

**STUDY TO ESTABLISH THE PRESENCE AND QUANTIFICATION OF THE
ELEMENTS AT VARIOUS RANGES**Abhijit A Parmar^{*}, Dr. G.Rajesh Babu^{**}, Dr. M.S.Dahiya^{***}^{*} Research Scholar, Institute of Forensic Science, Gujarat Forensic Sciences University Gandhinagar, Gujarat, India^{**}Assistant Professor, Institute of Forensic Science, Gujarat Forensic Sciences University, Gandhinagar Gujarat, India^{***}Director, Institute of Forensic Science, Gujarat Forensic Sciences University, Gandhinagar, Gujarat, India

Abstract: One of the most important and most conventional subjects of forensic science is ballistics. This is the study of firearms and ammunitions. In most of the crimes those are related to firearms, there is a need of in-depth analysis in the involved material such as the firearm and bullets and the residue particles.

Though various microscopic examination and analysis are available for the identification of the firearms and ammunition, the reliable sources of information about the individual about the evidence of fire or gunshot is established through the gunshot residue analysis. By the terminology itself, it is understood that the analysis is being done on the residual matter obtained from the gunshot. This would be useful to determine the type of weapon or ammunition involved in the specific crime or incidence. So we present in this study of GSR analysis by SEM-EDX, EDXRF at various ranges from we collect the GSR. We explore the Qualitative and Quantitative analysis of GSR particle at various ranges to determine the presence of GSR particle at various ranges.

1. INTRODUCTION

The investigation of gunshot residue (GSR) is essential to the forensic examination of suspected cases of firearm use. Antimony, lead and barium are the major elements present in GSR. The amount of these elements on the hands of shooters depends on several factors, such as the type of weapon and munitions used, the age and condition of the weapon, the suspect's personal hygiene and occupational habits, biometrics, and the time elapsed since the firing. Gunshot residue results from the discharge of a firearm. This includes primer, powder, and/or projectile material and products of their combustion. The residue that can be deposited on the hands of a shooter is usually the result of gases or particles escaping through openings in the weapon (such as the cylinder area of a revolver) or leaving the end of a barrel.⁽¹⁾

When a weapon is fired, a great volume of incandescent (circa 2000 ° C) gaseous material is produced. This gaseous material is mainly the combustion products from the propellant and consists of carbon dioxide, carbon monoxide, water as steam and oxides of nitrogen. In amongst this vast cloud of gases are also partially burnt and unburnt propellant particles and combustion products from the priming compound. These solid particles are collectively called GSR particles. Less frequently, they are also referred to as firearms discharge residues.⁽²⁾⁽³⁾

The GSR is found on:

- Hand of person who fired the gun and clothing.
- The target around the projectile hole, including cloths and explode skin.
- The firearms also contain GSR on its inner and outer surface.
- The area and the articles around the target and around the shooter.
- Intermediate target.⁽³⁾

Because of air and atmosphere we also collect the GSR particle at some distance.

The goals of study are:

Collection of GSR particle at various ranges for microscopic analysis, To establish the presence of various elements in the GSR particle, To establish the quantity of the elements present in GSR particle, To establish the association of GSR particle with that of the ammunition, To establish the possible association of the elemental concentration and the range of firing.

2. METHODOLOGY

Samples were collected from ballistics research center and testing range (BRCTR). Samples were taken in drawing sheet by placing it before the target and then by universal receiver the AK-47 rifle (7.62X39 caliber), the SLR rifle (7.62X51 caliber) and 9mm pistol fired on paper at 5M, 6M and 10M. Sample were placed in plastic cover for avoid from contamination

GSR. Total 50 sample were collected. The samples show the entry and exit hole of that projectile. Some sample and their result are shown below:

Sample No.	Gun Type	Bullet or Cartridge Case	Distance
1.	AK-47	Soft steel core	5 meters
2.	SLR rifle	7. 62mm pointed bullet	5 meters
3.	AK-47	Soft steel core	5 meters
4.	SLR rifle	7. 62mm pointed bullet	6 meters
5.	AK-47	Soft steel core	6 meters
6.	AK-47	Soft steel core	10 meters
7.	AK-47	Soft steel core	10 meters
8.	SLR rifle	7.62 mm pointed bullet	10 meters
9.	9mm	Round Nose bullet	5 meter
10.	9mm	Round Nose bullet	5 meter

TABLE NO. 1

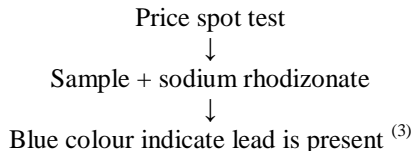
3. Experimental work

I have done analysis of both part first for entrance hole of bullet and the periphery part of sample by spot test, EDXRF and SEM. So I have done analysis of only that part where the Probability of getting the GSR was high. Spot test was done for GSR detection. Following test were performed for all samples.

For nitrite

Sample of firing marks and periphery
↓
Take Photo bromide paper was dipped in alpha-naphthyl amine and sulphanilic acid.
↓
This paper is then swabbed on suspect site; appearance of pink colour shows the presence of nitrate.

