

# ESTIMATION OF BLACKENED BULLET RESIDUE OF PROJECTILES FIRED THROUGH POLY COATED WINDOWPANE BY NAA AND ICP-AES TECHNIQUES

Kulbhooshan Gupta<sup>1</sup>, JagdishNaik<sup>1</sup>, N.P.Waghmare<sup>1\*</sup>

<sup>1</sup>Goa State Forensic Science Laboratory, Verna Goa.

## Abstract:

Systematic investigations of bullet residues deposited around the entry of bullet hole when fired through poly coated thin film windowpane has been carried out. The trace elements originating from bullet residues on filter paper were produced by firing through poly coated thin film windowpane on filter papers from various positions of thin films (front and back side from muzzle end) using .315" rifle, 9mm pistol, .303" and AK47 rifles as firearms. Antimony, Barium and Copper were quantitatively determined by RNAA after irradiating the samples in the Nuclear Reactor. Nickel, Lead and Zinc were analyzed in residual solutions by ICP-AES. Characteristics group of trace elements were quantitatively estimated in relation to type of glass, ammunition used and of the area around the bullet hole.

**Key words:** RNAA, windowpane, firearm, ammunition, blackened bullet residue, ICP-AES.

## Introduction:

Neutron Activation analysis (NAA) is well established method for the quantitative estimation of the elements at trace/lower level. Forensic applications of trace element analyses are guided by the concept that materials of forensic interest can be "uniquely" characterized by their trace element profile. Practically the trace element profile can help to establish the common origin of target materials. Hence it is only natural that the nuclear activation technique, with its high sensitivity and specificity coupled with almost universal applicability, has been applied with fervor to a variety of matrices [1-2]. Trace elemental analysis has become an indispensable tool of almost every forensic laboratory and the present day modern analytical techniques both nuclear and non-nuclear formed an integral part of such analysis. Hence the application of Neutron Activation Analysis in forensic science has turned out to be known as forensic activation analysis (FAA) and in general, it could be the most sensitive trace analytical technique for many elements in the periodic table. Further, the capability to perform simultaneous multi element analysis has attracted the major research effort for many years [3-5].

Trace element concentrations of forensic samples are frequently determined by neutron activation analysis in order to find out whether the samples have a common origin or not? The detection of blackened bullet residue fired through poly coated intermediate target is undoubtedly of great importance in shooting cases provided (a) the detection method is reliable (b) the investigating officer /crime scene manager are familiar with all the pre requisites of sampling procedure etc. Uses of Forensic Neutron Activation Analysis of bullet lead specimen are reported [6]. Soft pointed and jacketed bullet hole identification fired through paper target backed by poly coated thin film glass sheets were analyzed in the present work. The identification of bullet holes in a variety of thickness of glass is often required in criminal cases involving shooting incidence through windowpane. This is effectively done by Neutron Activation Analysis of the paper/cloth material in which the holes are present. As lead and nickel are not easily amenable to be analyzed by NAA, ICP-AES of residual solutions after performing RNAA were carried out by the earlier reported procedure (for Pb, Ni and Zn) [7-8]. Combination of analytical technique i.e. RNAA/ICP-AES provided the trace element quantitative information for six characteristic trace elements viz. Barium, Copper, Antimony, Lead, Nickel and Zinc (refer

figure 1 for schematic diagram of radio chemical separation and ICP-AES for Ba, Cu, Sb, Pb, Ni and Zn). Table IV indicates the production and decay properties of radionuclides detected in this study.

## Method and Materials:

### Sample preparation and irradiation conditions:

The soft pointed and jacketed bullet specimens on filter paper (fired through polypaper coated thin film windowpanes) were collected and prepared for neutron irradiation with minimum handling to avoid any chance of contamination. Each specimen was shown in half and a slice weight was cut from each half to supply duplicate samples. The specimens of blackened bullet residue were packed inside the clean polythene separately. Appropriate standards quantities of the elements Ba, Cu, Sb were prepared for irradiation simultaneously with the specimens to be analyzed. Standards were prepared by pipetting known volume of solutions of known concentration on cleaned polythene under IR lamp keeping a distance. In present work, high purity germanium (HPGe) detector having high and good sensitivity was utilized for gamma spectrometry and counting.

