EXHIBIT 3

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Studies of the Trace Element Content of Bullet Lead and Jacket Material

This note concerns the characterization of bullets and bullet jacket material through their trace element content, a technique of potential utility when a bullet or bullet fragment is deformed so that the more conventional techniques are inapplicable. Among the trace element analysis techniques which have been already applied to this problem are atomic absorption spectrometry [*1*] and neutron activation analysis [*2*]—the latter technique being that employed in the present study. Results from these studies (for rather few trace elements) suggest that bullet lead compositions may be sufficiently constant for a given manufacturer and calibre for characterization to be possible. This is feasible despite the possibility that manufacturers supply lead to each other and the batch nature of the manufacturing process. In the recent work of Lukens et al [*2*], the elements Sb, Cu, and As were determined, but the measurement of other trace elements was impeded by the intense antimony radioactivity present in the irradiated specimens, resulting from activation of the substantial Sb content of bullet lead.

The present work employed Ge(Li) gamma-detectors of very much improved energy resolution, expected to facilitate resolution of the gamma radiations from various elements present. In addition, a relatively long irradiation at a neutron flux of 10^{14} n·cm^{-2}·s^{-1} was employed to permit more intensive activation of the elements present in lower concentrations. Further, a radiochemical separation of antimony radioactivity was carried out after the irradiation with a view to reducing the background against which the radiations from the minor trace elements would have to be measured. It was hoped that this would permit the measurement of many more elements in bullet lead than was possible with the techniques employed previously [*2*]. The desirability of this objective has been emphasized by Brunell et al [*1*] while determining four elements (Sb, Cu, Bi, and Ag) by atomic absorption spectrometry.

The composition of bullet jacket material has been studied by Omilon [*3*] by spark spectrography. The data obtained were semi-quantitative in nature, but indicated that a variation in the trace element composition might exist between bullet jackets from the same batch of ammunition. The present study, therefore, included an attempt to obtain preliminary quantitative data in the same field.

Experimental Procedure and Results

Sample Preparation and Irradiation Conditions

The bullet lead specimens were prepared for irradiation by a procedure designed to minimize surface contamination. Each bullet was sawn in half and a slice weighing 400 mg

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was cut from each half to supply duplicate samples. In the case of jacketed bullets, the bullet slices were placed in 5 ml of cold, concentrated nitric acid to dissolve away the jacket material; after the initial vigorous reaction had subsided, the slice was treated with a further portion of fresh concentrated nitric acid. This second treatment was observed to remove the remaining flecks of jacket material embedded in the lead. As a further precaution against possible surface contamination introduced by the sawing operation, the bullet slices were then etched further with 3 *N* HNO_3 heated to a temperature of 60°C. The bullet slices were finally washed three times with deionized distilled water, dried, and weighed. The total weight loss during the preparation procedure was typically 50 percent of the initial sample weight.

Bullet jacket samples were obtained by peeling the jacket from one bullet each of two varieties and dissolving 25 percent (by weight) of the jacket material with 3 *N* nitric acid at 60°C to remove any adhering lead which might be present.

The prepared specimens of bullet and jacket material were encapsulated separately in silica ampoules which had been washed in hot concentrated nitric acid and rinsed with distilled deionized water. Standard quantities of several elements including Sb, Ag, Cu, Cr, Zn, Au, and As were prepared for irradiation simultaneously with the specimens to be analyzed by pipetting known volumes of solutions of known concentration on to Whatman No. 42 ash-less filter paper, air drying, and encapsulation in silica ampoules.

Following a preliminary experiment involving an irradiation at the University of Washington (Seattle) reactor in a flux of 10^{12} $n \cdot cm^{-2} \cdot s^{-1}$, the high intensity irradiations were carried out in the N. R. U. reactor at Atomic Energy of Canada Limited, Chalk River, Ontario, at a neutron flux of 10^{14} $n \cdot cm^{-2} \cdot s^{-1}$ for a duration of 24 h; the standards were distributed spatially among the samples to be analyzed to reduce the effects of spatial variations of the neutron flux. Gamma radiation measurements on the irradiated samples began 72 h after the end of irradiation, following return of the irradiated samples to the laboratory.

Chemical Separation and Radiation Measurements

The method chosen to remove most of the radioantimony from the irradiated bullet material was the generation of stibine gas (SbH_3). This method was expected to provide reasonably good purification from antimony activities in a short time, while at the same time causing very little loss of most other elements present in the dissolved bullet solution. This latter aspect was important since it was intended to preserve the analysis as a multi-element operation without the necessity of numerous chemical yield determinations.

