and Development Establishment, Gwalior, approved by the Research Degree Committee. I have put-in more than 200 days of attendance with the supervisors at the research centre.

I, further declare that to the best of my knowledge, the thesis does not contain any part of any work, which has been submitted for the award of any degree either in this University or in any other University/Deemed University without proper citation.

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CERTIFICATE

This is to certify that the work entitled “**Sample preparation and analysis of environmental markers of chemical warfare agents by means of tandem mass spectrometry**” is a piece of research work done by **Mr. Vijay Kumar Tak**, under our guidance and supervision for the degree of Doctor of Philosophy of **Jiwaji University, Gwalior, M.P., India** and that the candidate has put-in attendance of more than 200 days with us.

To the best of my knowledge and belief the thesis:

- I. Embodies the work of the candidate himself.
- II. Has duly been completed.
- III. Fulfils the requirements of the Ordinance relating to the Ph. D. degree of the University, and
- IV. Is up to the standard both in respect of contents and language for being referred to the examiner.

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This is to certify that the work embodied in this thesis entitled "SAMPLE PREPARATION AND ANALYSIS OF ENVIRONMENTAL MARKERS OF CHEMICAL WARFARE AGENTS BY MEANS OF TANDEM MASS SPECTROMETRY" has been carried out by Mr. Vijay Kumar Tak, at VERTOX Laboratory, Defence Research & Development Establishment, Gwalior, by himself.

DIRECTOR

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List of abbreviations

AED	Atomic emission detector
APCI	Atmospheric pressure chemical ionization
AAPAs	Alkyl alkylphosphonic acids
APAs	Alkylphosphonic acids
ANOVA	Analysis of variance
CWAs	Chemical Warfare Agents
CWs	Chemical Weapons
CWC	Chemical Weapons Convention
CRCs	Convention Related Chemicals
CE	Capillary electrophoresis
CI	Chemical ionization
CID	Collision induced dissociation
DLLME	Dispersive liquid-liquid microextraction
DEHP	Di-(2-ethylhexyl) phosphate
DiPrAE	<i>N,N</i> -diisopropylaminoethanol
DBAE	<i>N,N</i> -dibutylaminoethanol
DHE	Dihexyl ether
DAAPs	<i>O,O'</i> -dialkyl alkylphosphonates
DPrMP	Dipropyl methylphosphonate
DEiPrP	Diethyl isopropylphosphonate
DMMP	Dimethyl methylphosphonate
DEEP	Diethyl ethylphosphonate
DBMP	Dibutyl methylphosphonate
DBEP	Dibutyl ethylphosphonate
DHMP	Dihexyl methylphosphonate
DOMP	Dioctyl methylphosphonate
DDMP	Didecyl methylphosphonate
EI	Electron ionization
ESI	Electrospray ionization
EPA	Ethylphosphonic acid
EMPA	Ethyl methylphosphonic acid
EIC	Extracted ion chromatogram

EF	Enrichment factor
EDEA	<i>N</i> -ethyl-diethanolamine
ENB	1-Ethyl-2-nitobenzene
FID	Flame ionization detector
FTIR	Fourier transform infra-red
EME	Electromembrane extraction
GC	Gas chromatography
GC-MS	Gas chromatography-mass spectrometry
HF-LPME	Hollow fiber liquid phase microextraction
HFLLLME	Hollow fiber liquid-liquid-liquid microextraction
HepMPA	<i>n</i> -Heptyl methylphosphonic acid
HMPA	<i>n</i> -Hexyl methylphosphonic acid
IT	Ion trap
IBHBI	1,3-imidazolium-bis-(1-hexyl-benzyl-imidazolium) trifluoride
iPrMPA	Isopropyl methylphosphonic acid
iPrOMP	Isopropyloctyl methylphosphonate
iBMPA	Isobutyl methylphosphonic acid
LLE	Liquid-liquid extraction
LPME	Liquid phase microextraction
LC	Liquid chromatography
LC-MS	Liquid chromatography-mass spectrometry
LC-MS/MS	Liquid chromatography-tandem mass spectrometry
LOD	Limit of detection
LOQ	Limit of quantification
MS	Mass spectrometry
MS/MS	Tandem mass spectrometry
MPA	Methylphosphonic acid
MMPA	Methyl methylphosphonic acid
MDEA	<i>N</i> -methyldiethanolamine
NPD	Nitrogen-phosphorous detector
NMR	Nuclear magnetic resonance
NBMI	1,9-Nonanediyl-bis-(3-methylimidazolium)bisfluoride
NPOE	2-Nitrophenyl octyl ether

NPPE	2-Nitrophenyl pentyl ether
OPCW	Organization for the Prohibition of Chemical Weapons
OPT	Official Proficiency Test
OCAD	OPCW Central Analytical Database
PMPA	<i>n</i> -Pentyl methylphosphonic acid
PiPrPA	<i>n</i> -Pentyl iso-propylphosphonic acid
PinMPA	Pinacolyl methylphosphonic acid
QTOF	Quadrupole time-of-flight
QqQ	Triple quadrupole
ROP	Recommended Operating Procedures
RI	Retention indices
RSD	Relative standard deviation
RSM	Response surface model
SPE	Solid phase extraction
SPME	Solid phase microextraction
SDME	Single drop microextraction
SLM	Supported liquid membrane
TEA	Triethanolamine
TEHP	Tris-(2-ethylhexyl) phosphate
TIC	Total ion chromatogram
UHPLC-QTOF	Ultra-high performance liquid chromatography- quadrupole time-of-flight
WWI	World War I
WWII	World War II
μ-EME	Microfluidic electromembrane extraction