For lead



Instrumental work

1. EDXRF analysis

First of all kept input voltage 100v AC for EDXRF machine. Liquid nitrogen was poured in the dewar. The system and computer were switched on and in computer by starting the EDX application was started, the status was checked for machine. The sample was analyze in EDXRF, For this first of all I got the part of the sample in which chance of getting GSR was higher than it put in sample holder part of EDXRF machine and analysis was started. Than the data was stored and all result was saved for make a conclusion. For this analysis method no any extraction method was used, only the part of sample put in instrument and analysis was done.⁽⁴⁾⁽⁵⁾⁽⁶⁾

2. RAMAN Spectroscopy

All Raman images and spectra were recorded using an alpha 300 RAS

Equipped with:

- a CCD camera,
- an UHTS spectrometer,
- a frequency-doubled Nd:YAG laser used for 532 nm excitation,
- a Zeiss 50x objective (N.A.= 0.7; WD: 1.1).

The camera and spectrometer parameter are listed below:

- Width [Pixels] :1024
- Height [Pixels] :127
- Temperature [°C]: -65
- Readout : Full Vertical Binning
- Excitation Wavelength : [nm] 532
- Grating [g/mm] : 600, BLZ= 500 nm
- Spectral Center [rel.1/cm]: 2000

The measurements were acquired using the WITec Control software and analysed with the WITec Project and the WITec Project Plus software, respectively. The principles of the data analysis features used, are explained in detail in the Software appendix.⁽⁸⁾

SEM-EDX analysis

First of all main switch of SEM, EDXRF, rotary, EDSPC, EDS, monitor, printer were started.

Stand by button was pressed and wait for 2 minutes. Then the PC and power button were started, after PC log on was completed, start the SMARTSEM user interface to start the software. Then the server was put for initialization by vent the SEM for load the sample. Then go to the SEM control vacuum tube.

Then by setting the portion of sample of stub size on SEM stub where the chance of getting GSR particle was higher and noted down the stub no. The analysis was started. The contrast and brightness was set by select the levels of tool bar in application. The magnification was also increased by tool bar. Image was got where the particle of GSR were clearly seen was take in INCA software. Navigation part was stated in application to find the site of interest in image for take a graph. Quantification results were seen and generate report and file was saved. These analyses were done for all samples.

4. RESULT AND DISSCUTION

Sample	Firearm	Test	Observation	Result
1.	AK-47	Walker's Test	Pink colour present	Nitrite present
2.	SLR rifle	Walker's Test	Pink colour present	Nitrite present
3..	AK-47	Walker's Test	Pink colour present	Nitrite present
4.	SLR rifle	Walker's Test	Pink colour present	Nitrite present
5.	AK-47	Walker's Test	Pink colour present	Nitrite present
6.	AK-47	Walker's Test	No colour Present	Nitrite absent
7.	AK-47	Walker's Test	No colour present	Nitrite absent
8..	SLR rifle	Walker's Test	No colour present	Nitrite absent
9.	9mm	Walker's Test	No colour Present	Nitrite absent
10.	9mm	Walker's Test	No colour present	Nitrite absent

TABLE NO.2 WALKER'S TEST RESULT

Sample	Firearm	Test	Observation	Result
1.	AK-47	Price spot test	Blue colour present	Lead present
2.	SLR rifle	Price spot test	Blue colour present	Lead present
3..	AK-47	Price spot test	No colour present	Lead absent
4.	SLR rifle	Price spot test	Blue colour present	Lead present
5.	AK-47	Price spot test	Blue colour present	Lead present
6.	AK-47	Price spot test	Blue colour Present	lead present
7.	AK-47	Price spot test	No colour present	Lead absent
8..	SLR rifle	Price spot test	No colour present	Lead absent
9.	9mm	Price spot test	No colour present	Lead absent
10.	9mm	Price spot test	No colour present	Lead absent

TABLE NO.3 PRICE SPOT TEST RESULT

EDXRF RESULT

Sample : SAMPLE 3 GSR AT 5M
 Operator:
 Comment: Quickseasy Air-Metal
 Group : easy
 Date : 2014-01-22 13:14:41

Measurement Condition

Analyte	TG kV	uA	FI Acq. (keV)	Anal. (keV)	Time (sec)	DT (%)
Ti-U	Rh 50	182-Auto	Out 0 - 40	0.0 - 40.0	Real - 100	24
Na-Sc	Rh 15	1000-Auto	Out 0 - 20	0.0 - 4.4	Real - 99	24

Quantitative Result

Analyte	Result	Std.Dev.	Proc.-Calc.	Line	Intensity (cps/uA)
Ca	86.721 %	0.277	Quan-FP	CaKa	1.532
Ba	7.672 %	0.287	Quan-FP	BaLa	0.114
Fe	2.320 %	0.043	Quan-FP	FeKa	0.424
Cu	2.237 %	0.027	Quan-FP	CuKa	0.895
Pb	0.658 %	0.025	Quan-FP	PbLb1	0.229
Zn	0.327 %	0.016	Quan-FP	ZnKa	0.158
Sb	0.252 %	0.018	Quan-FP	SbKa	0.013

EDXRF SPECTRA AT 5 METER SAMPLE 3

Sample : SAMPLE 4 GSR AT 5M
 Operator:
 Comment: Quickseasy Air-Metal
 Group : easy
 Date : 2014-01-22 13:36:50

Measurement Condition

Analyte	TG kV	uA	FI Acq. (keV)	Anal. (keV)	Time (sec)	DT (%)
Ti-U	Rh 50	90-Auto	Out 0 - 40	0.0 - 40.0	Real - 99	24
Na-Sc	Rh 15	598-Auto	Out 0 - 20	0.0 - 4.4	Real - 99	26