Of the trace elements expected to be present in bullet material, the ones expected to be affected by the generation and evolution of stibine gas from acid solutions treated with metallic zinc would be those elements such as arsenic and selenium which also produce volatile hydrides under such conditions. To study such effects prior to any chemical separation, the irradiated samples upon arrival in the laboratory were transferred to clean polyethylene vials, and a preliminary gamma analysis was performed via the techniques outlined below.

Following this measurement, each irradiated bullet sample was dissolved in 5 ml of 3 *N* HNO_3 at 60°C; 15 ml of concentrated HCl was then added and the whole solution evaporated to a minimum volume to remove excess nitric acid. The solution was then made up to known volume with concentrated HCl to dissolve completely the $PbCl_2$ produced.

The resulting solution was then heated to 100°C and added to 5 g of "mossy" zinc, resulting in the generation of H_2 gas carrying with it SbH_3 plus AsH_3 and H_2Se. The

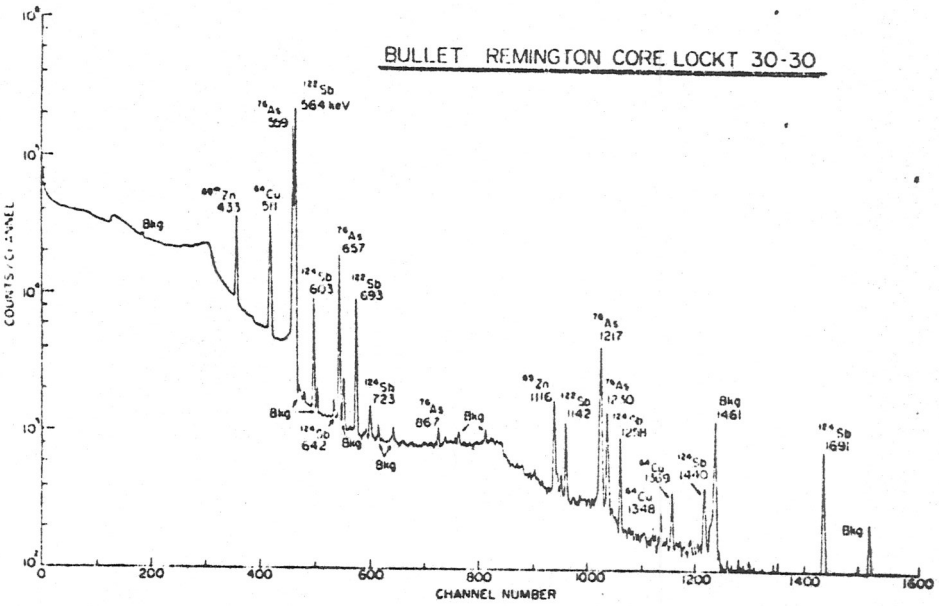

FIG. 1—*Gamma spectrum from irradiated bullet material following radiochemical Sb separation.*

evolved gases were passed through 10 percent lead acetate solution and then through 1.6 percent $HgCl_2$ solution to trap selenium, arsenic, and antimony activities. During the gas evolution three aliquots of 1 mg of Sb^{+3} carrier were added to the acid-zinc mixture to improve the removal of radioantimony. At the end of the evolution process, enough acid was added to dissolve completely the added zinc metal. The resulting clear solution was then encapsulated for radioassay.

Gamma radiation spectra were measured by means of a Ge(Li) detector[2] with an active volume of 25 cm^3 together with conventional electronic apparatus. The spectrometer system exhibited a resolution of 2.44 keV (FWHM) for a gamma energy of 609 keV. Figures 1 and 2 show two typical gamma spectra for different bullet specimens after radiochemical separation of the majority of the antimony activity.

The bullet jacket samples were measured by the same technique after transfer from the irradiation capsule to an unirradiated vial, but without any chemical treatment.

The spectra were analyzed by means of the computer program SAMPO [4]. Calibration of the spectrometer, both with respect to gamma energy and to detection efficiency, was achieved by means of a source of ^{226}Ra radioactivity and the associated daughter activities.

Results

The spectra measured prior to radiochemical separation of the antimony (not shown in the present figures) revealed gamma lines in high intensity arising from the decay of ^{122}Sb and ^{124}Sb radioactivity. Lesser intensities of gamma radiations due to the decay of ^{76}As, ^{65}Zn, and ^{64}Cu radioactivities could also be recognized.

In the spectra measured after Sb separation, of which Figs. 1 and 2 are typical, radioantimony gamma radiations are still dominant. Other gamma radiations, however, are

[2] Oak Ridge Technical Enterprises Corporation, Oak Ridge, Tennessee.

