REVIEW

of dissertation for the acquisition of:

educational and scientific degree " **doctor** "	X
scientific degree **"Doctor of Science"**	
	the true is indicated by the sign "X"

Author of the dissertation:

		Milazim		**Tahirukaj**	Kosovo Forensic Science Agency, Pristina Laboratory
academic position	scientific degree	name	middle name	last name	workplace

Topic of the dissertation:

DEVELOPMENT AND VALIDATION OF SEM/EDAX METHOD FOR ANALYSIS OF GUNSHOT RESIDUES

Scientific area:

4	Natural Science
code	name

Professional area:

4.2	Chemical Sciences
code	name

Scientific specialty:

The review was written by:

Assoc.Prof.	Dr.	Stela	Ivanova	Georgieva-Kiskinova	UCTM
academic position	scientific degree	name	middle name	last name	workplace

1. Completion of the provided documents:

A) The dissertation and the competition documents are in full compliance with the Regulations.	4 points	**X**
B) The documents are complete but do not fully comply with the requirements of the Regulations.	2 points	

C) The documents are not completed in accordance with the requirements of the Regulations.	0 points	
		one of the answers given is marked with the sign "X"

Missing documents and violated standards must be described if response C is marked.

2. Meeting the minimum requirements under the Regulations:

A) The candidate meets the minimum requirements	20 points	X
B) The candidate doesn't meet the minimum requirements	0 points	
		one of the answers given is marked with the sign "X"

It must be filled in if answer B is marked. The publication activity of the candidate is analyzed. The response of the results achieved (quoted) is analyzed.

Milazim Tahirukaj's results are summarized in two scientific publications in journals with high IF (Scopus database). I dare to say, that the results presented in Milazim Tahirukaj's doctoral thesis are fully reflected in both publications. The first publication presents: 1) the results from a study of the effect of SEM/EDS working parameters on the method performance and quality of GSR data, as well as a development of a database of iGSR encountered in Republic of Kosovo; 2) The optimal operating parameters of the SEM/EDS established by a one-variable-at-a-time approach and the validation of the method according to ASTM1588-10; 3) analytical characteristics of the method such as precision, trueness and expanded uncertainty for PbBaSb particles were evaluated. Evidence is presented for the applicability of the validated SEM/EDS method to identify characteristic iGSR particles in Kosovo gunfire samples. The manuscript also demonstrates that the method can provide legal proof of the existence of iGSR on a specific surface. Despite the short period since the publication of the article, two citations from foreign scientists of the presented data can be found, which shows interest in the reported topic. In the second article, a summary assessment of the persistence of firearms typically used in criminal cases in the Republic of Kosovo is made. Results are presented describing the number of particles identified by SEM/EDS as a function of time, gun caliber, number of produced shots, and weather conditions as firearms commonly used in criminal cases in Kosovo were investigated, as the experiments were conducted outdoors in two annual seasons. The applied SEM/EDS method was optimized and a validated protocol with analytical characteristics was obtained: sensitivity of 95%, bias of -5%, repeatability of 2% (RSD), intra-laboratory reproducibility of 2% (RSD), and expanded uncertainty of the number of GSR particles of 6% at a coverage factor of $k = 2$. The scientific publication presented in this way does not yet have any citations noticed, but I attribute this to its recent publication and in no way diminishes the scientific resonance of the results. The doctoral student has also participated in 2 international scientific forums, where he has

disseminated and presented his results to a wide scientific audience. The two publications on the subject of the dissertation work with quartile Q2 (Scopus database), the 2 participations in scientific forums, and the successfully passed exams of the doctoral minimum show that Milazim Tahirukaj meets, even exceeds, the minimum requirements, according to the Regulations of the UCTM, Sofia, in both: educational and scientific part.

3. The relevance of the topic of the dissertation:

A) The topic is relevant and new (there are no known results on the topic by other authors)	8 points	
B) The topic is relevant and results from other authors are known	6 points	X
C) The topic is not relevant, but results from other authors are known	2 points	
D) The topic is not relevant and no results from other authors are known	1 point	
E) The topic does not correspond to the level of dissertation	0 points	
		one of the answers given is marked with the sign "X"

The evaluation of the relevance of the dissertation must be substantiated
Fundamental research in criminal science, and in particular, the study of gunshot residue, has made a crucial contribution to the application of investigative evidence by judicial authorities in many countries. The application of scanning electron microscopy (SEM/EDS) to image acquisition and analysis of crime materials including gunshot residue (GSR), paint and glass analysis, banknotes and coins, counterfeits, hair and fiber comparison, as well as forensic toxicology is an up-to-date technique for analysis, and undoubtedly any improvement in the operating parameters of SEM/EDS, as well as validation of the used analysis protocol, would lead to an expansion of the possibilities for interpreting results from SEM/EDS measurements. The results obtained would benefit forensic scientists by providing a helpful tool, for example, in hypothesizing the time interval between the firing of a firearm and the sampling of GSR, making the problem traditionally actual. By fulfilling the goals the creation of fundamental prerequisites for the search for effective solutions to social challenges directly related to the detection of human crimes will be supported.