Quantitative Result

Analyte	Result	Std.Dev.	Proc.-Calc.	Line	Intensity (cps/uA)
Ca	83.103 %	0.264	Quan-FP	CaKa	2.670
Cu	9.132 %	0.046	Quan-FP	CuKa	7.056
Ti	2.510 %	0.101	Quan-FP	TiKa	0.200
Fe	2.265 %	0.042	Quan-FP	FeKa	0.844
K	1.120 %	0.151	Quan-FP	K Ka	0.077
Zn	1.011 %	0.020	Quan-FP	ZnKa	0.941
Pb	0.842 %	0.026	Quan-FP	PbLb1	0.322
Ac	0.161 %	0.017	Quan-FP	AcLa	0.149
Sb	0.156 %	0.009	Quan-FP	SbKa	0.156

EDXRF SPECTRA AT 5 METER SAMPLE 4

Sample : SAMPLE 5 GSR AT 5M
 Operator:
 Comment: Quickseasy Air-Metal
 Group : easy
 Date : 2014-01-22 13:01:04

Measurement Condition

Analyte	TG kV	uA	FI Acq. (keV)	Anal. (keV)	Time (sec)	DT (%)
Ti-U	Rh 50	112-Auto	Out 0 - 40	0.0 - 40.0	Real - 100	24
Na-Sc	Rh 15	676-Auto	Out 0 - 20	0.0 - 4.4	Real - 98	26

Quantitative Result

Analyte	Result	Std.Dev.	Proc.-Calc.	Line	Intensity (cps/uA)
Ca	90.778 %	0.274	Quan-FP	CaKa	2.611
Ti	3.543 %	0.118	Quan-FP	TiKa	0.230
Fe	2.997 %	0.046	Quan-FP	FeKa	0.894
Pb	1.390 %	0.027	Quan-FP	PbLb1	0.822
K	0.976 %	0.133	Quan-FP	K Ka	0.065
Ac	0.179 %	0.014	Quan-FP	AcLa	0.164
Cu	0.138 %	0.019	Quan-FP	CuKa	0.091

EDXRF SPECTRA AT 5 METER SAMPLE 5

Sample : SAMPLE 6 GSR AT 6M
 Operator :
 Comment : Quick&easy Air-Metal
 Group : easy
 Date : 2014-01-22 13:36:50

Mesurement Condition

Analyte	TG kV	uA	FI Acq. (keV)	Anal. (keV)	Time (sec)	DT (%)
Ti-U	Rh 50	90-Auto	Out 0 - 40	0.0 - 40.0	Real - 99	24
Na-Sc	Rh 15	598-Auto	Out 0 - 20	0.0 - 4.4	Real - 99	26

Quantitative Result

Analyte	Result	Std.Dev.	Proc.-Calc.	Line	Intensity (cps/uA)
Ca	83.103 %	0.264	Quan-FP	CaKa	2.670
Cu	9.132 %	0.046	Quan-FP	CuKa	7.056
Ti	2.510 %	0.101	Quan-FP	TiKa	0.200
Fe	2.265 %	0.042	Quan-FP	FeKa	0.844
Sb	1.120 %	0.151	Quan-FP	K Ka	0.077
Zn	1.011 %	0.020	Quan-FP	ZnKa	0.941
Pb	0.542 %	0.026	Quan-FP	PbLb1	0.322
Ac	0.161 %	0.017	Quan-FP	AcLa	0.149
K	0.156 %	0.009	Quan-FP	SbKa	0.156

EDXRF SPECTRA AT 6 METER SAMPLE 6

sample : SAMPLE 7 GSR AT 6M
 operator:
 Comment : Quick&easy Air-Metal
 Group : easy
 Date : 2014-01-22 11:52:11

Mesurement Condition

Analyte	TG kV	uA	FI Acq. (keV)	Anal. (keV)	Time (sec)	DT (%)
Ti-U	Rh 50	67-Auto	Out 0 - 40	0.0 - 40.0	Real - 100	24
Na-Sc	Rh 15	396-Auto	Out 0 - 20	0.0 - 4.4	Real - 100	25

Quantitative Result

Analyte	Result	Std. Dev.	Proc.-Calc.	Line	Intensity (cps/uA)
Ca	76.392 %	0.247	Quan-FP	CaKa	3.782
Ti	20.492 %	0.202	Quan-FP	TiKa	2.476
K	1.519 %	0.137	Quan-FP	K Ka	0.167
Fe	0.972 %	0.037	Quan-FP	FeKa	0.459
Cu	0.376 %	0.021	Quan-FP	CuKa	0.402
Pb	0.249 %	0.025	Quan-FP	PbLb1	0.242

EDXRF SPECTRA AT 6 METER SAMPLE 7

sample : SAMPLE 8 GSR AT 10 M
 operator:
 Comment : Quick&easy Air-Metal
 Group : easy
 Date : 2014-01-22 11:52:11

Mesurement Condition

Analyte	TG kV	uA	FI Acq. (keV)	Anal. (keV)	Time (sec)	DT (%)
Ti-U	Rh 50	67-Auto	Out 0 - 40	0.0 - 40.0	Real - 100	24
Na-Sc	Rh 15	396-Auto	Out 0 - 20	0.0 - 4.4	Real - 100	25

Quantitative Result

Analyte	Result	Std. Dev.	Proc.-Calc.	Line	Intensity (cps/uA)
Ca	76.392 %	0.247	Quan-FP	CaKa	3.782
Ti	19.492 %	0.202	Quan-FP	TiKa	2.476
K	1.519 %	0.137	Quan-FP	K Ka	0.167
Pb	1.249 %	0.115	Quan-FP	PbLb1	0.242
Fe	0.972 %	0.037	Quan-FP	FeKa	0.459
Cu	0.376 %	0.021	Quan-FP	CuKa	0.402