Some common cartridges (except AK-47 ammunition) manufactured in the Indian ordinance factories were chosen for experiments (see Table I and II). The soft pointed as well as jacketed bullets were fired through normal as well as poly paper coated thin film windowpane keeping paper/cloth target backside of glass target. The residues produced at exit side of paper target as a result of firing of the blackened bullet residue pattern were collected on a piece of dry whatman filter paper. Sixteen blackened bullet residues were obtained from the various ammunition. These samples along with the control filter paper were taken for analysis. The samples along with controls and standards were irradiated in the nuclear reactor, a swimming pool type reactor, at a neutron flux about  $10^{12} \text{ n.cm}^{-2}.\text{sec}^{-1}$  for appropriate period. After irradiation, the samples were digested with nitric acid and perchloric acid mixture in the presence of microgram carriers. After digestion, 2 ml hydrochloric acid was added and solution made up to 10 ml by adding water. 5ml solutions were used by RNAA (Radiochemical Neutron Activation Analysis) for determination of Ba, Cu, and Sb. Remaining 5ml solutions were subsequently used for determination of Pb, Zn, and Ni by ICP-AES after considerable decay of radioactivity.

The bullet hole is identified from antimony, lead & zinc contents at and around the hole. It is found that antimony, lead are usually present were in microgram amounts. Although lead would be expected to be present in a similar pattern and in larger amounts. Antimony pattern is consistent, reproducible to satisfactory extent for this purpose and interpretation on the basis of the presence of antimony, lead and zinc can be valid. The control values of samples are generally barium free, though antimony has been found on some samples depending on the residue deposited at front, back or without film. Nevertheless the concentration difference of antimony and zinc between firing through an intermediate and control samples is generally significant in a decision as to whether the firing through normal or poly paper coated thin film has taken place or not.

## Result and Discussion:

NAA result showed that copper and antimony and lead (lead by ICP-AES) are the indicator elements in most of the samples. It was found that abundance of lead WAS very significantly higher in some samples. It was revealed that barium could not be detected due to high-induced activity in the samples after neutron irradiation. Hence, presence of barium cannot be conclusive. Results confirmed that lead, antimony and copper are present in the backside of paper target. Analytical techniques can play vital role to analyse blackened bullet residue on paper or cloth target backed by polypaper coated thin film glass pane.

It is also revealed that using the same weapon and ammunition type, deposits vary from one ammunition to another. In our method we have so far tested four weapons i.e. .315"/8mm, AK47, .303" rifles and pistol where leakage or thickness of windowpanes is expected to be present. Barium and nickel could not be detected for any of the weapons used in these experiments. Amount of lead is more in the deposits of blackened bullet

residue obtained in an intermediate target. Table-III shows the considerable differences in the minimum and maximum values of the element detected.

### Conclusion:

Since the control dry filter paper samples and those on which the residue was collected were varying in weight, the concentration of the element in ppm as measured can be the basis of establishing the presence of a significant level of particular elements in the blackened bullet residue. The variation could be due to round to round variation not only in the priming composition but also in the extent of decomposition of the bullet residue when fired. No relation was found in the tests between the amount of barium or antimony, or lead and the number of shots. Many factors can play important roles in the amount of blackened bullet residue. Tests were made in order to establish the retention of the bullet residues on filter paper. Our findings demonstrated that bullet residues were not removed by cold water or wash by soap or detergent.

The study clearly established that the NAA/ICP-AES techniques are sufficient to detect the glass firing residue of all types of glass plate of manufactured windowpanes through the elements antimony, lead and to some extent zinc that a weapon has been fired. Despite the very miniscule amounts of materials which some of the deposit feature represents, most important elements were copper, lead, antimony and zinc. Significant amount of lead, antimony and Zinc (to some extent in some of the samples) were observed. The antimony observed could be naturally expected to arise from the antimony hardened found in bullet lead and also primers. Copper on the other hand, evidently arises from jacketed material of bullet jacket. The analytical data for the visible /invisible features of the complete bullet residue patterns were significant.