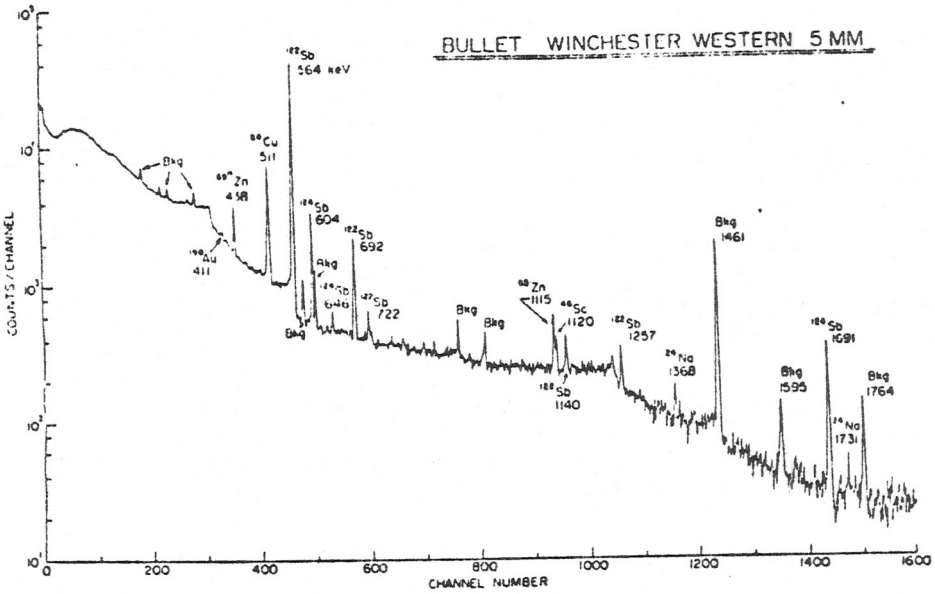

FIG. 2—*Gamma radiation spectrum from irradiated bullet material following radiochemical Sb separation.*

now observed in increased relative intensities as indicated in these figures. From a comparison of the gamma spectra measured before and after Sb separation, the efficiency of the Sb separation could be measured. It was found to vary from 98.9 percent to 99.9 percent and was nearly always close to the higher of these two figures.

From the relative gamma intensities measured from the irradiated samples and standards, the weights of the trace elements present in each sample and, hence, the concentrations of these elements in bullet materials were calculated. A similar calculation was performed for the bullet jacket material samples. The results are shown in Tables 1 and 2. The errors quoted are either the spread between replicate determinations or the total errors calculated from the known contributions from several sources (such as statistical and other uncertainties in radioactivity determinations, and errors in volume meaurements). The larger of the measured or calculated errors was used in each case.

Discussion

The technique used in this present work permitted the determination of the concentrations (or their upper limits) of eight elements in bullet lead material and four elements in bullet jacket material. Duplicate samples from the same bullet gave in each case results showing quite good agreement. It is clear, however, that for the several kinds of ammunition samples studied, there was a substantial variation in the trace element content between samples of different manufacture, and the different ammunition varieties could have been clearly distinguished from each other. The number of samples examined was, of course, too small to permit conclusions to be drawn regarding the feasibility of bullet characterization; nevertheless the results appear sufficiently promising to warrant extended studies of the variation of these trace elements throughout a wider selection of ammunition samples.

TABLE 1—*Element concentrations in bullet lead.*

Bullet Type	Sb, %	Au, ppm	Ag, ppm	As, ppm	Cr, ppm	Cu, ppm	Te, ppm[b]	Zn, ppm
CIL Dominion .30-30	1.7 ± 0.2	0.012 ± 0.001	35 ± 4	<120	<1.8	7.0 ± 2.5[a]	<9	<1.8
CIL Dominion .22 Long rifle	2.2 ± 0.3	0.016 ± 0.003	39 ± 7	<200	0.6 ± 0.2	52 ± 20	<10	<1.8
Winchester-Western 5 mm	.052 ± 0.004	0.023 ± 0.001	64 ± 42	<12	<0.8	33 ± 10[a]	<7	<1.5
Remington Core-Lockt .30-30	2.1 ± 0.5	0.093 ± 0.006	81 ± 5	490 ± 50	<0.4	19 ± 7[a]	28 ± 10	1.4 ± 0.4

[a] Uncertain due to possible contamination with jacket material.
[b] Measured through ^{131}I produced via $^{130}Te(n, \gamma)\ ^{131}Te \xrightarrow{\beta^-} {}^{131}I$, observed in acid solution remaining after SbH_3 evolution.