EDXRF SPECTRA AT 10 METER SAMPLE 8

Sample : SAMPLE 9 GSR AT 10M
Operator :
Comment : Quick&easy Air-Metal
Group : easy
Date : 2014-01-22 12:27:49

Mesurement Condition

Instrument : EDX-700	Atmosphere : Air	Collimator : 10 (mm)	Spin : No
Analyte	TG kV	uA	FI Acq. (keV) Anal. (keV) Time (sec) DT(%)
Ti-U	Rh 50	150-Auto	Out 0 - 40 0.0 - 40.0 Real - 99 24
Na-Sc	Rh 15	887-Auto	Out 0 - 20 0.0 - 4.4 Real - 99 26

Quantitative Result

Analyte	Result	Std.Dev.	Proc.-Calc.	Line	Intensity(cps/uA)
Ca	71.091 %	0.233	Quan-FP	CaKa	1.746
Ti	14.719 %	0.159	Quan-FP	TiKa	0.969
Ba	9.292 %	0.270	Quan-FP	BaLa	0.214
Cu	2.849 %	0.027	Quan-FP	CuKa	1.489
Fe	1.551 %	0.038	Quan-FP	FeKa	0.368
Zn	0.309 %	0.015	Quan-FP	ZnKa	0.195
Ac	0.136 %	0.014	Quan-FP	AcLa	0.096
Co	0.053 %	0.025	Quan-FP	CoKa	0.016

EDXRF SPECTRA AT 10 METER SAMPLE 9

Sample : SAMPLE 10 GSR AT 10 M
Operator :
Comment : Quick&easy Air-Metal
Group : easy
Date : 2014-01-22 12:38:25

Mesurement Condition

Instrument : EDX-700	Atmosphere : Air	Collimator : 10 (mm)	Spin : No
Analyte	TG kV	uA	FI Acq. (keV) Anal. (keV) Time (sec) DT(%)
Ti-U	Rh 50	500-Auto	Out 0 - 40 0.0 - 40.0 Real - 99 12
Na-Sc	Rh 15	1000-Auto	Out 0 - 20 0.0 - 4.4 Real - 100 5

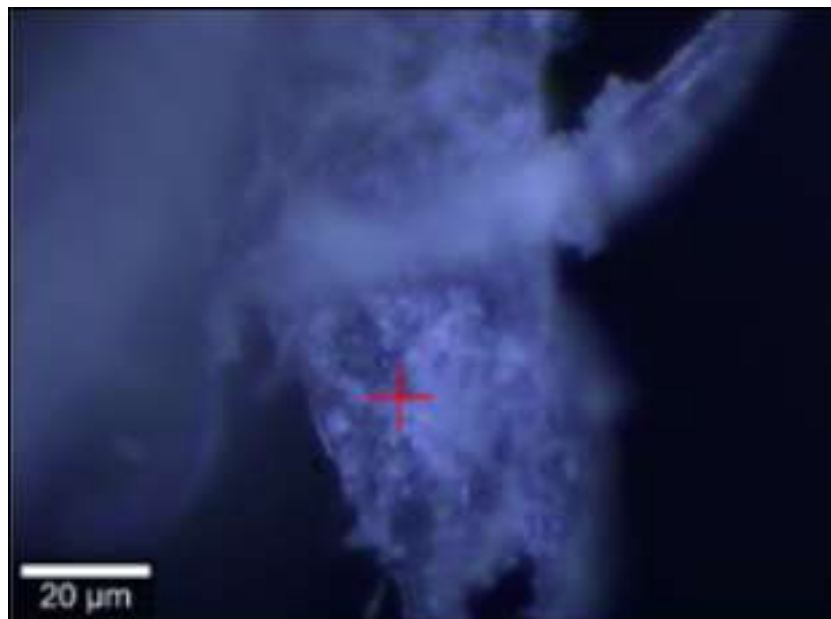
Quantitative Result

Analyte	Result	Std.Dev.	Proc.-Calc.	Line	Intensity(cps/uA)
Ca	63.989 %	0.807	Quan-FP	CaKa	0.103
Ti	10.070 %	0.297	Quan-FP	TiKa	0.052
Ba	8.785 %	0.635	Quan-FP	BaLa	0.016
Cu	6.709 %	0.072	Quan-FP	CuKa	0.278
K	4.953 %	0.577	Quan-FP	K Ka	0.015
Fe	1.584 %	0.084	Quan-FP	FeKa	0.031
Zn	0.712 %	0.038	Quan-FP	ZnKa	0.036
Hg	0.527 %	0.051	Quan-FP	HgLa	0.018
Sb	0.252 %	0.018	Quan-FP	SbKa	0.013