4. Knowledge of the problems, subject of research in the dissertation:

A) The doctoral student knows in detail the achievements of other authors on the topic of the dissertation	8 points	X
B) The doctoral student is partially familiar with the achieved results on the topic of the dissertation	4 points	
C) The doctoral student has no prior knowledge of the status of the problems in the dissertation	0 points	

		one of the answers given is marked with the sign "X"

The evaluation must be substantiated if answer C is marked.
The literature review is extensive (a total of 107 literature sources are cited) and demonstrates the student's well-informed, in-depth knowledge of the subject and his ability to deal critically with scientific literature and facts.

5. Type of research:

A) Theoretical	4 points	
B) Applied	4 points	X
C) Theoretical with application elements	4 points	
D) It does not correspond to the level of dissertation	0 points	
		one of the answers given is marked with the sign "X"

The level of research must be substantiated if answer D is marked.
The main type of research can be referred to as fundamental with an applied orientation, however, it should be noted that they are based on a serious theoretical foundation. The applied elements are proven by the demonstrated actual testing of the validated SEM/EDS method in laboratory practice.

6. Objectives of the research:

A) Realistic and of scientific and / or applied interest	8 points	X
B) Realistic, but not of scientific and / or applied interest	3 points	
C) Unattainable (unrealistic)	0 points	
		one of the answers given is marked with the sign "X"

Objectives must be specified. The type of the set objectives must be justified.
The aim of the dissertation work is to develop and validate the SEM/EDS method for determining traces of combustion of inorganic explosives and to expand the possibilities for interpreting the obtained data. To achieve this goal, the dissertation sets out five tasks, as follows: 1. Studying the influence of the operating experimental conditions of the SEM/EDS system on the quality of the obtained results and establishing the optimal operating parameters, using the intensity of the EDS signal, contrast and brightness of the SEM image as optimization criteria.

2. Validation of the optimized SEM/EDS method for determining traces of gunpowder burns of inorganic origin according to the requirements of ASTM 1588-17. Evaluation of the main characteristics and uncertainty of GSR particle number measurement.

3. Development of a database for the elemental composition of the GSR of criminal incidents in the Republic of Kosovo applying the validated method.

4. Investigation of the dependence of the number of GSR particles on time after firing a shot with a weapon of a different caliber in the open air and during the performance of usual work activities.

5. Investigation of the dependence of the number of GSR particles on time after firing a shot and the number of shots for weapons of different calibers.

The main goal set in the dissertation, as well as the tasks for its implementation, are completely realistic and, as can be seen, fulfilled 100%. Represent scientific and applied interest in criminal evidence collection.

7. Methods of research:

A) Adequate to research and set objectives	8 points	X
B) Partially appropriate, enabling part of the scientific objectives and / or applications to be achieved	4 points	
C) Inappropriate methods	0 points	
		one of the answers given is marked with the sign "X"

Methods must be specified. The type of methods used is justified.

The SEM/EDS method is traditionally used to study the morphology of gunpowder particles. This is due to the possibility of providing both a high resolution of the image and establishing the elemental composition of the material without disturbing its structure, which helps to carry out additional analyzes if necessary. In this regard, the choice of method and approaches were made completely adequately. The effect of operating parameters of the SEM/EDS method on the ability to improve the identification and quantification of particles characteristic of inorganic explosive burn traces (GSR) was investigated, and the optimal operating parameters were established: accelerating voltage 25 kV, operating distance 10 mm; beam size 5.0 using scanning electron microscope, Quanta 650, FEI, USA, coupled with EDS (Energy Dispersive X-ray Spectrometer) with SDD (Silicon Drift Detector), EDAX, Octane plus a detector. Certified materials were used for method validation: CRM, ENSFI GSR PT sample with code SPS-5P-2A (GSR 2005 PT edition). The CPM for the GSR is a 12 mm diameter double-adhesive carbon layer tape (Pelco™, TedPella, CA) mounted on a 12.7 mm aluminum base-holder (Zeiss model, Al). The thesis shows that the chosen method validation approach satisfies the requirements of ENSF and ASTM 1588-10 for the studied groups of GSR particles. The validated SEM/EDS method was applied to identify particles characteristic of inorganic explosives in samples from firearm incidents in Kosovo and it was demonstrated that the method can provide legal evidence of the existence of GSR on a specific surface. The applied orientation of the

obtained results is proven by the demonstrated real testing of the proposed approaches and methods in laboratory practice. As a result of the obtained data, as a result of the study carried out in the dissertation, a database was developed for the elemental composition of particles characteristic of GSR, which allows identifying new weapons that appeared in Kosovo, and a basis was laid for revising the work protocol for taking samples from GSR in the Republic of Kosovo.

8. Contributions of the dissertation:

A) With lasting scientific and / or applied response, they form the basis for new research and applications	20 points	
B) They are of significant scientific and / or applied interest, complete and / or summarize previous research	16 points	X
C) They are of scientific and / or applied interest	12 points	
D) Lack of significant contributions	8 points	
E) Lack of contributions	0 points	
		one of the answers given is marked with the sign "X"

Contributions must be specified. The type of results achieved must be justified.