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TABLE 2—*Element concentrations in bullet jackets.*

Bullet Type	Zn, %	Sb, ppm	Au, ppm	Ag, ppm
Winchester-Western 5 mm	8.5 ± 0.1	2.4 ± 0.2	0.6 ± 0.1	34 ± 5
Remington Core-Lockt .30-30	5.8 ± 0.1	4.3 ± 0.4	1.0 ± 0.1	84 ± 8

In the case of bullet lead material, the problems posed by the overwhelming antimony radioactivity present in irradiated samples have not been overcome completely. A residue, after chemical separation, of 0.1 percent of the radioantimony initially present is still enough to provide an uncomfortably high background against which to measure many of the trace elements present. More efficient, although more tedious, chemical separation techniques are available and the upper limits quoted in this paper could probably be reduced if these were employed.

There is an equivalent problem in the case of the analysis of bullet jacket material by the present technique. The overwhelming zinc and copper activities impede the determination of other elements measured through radioactivities of short half-life.

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Bullet Residues

The Treasury Department has found that barium, copper, and antimony residues are left on leather, paper, and fabric by commercially manufactured bullets from the major producers. Tests are now underway to relate the quantities of trace elements found to other variables, such as the caliber, bullet shape, and manufacturer of the cartridge.

The Maryland State Police submitted for analysis a man's shoe with two irregular rips. The shoe had been removed from a burglary suspect who denied being present at the scene of the crime and also denied that the holes in his shoe had been made by a bullet. Analysts moistened a piece of filter paper with one percent nitric acid and rubbed it over the suspected entrance hole; they took similar swabs at other points on the shoe. A third swab was taken from the barrel of the gun employed. Figure 7 shows the levels of copper and antimony found on the shoe and in the gun barrel.

Origin of Sample	Copper (counts/minute in photopeak)	Antimony (counts/minute in photopeak)	Ratio (Antimony/Copper)
Around entrance hole in shoe	128,054	15,720	0.11
Other parts of shoe	894	not detectable	
Gun barrel	109,391	12,285	0.12
Blank (filter paper plus 1% nitric acid)	351	185	

Figure 7.

Metals

Determination of trace element composition of metal objects has been extremely useful in a large number of criminal case investigations undertaken by the Treasury Department. One note of caution is necessary, however, in the analysis of very small metal fragments that contain nonalloyable components. In such situations a heterogeneous condition exists. Nonalloyable components are mixed unevenly throughout the metal material, so that the quantitative variations in the trace element concentrations increases for these components as the sample size decreases. Thus the accurate comparison of sample origins involving very small metal fragments is more difficult than the comparison of large samples.

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Copper Alloys

In seizing an unregistered distillery, revenue agents collected two pieces of insulated copper wire from a fuse box. Similar wire was taken later from a pump found in a suspect's car. Analysis at the Treasury Department showed that the specimens contained trace quantities of molybdenum, chromium, gold, antimony, silver, zirconium, and iron in the same relative abundance.

Copper electrical wire collected from a pump in an illicit distillery was compared with wire found inside the suspect's residence and outside his house. Analytical results are shown in Figure 8.

Description	Copper	Gold	Silver	Cobalt
Wire from pump—A	100	1.46×10^{-4}	6.0×10^{-3}	0.02
Wire from pump—B	100	1.45×10^{-4}	6.0×10^{-3}	0.02
Wire near house	100	1.45×10^{-4}	6.3×10^{-3}	0.03
Wire from house	100	1.46×10^{-4}	6.3×10^{-3}	0.03

Figure 8. Percentage of Radionuclides Found in Copper Wires

In yet another investigation of an illegal distillery, neutron activation analysis proved that a copper still cap and two scraps of copper metal from the suspect's house had the same relative amounts of gold, cerium, silver, zinc, cobalt, and chromium.

Iron Alloys

A steel burner stand taken as evidence from an illegal distillery was analyzed by NAA, along with a portion of a steel barrel found in the suspect's home. Antimony and zirconium were present in each sample in the same ratios.

Another case brought to the Treasury Department laboratory concerned a violation of the National Firearms Act. An illegal length Hopkins-Allen 12-gauge shotgun was discovered by the authorities; its barrel had been sawed off to 16 inches and had been fitted with a cut down stock. Subsequent investigation uncovered a portion of a 12-gauge shotgun at a suspect's place of business. The Treasury Department was asked to determine if the barrel were once part of the sawed-off weapon in question. NAA of the metal taken near the cut area on each specimen showed that both contained iron, manganese, iridium, and cobalt in the same proportions. This evidence, along with physical measurements taken on both the gun and suspected gun barrel, indicated beyond reasonable doubt that the cut barrel had been removed from the shotgun in question.