EDXRF SPECTRA AT 05 METER SAMPLE 10

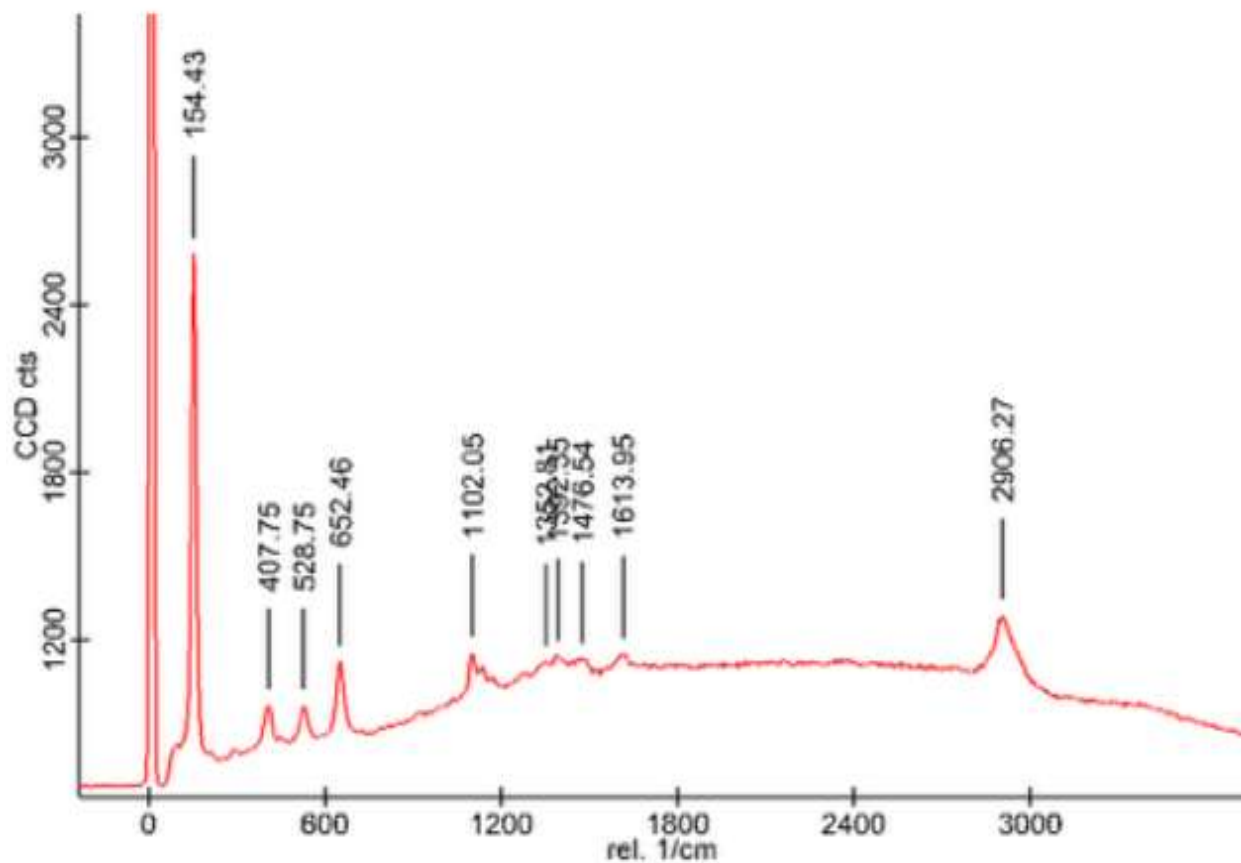
RAMAN Spectroscopy Results

The following pages shows the result of the sample measurement done on your samples. For each sample a VIDEO IMAGE is captured in bright field microscopy. In the VIDEO IMAGE indication of SINGLE SPECTRA positions are marked by +(cross hair), LINE SCAN is marked by - (straight line across the image) and RAMAN IMAGE is marked by □(square). Depending on the size of the scan it can be done using PIEZO SCANNER(>200 microns) or STEPPER MOROR SCANNER(<200 microns). For the DATA EVALUATION purpose all spectra are COSMIC RAY removed and BACKGROUND SUBTRACTED(see software appendix). Afterwards the K - MEANS CLUSTER ANALYSIS is used in order to find different spectra that may represents different materials or differences within the material. Using these spectra in combination with the recorded spectral array, the BASIS ANALYSIS is performed in order to calculate false color RAMAN IMAGES for the scanned area. Each of these images shows the intensity distribution for the scanned area of one spectrum. The COLOR COMBINED RAMAN IMAGE is the result of coloring each false color Raman image and their combination. The colors of all shown color combined Raman images correspond to the colors of the spectra typically shown next to it. Mixed colors correspond to mixed spectra or – in other words – to mixed components, effects, etc.



Optical image of the sample 8

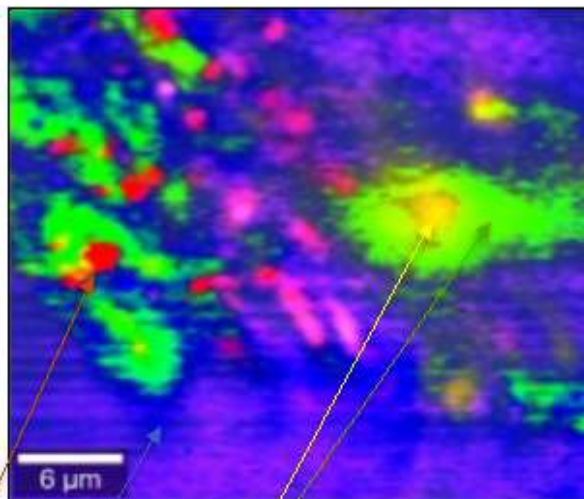
The cross marked in RED is the point where a single spectrum was acquired shown below.



