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Lead Isotope Ratios in Lead Smears and Bullet Fragments and Application in Firearm Investigations

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ABSTRACT: This paper describes the use of lead isotopes ratios for an investigation of a homicide. Lead smears and bullet fragments recovered from a victim's clothing were compared with lead samples from two suspect bullets.

More than 90 bullets of the same caliber and produced by the same manufacturer as the suspect bullets were analyzed. The lead isotopic compositions were found homogeneous within individual bullets. Different bullets from the same ammunition box also showed indistinguishable lead isotope ratios. Bullets from ammunition boxes produced in 27 different months were analyzed. The results were compared with those obtained for bullets and bullet fragments from the homicide case. The high precision isotope ratio measurements could distinguish between lead from the homicide case and nearly all bullets from the ammunition boxes that were investigated. The isotope ratios for bullets from one of the ammunition boxes were indistinguishable from those for the homicide bullets. Some bullets of the same caliber as the suspect bullets but produced by different manufacturers were also analyzed by this technique. All these bullets showed clear differences in their lead isotope ratios.

KEYWORDS: criminalistics, lead isotope ratios, firearms investigations, bullet analysis, lead smears

In some firearm investigations, such as those involving "death by gunshot," the fatal bullet is not found or it is badly damaged and the identity of the responsible firearm is unknown. The traditional examination procedure, which involves comparing the striations on the evidence bullet with those on the test bullet fired from the suspect's gun, cannot be used to connect a weapon with the gunshot wounds. Nevertheless, two other possibilities exist to identify the ammunition or the firearm used:

1. X-ray photographs of the victim's clothing or body may reveal small bullet fragments that can be recovered for analysis. These fragments can be compared with lead material recovered from the ammunition used or from the suspect firearm.
2. For shorter firing ranges (generally less than about 50 cm) fragments of unburnt or partially burnt firearm propellant may be found on clothing close to the entrance of the bullet hole. The composition of propellant traces can be compared with that obtained from the suspect ammunition.

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Analysis of trace element composition of the bullet has proven useful for comparison of different bullets. Several analytical techniques have been used to determine trace metals in bullets [*1–5*]. For the examination of very small bullet fragments found in a victim's clothing, the amount of material is generally too small and also the risk of contamination (by blood, clothing material, etc.) makes elemental analysis less suitable.

Taylor et al. [*6*] used a scanning electron microscope (SEM) equipped with an X-ray microanalyzer (EDS) to locate and analyze the small lead fragments found in clothing at the entrance and exit wounds. They also examined cartridge casing and jacket materials found in clothing. The same analytical method was used to identify bullet particles in bone fragments [*7*]. The SEM/EDS analysis, however, exhibits low sensitivity (only antimony can be detected in addition to lead) and the method can only distinguish between some types of bullets. A more sensitive analytical method is proton-induced X-ray emission analysis (PIXE). This method was used to detect bullet residue (lead) in bone [*8*]. However, no attempts were made to measure trace elements in the bullet fragments by PIXE.

The method suitable for comparison of small amounts of lead is the determination of lead isotope ratios. Lead occurs in nature as a mixture of four stable isotopes (^{204}Pb, ^{206}Pb, ^{207}Pb and ^{208}Pb). Three of these isotopes, those with mass 206, 207 and 208, are the end products of the natural radioactive decay series starting with ^{238}U, ^{235}U, and ^{232}Th, respectively. There is a large variation in the relative abundance of these lead isotopes. Lead isotope ratios provide a means of determining the age and provenance of rocks and minerals [*9*].

Lead ores display a wide variation in their isotopic composition; this composition can vary even within one mine. According to information received from the Swedish manufacturer of metallic lead, the raw material for all the lead produced in Sweden is no longer taken from mines. Instead, different kinds of scrap lead is used. Bullet lead is thus produced from lead accumulators, cables, tubes, lead granulate from industries and partly from discarded bullets after series of melting and refining operations. Unwanted elements, like sulphur, are removed and antimony is added according to product specification. This means that we can expect random variations in the isotopic composition between different lots of manufactured lead.