The dissertation presents an in-depth study on the possibility of optimizing basic parameters of the SEM/EDS method to find optimal conditions for determining the composition of gunpowder particles (GSR). A consequence of the scientific work are the prominent contributions consisting in expanding knowledge and obtaining new data on the applicability of the SEM/EDS method to determine the elemental composition and stability of these particles such as:

- A study of the effect of working parameters and validation of SEM/EDS method for determination of elemental composition of commonly encountered GSR samples in shooting incidents in Kosovo was done for the first time. Based on the obtained results, a database of elemental composition by SEM/EDS of inorganic GSR encountered in R Kosovo, which allowed new "incomers" on the marked to be identified, was developed.

- The persistence of gunshot residue produced by firearms from criminal cases in R Kosovo was assessed by SEM/EDS as a function of postdischarge time, number of shots, and caliber of ammunition. The data obtained in this study are a base for reconsideration of GSR sampling protocol in Kosovo.

As the most significant contribution, I would single out the demonstration of the real application of the validated SEM/EDS method as a result of the recommendations made for the revision of the working protocol for the sampling of GSR in the Republic of Kosovo.

9. Evaluation of the compliance of the dissertation summary with the dissertation:

A) Full compliance	4 points	X

B) Compliance of the main parts	2 points	
C) Lack of compliance of the main parts	0 points	
		one of the answers given is marked with the sign "X"

The evaluation must be substantiated if answer C is marked.
The abstract is presented in 42 pages and represents an abbreviated version of the essence of the dissertation, reflecting the research work carried out with the presented purpose and tasks, methods and techniques of analysis used and main results obtained. The main observations and conclusions from the dissertation work are reproduced and the contributions of the dissertation research are adequately indicated.

10. Participation of the doctoral student in the achievement of the results of the dissertation:

A) The doctoral student has at least an equal participation	8 points	X
B) The doctoral student has secondary participation	5 points	
C) The participation of the doctoral student is unnoticeable	0 points	
		one of the answers given is marked with the sign "X"

Critical notes must be provided if one of the items B or C is marked.
I have no doubt that the presented analyses and results of M. Tahirukai's dissertation work are reliable. During the implementation of the individual doctoral plan, he demonstrated skills for independent thinking and analysis, abilities for accurate and structured exposition, as well as abilities for summarizing achieved scientific results. His many years of experience as an expert at the ballistics laboratory of the Kosovo Forensic Science Agency undoubtedly contributed to the correct implementation of the set goals. The doctoral student is a member of the team of the specified laboratory, where his main work task is the collection of evidentiary material to determine the type of fire particles and their characterization. Together with the nine people participating in the study (experts from the Ballistics Laboratory of the Kosovo Forensic Science Agency), analyses were carried out on 555 samples from 144 incidents in Kosovo, of which 14% were found to be positive for GSR. The firearms selected for the analysis were used in real crime cases in Kosovo, and the ammunition used in the tests was provided by the Ballistics Laboratory of the Kosovo Forensic Science Agency. Also included in the study were weapons submitted for registration in the Integrated Ballistic Identification System (IBIS) database, as well as the Glock 19 (9x19 mm) sub-caliber pistol used by the Kosovo Police Service. What has been mentioned so far gives me a reason to believe that the Milazim Tahirukaj actively participated both in the implementation of the set experimental

tasks and in summarizing the results and presenting them to the scientific community in the form of publications and participation in scientific forums.

11. Critical notes:

A) Lack of critical notes	8 points	X
B) Critical notes of a technical nature	7 points	
C) Critical notes that would partially improve the results achieved	4 points	
D) Significant critical notes	0 points	
		one of the answers given is marked with the sign "X"

Critical notes must be provided if one of the answers C or D is marked.
I have no critical remarks on the presented dissertation. The the doctoral thesis was repeatedly discussed in the Department of Analytical Chemistry during the implementation of the individual plan for the doctoral dissertation.

12. Conclusion

A) The evaluation of the dissertation is **POSITIVE**	This evaluation is assigned to a total number of at least 65 points	X
B) The evaluation of the dissertation is **NEGATIVE**	This evaluation is assigned to a total number below 65 points	
		one of the answers given is marked with the sign "X"

To be filled in at the request of the reviewer
Milazim Tahirukai's doctoral dissertation submitted to me for review contains a large volume of experimental work with significant results in the field of validation of the SEM/EDS method for the analysis of gunpowder particles with direct application in ballistic laboratories. The topicality of the issues involved, the well-presented information, and the presentation of the results in 2 scientific publications, and 2 participations in scientific conferences fully meet the requirements for formulating a dissertation work and the Rules of HTMU for the acquisition of scientific degrees. The total number of points according to indicators from 1-11 of the presented review is 94. All this gives me a reason to POSITIVELY evaluate Milzim Tahirukai's doctoral dissertation for the award of the educational and scientific degree "Doctor" in scientific specialty 4.2. Chemical Sciences (Analytical Chemistry).

	The review was written by:	
date		signature