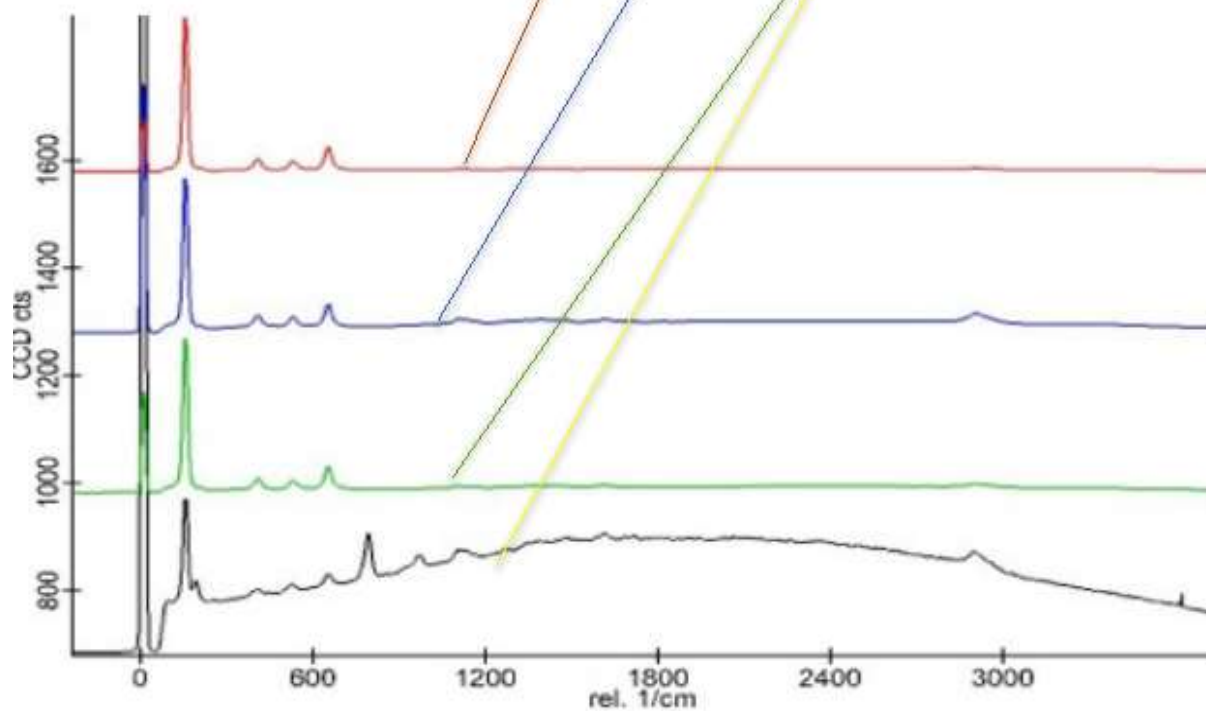
Raman spectra of selected location of sample 8



Optical image of the sample A. The box marked in RED is the area where an image was acquired.

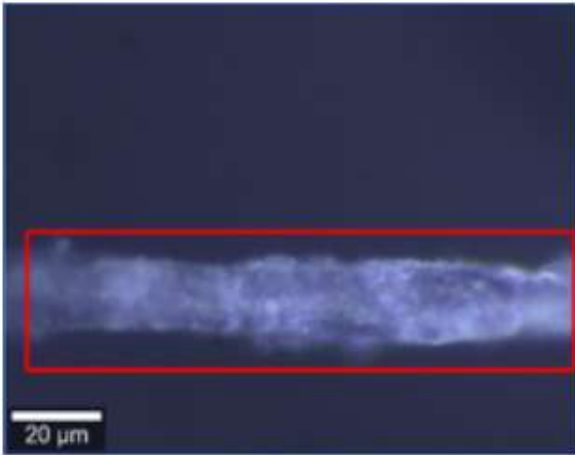


False color RAMAN image generated from the image to the left.

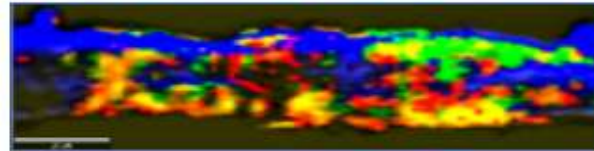


Different RAMAN spectrum color coded according to the image shown above.