The sensitivity and the precision of determination of lead isotope ratios make this an attractive method for forensic examination of bullet lead and lead fragments obtained from clothing. Reliable analyses with high precision can be carried out using samples containing less than 1 μg lead. The precision in the determination of various isotope ratios can be better than 0.1% [*10*]. The risk of contamination is restricted only to lead from sources other than a bullet. When working with extremely small quantities of lead, the sample preparation must be carried out in special lead-free environments. The contamination of lead smears on clothing by the clothing itself, blood, etc., presents a much less serious problem compared with trace metal measurements, because almost 100% of the material examined is composed of lead.

The use of lead isotope ratios in gunshot examinations was first reported by Stupian [*11*]. The results obtained for a limited number of samples (six pairs of bullets) were presented. The uncertainties in the results were not given, but were presumably large, since two bullets from the same ammunition box differed markedly in their isotopic composition.

Keisch and Callahan [*12*] reported the determination of lead isotope ratios in 14 bullets with high precision measurements. These workers could distinguish between all except two pairs of bullets that were produced by the same manufacturers but were of different caliber. Furthermore, this same technique was used to analyze five primers and nine hand swabs for possible detection of gunshot residues and the experimental results were related to the weapon when test-fired. This application may be questioned because of a serious risk of contamination of the shooting hand by lead from other sources. Keisch and Callahan [12] suggested, however, that lead isotopic composition should prove valuable for analysis of lead material scraped from a bone that had been grazed by a bullet or obtained from inside a gun barrel.

Another study on the use of stable isotopes in forensic science has recently appeared [13]. The authors, who analyzed eight samples of scrap ammunition, maintain that the isotope method should be applied whenever conventional chemical methods fail. They also present arguments in favor of this method over conventional analytical techniques.

In this study, the measurements of lead isotope ratios have been applied to a homicide case. One person was killed and another slightly wounded by an unknown firearm. The murderer fired two shots, one at each of the victims, and escaped from the scene of the crime. Neither the weapon nor the cartridge cases were found. After a long search, two .357 Magnum bullets were found close to the scene of the crime. The bullets were identified as coming from Winchester Western Metal Piercing ammunition, with the front part of the lead bullet covered by a thin hood of copper alloy. The police were surprised by the appearance of the bullets (little deformation) and the place where they were found and wondered if these really were the right ones. The bullets and the clothes from both victims were sent to our laboratory. No blood, tissue material or fibers were observed on the bullets. The gunshot residue particles found on the victim's clothing had a composition that agreed with that of the primer from Winchester ammunition (the presence of aluminum in addition to lead, barium and antimony). The bullets are expected to leave many traces—lead fragments and smears—when passing through a body and clothing. We decided to recover possible traces of lead on the clothing and compare the lead isotope ratios with those of the bullets. In connection with this homicide case we performed a series of lead isotope measurements on about 100 bullets. This investigation served two purposes. First, we wanted to evaluate the potential of isotope ratio measurements using more experimental data than in previous reports. Second, we wanted to help the police in their efforts to find the ammunition box or the weapon that the murderer had used.

Materials and Methods

X-ray Photography

Radiographs of the clothing were obtained on a Hewlett-Packard 43805 N X-ray system (Faxitron series). The X-ray tube was operated at 30 kV. Polaroid film, type 55 P/N was used.

The radiograph of the T-shirt of the slightly wounded victim (B) is shown in Fig. 1. The dark arrows were put on the clothing by the police to indicate where the bullet touched the persons back. Several radiodense smears are seen in the photo at the area where the bullet passed through. Small samples from these smears were picked up and analyzed by X-ray microanalysis in a SEM. The analysis showed the presence of lead and small amounts of antimony in the smears.

The murdered person's (A) clothing was X-ray photographed at the entrance and exit wounds. The radiographs showed the presence of several radiodense fragments, particularly at the exit wound. Figure 2 shows the radiograph of the victim's shirt at the exit wound. The small dark fragments in the figure consisted of lead with a small amount of antimony.