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**Table I**  
Firearm-Cartridge combinations used for firings:

Firearm	Cartridge	Bullet weight (gms)
.315 rifle, RFI, 1973, Sr.No. 94 AB 2832	8mm, soft nose, K.F., 01	15.35
.303 Rifle No. III*, S.No. F507FTS	.303, Ball, MK-7, K.F.	11.4
AK-47 Rifle Sr.No. AFN-1198, 1996	7.62x39mm, 322, 74	9.6
9mm Pistol, Browning MK-I, 1203	9mm, ball, auto, K.F.,	7.2

**Table II**  
Data on the Ballistics Characteristics

Sr.No.	Caliber of Bullet	Type jacketed/ soft nose/ unjacketed	Shape of Nose	Length in mm	Weight in grain	Diameters in mm
1	8mm	Soft nose	Round nose	31.48	236.6	8.103
2	.303"	Jacketed	Sharp pointed	32.24	174.4	7.889
3	7.62x39mm	Jacketed	Sharp pointed	29.42	146.5	7.719
4	9mm	Jacketed	Flat pointed	14.75	114.2	9.212

**TABLE-III:**  
**NAA SAMPLE RESULTS:**

Sr. No.	Description of the sample	Wt of the sample taken for analysis (gm)	Amounts in microgram (Concentration in ppm)					
			Ba	Cu	Sb	Pb	Zn	Ni
1	Blackened bullet residue obtained on filter paper fired by 8mm soft nose bullet when No film was kept on windowpane at the muzzle end	0.412	ND	2 (5)	322 (782)	15220 (36942)	11 (19)	ND
2	=do=	0.341	ND	3.7 (11)	315 (924)	5380 (15777)	4 (12)	ND
3	Blackened bullet residue obtained on filter paper fired by 8mm soft nose bullet when thin film was kept on windowpane at the muzzle end	0.195	ND	5.4 (28)	56 (287)	1540 (7897)	12 (62)	ND
4	=do=	0.179	ND	0.14 (0.8)	ND	0.2 (1)	7 (39)	ND
5	=do=	0.191	ND	0.6 (3)	8 (42)	142 (743)	7 (37)	ND
6	=do=	0.172	ND	0.4 (2)	11 (64)	254 (1477)	9 (52)	3.2 (19)
7	=do=	0.154	ND	2.5 (16)	104 (675)	1134 (7364)	4 (26)	ND
8	Blackened bullet residue	0.415	ND	2	92	2560	11	ND

	obtained on filter paper fired by 8mm soft nose bullet when thin film was kept on windowpane at away from the muzzle end			(5)	(222)	(6187)	(27)	
9	=do=	0.093	ND	0.3 (3)	13 (140)	396 (4258)	6 (65)	ND
10	Blackened bullet residue obtained on filter paper fired by 9mm jacketed bullet when No film was kept on windowpane at the muzzle end	0.044	ND	0.2 (5)	ND	7 (159)	7 (159)	ND
11	=do=	0.049	ND	0.2 (4)	ND	3 (61)	5 (102)	ND
12	Blackened bullet residue obtained on filter paper fired by 7.62x39mm jacketed bullet when thin film was kept on windowpane at the muzzle end	0.058	ND	0.4 (7)	14 (241)	3 (52)	7 (121)	ND
13	=do=	0.048	ND	4 (83)	19 (396)	71 (1479)	13 (271)	ND
14	Blackened bullet residue obtained on filter paper fired by 7.62x39mm jacketed bullet when thin film kept on windowpane at away from the muzzle end	0.045	ND	0.7 (16)	ND	8 (178)	12 (267)	ND
15	Blackened bullet residue obtained on filter paper fired by .303" caliber jacketed bullet when No film kept on windowpane at the muzzle end	0.147	ND	0.4 (3)	24 (163)	1950 (13265)	10 (68)	ND
16	=do=	0.092	ND	0.6 (7)	ND	8 (87)	9 (98)	ND
	Control samples of filter paper for all samples	0.126	ND	ND	ND	2 (16)	7 (56)	ND

**Table IV**  
**Production & Decay Properties of Nuclides Detected**

Sr. No.	Element	Target Isotope	Product nuclide	Half life	Gamma Energy (KeV)	Isotopic abundance (%)	Cross section (barn)
1.	Ba	Ba <sup>138</sup>	Ba <sup>139</sup>	1.38 hrs	166	71.66	35
2.	Cu	Cu <sup>63</sup>	Cu <sup>64</sup>	12.8 hrs	511 & 1345	69.1	4.5
3.	Sb	Sb <sup>121</sup> & Sb <sup>123</sup>	Sb <sup>122</sup> & Sb <sup>124</sup>	67.2 hrs 60.3days	564,692 602	57.25 42.8	6.2 3.45

**Figure 1.****Schematic diagram of radio chemical separation and ICP-AES for Ba, Cu, Sb, Pb, Ni and Zn.**