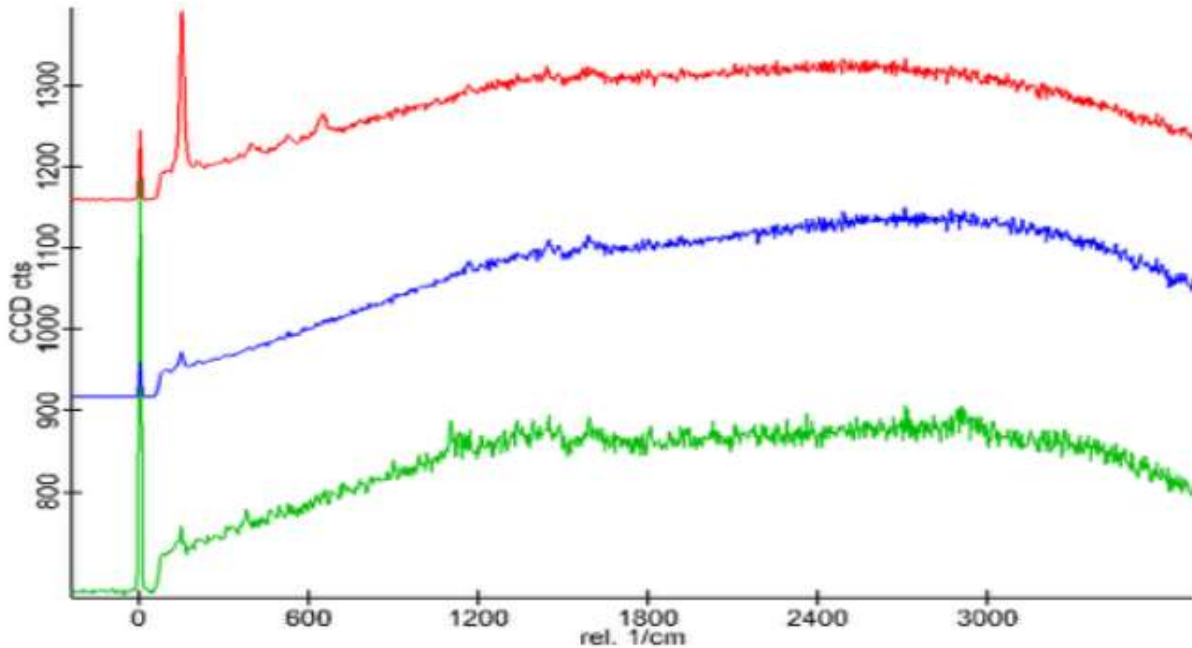
SAMPLE 10



Optical image of the sample 10.



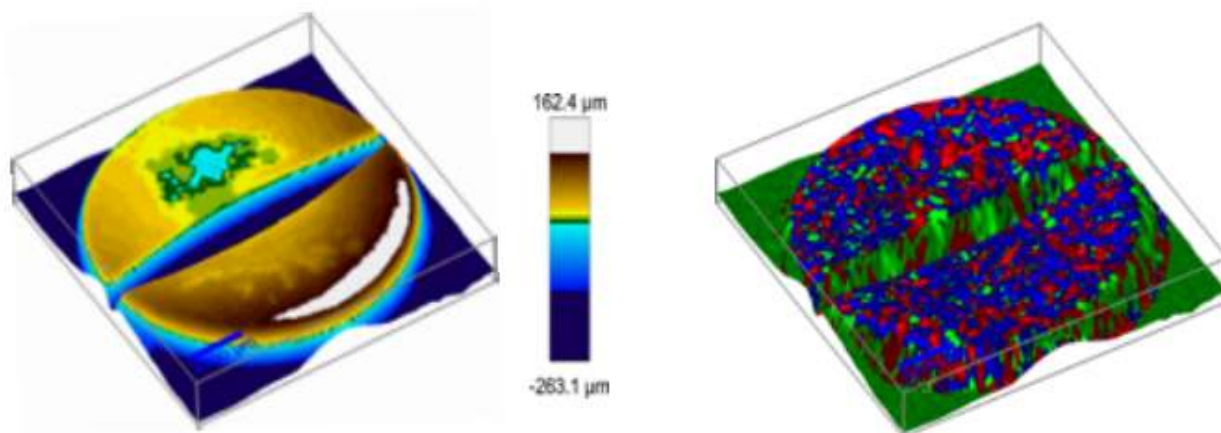
False color RAMAN image generated from the image to the left



Different RAMAN spectrum color coded according to the image shown above.

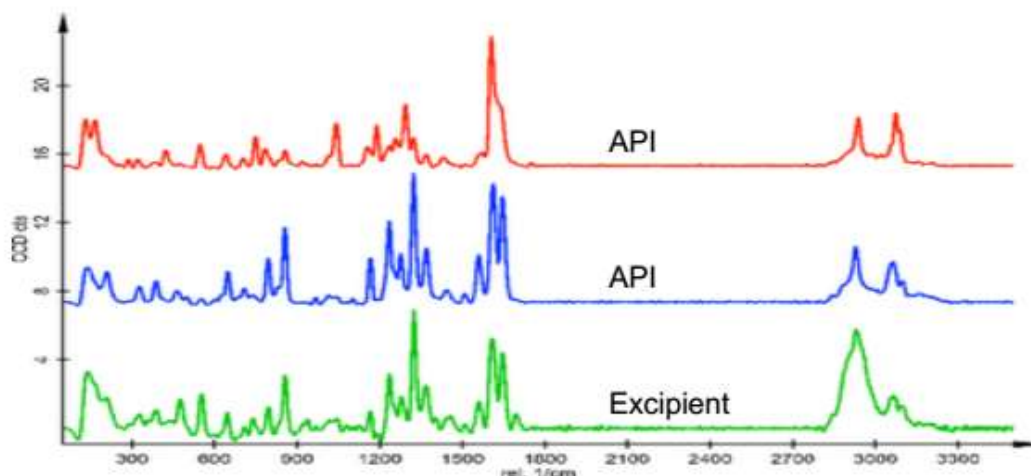
Ideal Case Measurement for samples

Different RAMAN spectrum color coded according to the image shown above. We have a system called True Surface. With this system we are able to measure the complete bullet impact point and do a profilometer scan and the follow that to do RAMAN scan. The advantage of such a system is you get the topographical features from Profilometer and then the chemical information using RAMAN spectroscopy. An example of such a scan is given below.



Complete profile of the aspirin tablet. The height variation in this case is around 450 microns.

Once the profile is acquired, it is easier to follow the same profile and do a complete analysis of the sample using RAMAN.