Lead Samples

All the bullets investigated in this study were of the same caliber (.357 Magnum). The projectiles were removed mechanically from the cartridges. The lead samples, weighing about 5 mg, were taken from the backside of the bullets if not otherwise stated. The

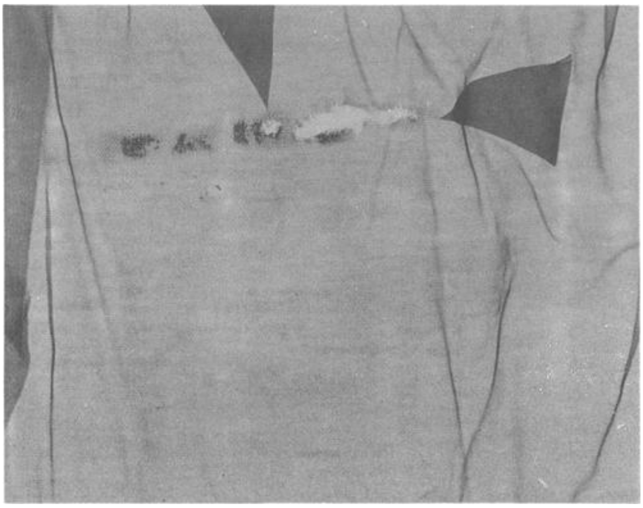

FIG. 1—*Radiograph of the clothing (T-shirt) of the slightly wounded victim. The bullet touched the victim's back.*

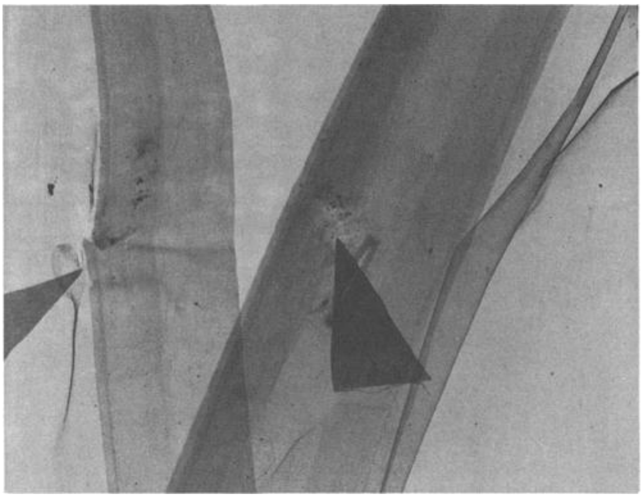

FIG. 2—*Radiograph of the clothing (shirt) of the murdered victim at the exit wound. The dark arrows were put on the clothing by the police to indicate the damage to the clothing caused by the bullet. The position of the different areas of the shirt in the radiograph is not exactly the same as on the body.*

samples from the evidence bullets were removed carefully not to destroy possible striation marks.

The lead fragments were carefully removed from the clothes by tweezers under a light microscope. The weight of the largest fragment recovered from victim A's clothes was less than 1 mg. The largest fragment was mechanically cleaned (under a light microscope). The lead smears from victim B's clothes were cut out. The fragments and the smears were analyzed by X-ray microanalysis in a SEM. The presence of small amounts of antimony in the fragments indicated that these originated from a bullet. The amount of antimony corresponded to that in bullet lead. Another origin of these traces of lead, like primer residue particles, is very improbable. The crime was committed during winter and both victims wore several additional layers of clothing, winter coats, etc. The lead fragments seen in Fig. 2 were found in the exit wound, after the bullet had passed through the body, on clothing closest to the body. The size and the weight of these fragments is also too high compared with those expected for primer residue particles. In addition, no aluminum and barium were detected in the fragments and smears. The primer of the actual ammunition consisted of Pb, Ba, Al and Sb.

Sample Preparation and Isotopic Analysis

The sample preparations and mass spectrometrical analyses were done at the Laboratory for Isotope Geology, Swedish Museum of Natural History in Stockholm, over a period of several years. During the course of the study, the preparation and analytical techniques were gradually refined.

To avoid any significant lead contamination during sample preparation, the work was carried out in a clean-air, lead-free laboratory normally used for low-contamination preparation of geological samples, using specially distilled high-purity chemicals. Bullet lead and lead fragments were dissolved overnight in 2-N nitric acid at about 60°C. Lead smears on clothing were leached using dilute HNO_3. Small aliquots of the solutions were diluted to a Pb concentrations of about 6 ppm in 0.03 N nitric acid. Initially, a more concentrated solution was used, but the presence of Sb, which commonly occurs in bullet lead, disturbed the following purification step. The purification was done by electrolytical deposition of the Pb, using a voltage of 1.9 V between two Pt electrodes inserted into the solution. The cathode was vibrated vigorously during the electrolysis to remove the H_2 gas bubbles that form on this electrode. Under these conditions, Pb is selectively deposited as PbO_2 on the anode while other elements remain in solution. After a few minutes, PbO_2 became visible as a thin brown coating on the anode. The PbO_2 was dissolved with a small drop of dilute HNO_3 + H_2O_2 solution, and mounted on a Re filament for mass spectrometrical analysis.