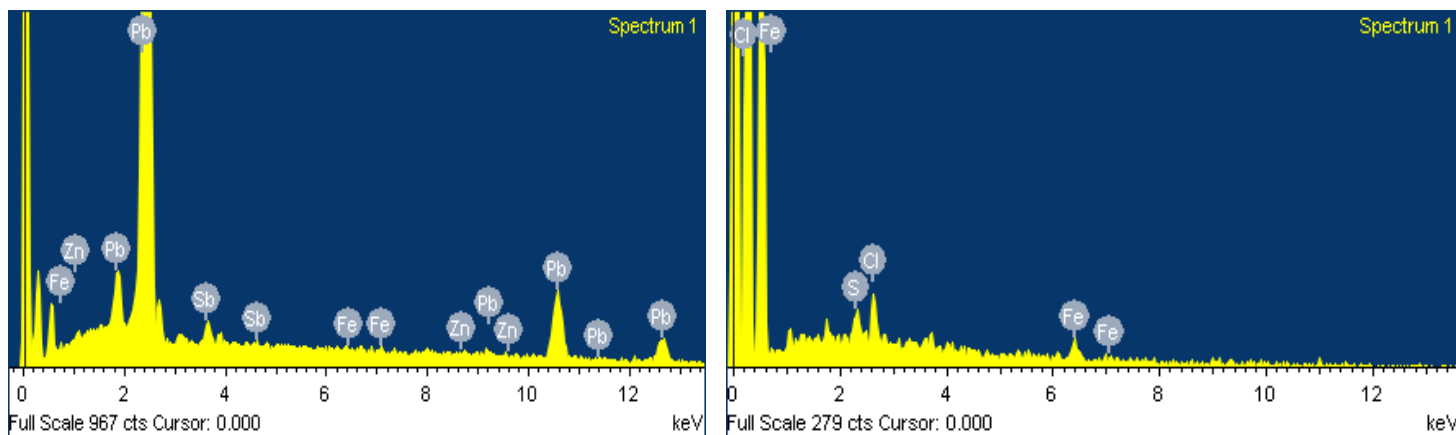
Different RAMAN spectrum color coded according to the image shown above.

- This results show that at 1.5 meter ranges the GSR particle were clearly seen like antimony(Sb), lead (Pb), Zinc(Zn), copper(Cu), Iron (Fe) and which seen in EDXRF graph as above. Nitrite was also detected in spot test.
- For range of 5 Meter EDXRF graph show that Iron (Fe), Lead (Pb), Zinc (Zn) and Copper (Cu) were present. Copper and Iron were found in relatively large quantity. In SEM Iron (Fe), Antimony (Sb) and Lead (Pb) were found. Portion of Lead is encouraging. In spot test at this range Nitrite (NO₂) and Lead (Pb) was detected.
- For range of 6 meter EDXRF graph shows that Iron (Fe), Lead (Pb), Antimony (Sb) and Zinc (Zn) were found again Iron was in relative high quantity. In spot test Nitrite (NO₂) and Lead (Pb) were found. Nothing was found in SEM. So from this result remarkable success was found in form of antimony, zinc and lead
- For range of 10m EDXRF Zinc (Zn), Cobalt (Co), Copper (Cu), Antimony (Sb), Mercury (Hg) and Iron (Fe) are found. In SEM Sulphur (S) and Iron (Fe) were found. In spot test Nitrite (NO₂) and Lead (Pb) were found.

The most ideal way to analyze the sample will be to send it for True Surface Scan, however with the present results we can summaries some points:

- The RAMAN spectrum shows high intensity in the lower wave numbers, indicating the presence of metal oxides.
- The spectrum at around 2900 cm^{-1} also indicates some organic residue.
- At some points in the sample there is also a lot of Fluorescence. This can indicate either presence of metals or of defects.
- RAMAN spectrum show peak in between 1300- 1600 cm^{-1} which also show the sign of di phenyl amine.

SEM Result: Analysis of GSR samples by Scanning electron microscope are shown as below:



5. CONCLUSION

At the range of **5 meter** (around 15 samples), the SEM results showed the presence of **Lead (Pb)**, **Antimony (Sb)** and **Iron (Fe)**, this proves that GSR could be detected at this ranges. In case of **10 meter** Range iron was found in both samples Calcium and **Sulphur** were found in less extent.

EDXRF results showed the presence of **Lead, Iron** and **Zinc** in concurrence with the SEM results. In the same way the range of **10 meters** (around 20 samples), the presence of **Iron (Fe)**, **Zinc (Zn)** and **Copper (Cu)** were found. There was a remarkable presence of mercury in the sample obtained at the range of **10 meters**.

The GSR analysis was found to be successful with **5 meter** and **10 meter** ranges but in case of lead and iron.

In case of **6 meter** (around 15 samples) also there is a presence of **Lead (Pb)**, **Iron (Fe)**, **Copper (Cu)**, **Zinc (Zn)** and **Antimony (Sb)**, which is more remarkable.

Even the spot test confirmed the presence of Lead (Pb) in the range of 10 meters.

In RAMAN spectroscopy the high intensity at low wave number show the presence of metal oxide and some organic peak also indicate the presence of GSR particles. near 1352 cm^{-1} peak also show the presence of diphenyl amine derivatives It shows that the average spectra of the four shots are quite similar. The spectral variance in the range of 1650–1300 cm^{-1} is more pronounced for diphenyl derivatives.

Though it is possible and establishes the presence of various specific elements relevant to the study those are distinctly available in the GSR particles. The further tests may be conducted for various ranges to get the firing result. Possible better result will be yielded if the samples are detected from other firearm and also get better result from different ranges.

6. REFERENCES

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