Procedures for high precision analysis of isotopic ratios are described in the literature [*10*]. The isotopic measurements for this study were made in static mode on a Finnegan MAT 261 mass spectrometer equipped with a 5-cup multicollector. Accuracy and reproducibility of the analytical results were regularly checked against the NBS common lead standard 981. The instrumental fractionation in the mass spectrometer between heavier and lighter Pb isotopes can vary with several per mil/a.m.u. depending on the exact analytical conditions. The routines for controlling this fractionation were improved during the study, and this contributed to a better analytical precision in the final measurements.

Results and Discussion

The Homicide Case

The comparison between lead isotope ratios for the two evidence bullets and those for lead fragments and smears from the two victim's clothing is shown in Table 1. The three ratios represent a set that completely defines the isotopic composition of the samples. The uncertainties represent two standard deviations. The four lead samples in Table 1 have indistinguishable isotopic compositions. We conclude that there is nothing to indicate that the questioned bullets are not those used in the shooting.

TABLE 1—*Lead isotope ratios in the bullets, lead fragments and lead smears from a homicide case.*

Sample description	Isotope ratios $^{206}Pb/^{204}Pb$	 $^{207}Pb/^{204}Pb$	 $^{208}Pb/^{204}Pb$
Bullet A	19.15 ± 0.08	15.69 ± 0.08	38.50 ± 0.15
Bullet B	19.11 ± 0.04	15.64 ± 0.04	38.45 ± 0.05
Lead fragment, victim A	19.06 ± 0.03	15.64 ± 0.02	38.41 ± 0.03
Lead smears, victim B	19.09 ± 0.03	15.62 ± 0.03	38.38 ± 0.04

Isotope Ratios for .357 Magnum Bullets from Various Manufacturers

Seven bullets of the same caliber as the suspected bullets A and B (.357 Magnum) were analyzed. The bullets were taken from the reference collection of the Swedish National Laboratory of Forensic Science. The manufacturers of the different bullets were known but not the time of production. The lead isotope ratios obtained for these bullets are presented in Table 2. All the bullets were easily distinguishable. Figure 3 illustrates the results from Table 2. The mean values of lead isotope ratios obtained for the evidence bullets A and B are included in this figure. The range of isotope ratios determined in this work is very similar to those obtained for bullets of different calibers and manufacturers by Keisch and Callahan [*12*]. The approximate uncertainties shown as squares in Fig. 3 indicate a good possibility to differentiate between bullets with this method.

Isotope Ratios for Winchester .357 Magnum Metal Piercing Bullets

In an effort to find the ammunition box that the murderer used, we concentrated our investigations on .357 Magnum Winchester ammunition.

Lead Samples from the Same Bullet—The homogeneity of bullet lead in terms of isotope ratios obtained for samples taken from different parts of the same bullet was tested. Two or three samples were collected from each of three different bullets. The results are presented in Table 3. The standard deviations observed for bullet 1 are relatively high, resulting from technical problems in our initial measurements, as described above. The precision in the other determinations is typical for high precision methods of measuring isotope ratios. The high reproducibility of the method is also evident because the samples taken from various parts of bullet 2 and 3 were subjected to analysis on different occasions.

Our results support the assumption that lead in the whole bullet exhibits the same isotopic composition. Lead, in contrast to other elements, represents almost 100% of the

TABLE 2—*Lead isotope ratios in samples of .357 Magnum bullets from various manufacturers.*

Sample (manufacturer)	Isotope ratios $^{207}Pb/^{206}Pb$ (± 0.001)	 $^{208}Pb/^{206}Pb$ (± 0.003)
Geco	0.889	2.139
Hirtenberger	0.859	2.098
Winchester	0.854	2.106
Lapua	0.838	2.052
Norma	0.830	2.029
Federal	0.804	1.987
Speer	0.764	1.893

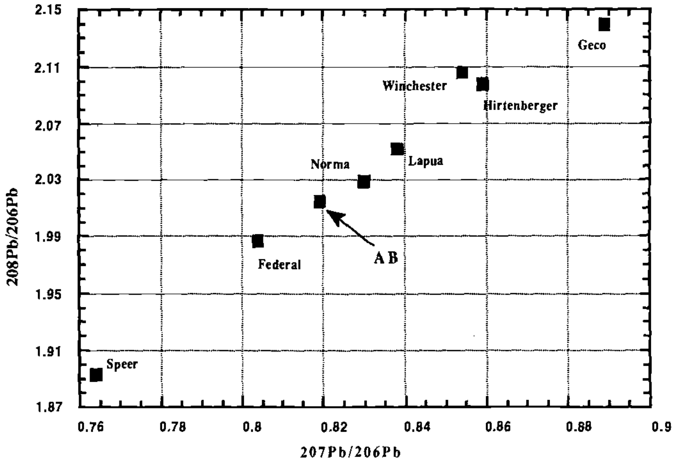

FIG. 3—*Lead isotope ratios in .357 Magnum bullets from various manufacturers. The data and the bullet numbers are listed in Table 2. The squares illustrate the approximate uncertainties in the measurements. The mean value of the isotope ratios for bullets A and B is also shown in the figure (AB).*

TABLE 3—*Lead isotope measurements on samples taken from different parts of the same bullet.*

Sample	Part	Isotope ratios	
		^{207}Pb/^{206}Pb	^{208}Pb/^{206}Pb
Bullet 1	I	0.856 ± 0.002	2.109 ± 0.006
	II	0.856 ± 0.002	2.107 ± 0.006
	III	0.855 ± 0.002	2.101 ± 0.006
Bullet 2	I	0.8230 ± 0.001	2.0235 ± 0.003
	II	0.8231 ± 0.001	2.0212 ± 0.003
Bullet 3	I	0.8183 ± 0.001	2.0116 ± 0.003
	II	0.8188 ± 0.001	2.0126 ± 0.003

bullet material. Accordingly, the isotopic composition should be the same as the original lead melt from which a large number of bullets is produced. Literature data concerning trace elemental composition in lead reveal considerable fluctuations for some elements across the same bullet [*2*]. Clearly, more investigations are needed to verify this.

Bullets from the Same Ammunition Box—We performed lead isotope measurements on two bullets from the same ammunition box to detect possible variations in lead isotopic composition within the same box. The results obtained for 13 pairs of bullets are shown in Table 4, for both high and lower precision experiments. The isotope ratios for bullets from the same box agreed within the experimental errors. This result is important for the practical use of lead isotope measurements in forensic firearm investigation. If unspent bullets are found in a suspect's possession, their isotopic composition may be compared with that of evidence bullets. Identical isotope ratios can be taken to indicate that the bullets may have the same origin; they may have come from the same box or lot. There is, however, always some chance of finding bullets from two production lots in the same ammunition box. But this chance is presumably very low because the production batches of bullet lead are large. We cannot estimate this probability unless we have exact infor-

TABLE 4—*Lead isotope ratios in bullets from various boxes of Winchester .357 Magnum Metal Piercing ammunition.*

Sample	Bullet no.	Isotope ratios	
		$^{207}Pb/^{206}Pb$	$^{208}Pb/^{206}Pb$
Box 1	1	0.827 ± 0.002	2.030 ± 0.006
	2	0.827 ± 0.002	2.028 ± 0.006
Box 2	1	0.815 ± 0.002	2.008 ± 0.006
	2	0.813 ± 0.002	2.004 ± 0.006
Box 3	1	0.824 ± 0.002	2.024 ± 0.006
	2	0.823 ± 0.002	2.023 ± 0.006
Box 4	1	0.813 ± 0.002	2.003 ± 0.006
	2	0.814 ± 0.002	2.004 ± 0.006
Box 5	1	0.823 ± 0.002	2.021 ± 0.006
	2	0.824 ± 0.002	2.021 ± 0.006
Box 6	1	0.816 ± 0.002	2.009 ± 0.006
	2	0.815 ± 0.002	2.007 ± 0.006
Box 7	1	0.822 ± 0.002	2.019 ± 0.006
	2	0.823 ± 0.002	2.022 ± 0.006
Box 8	1	0.8224 ± 0.001	2.0177 ± 0.003
	2	0.8212 ± 0.001	2.0181 ± 0.003
Box 9	1	0.8143 ± 0.001	2.0028 ± 0.003
	2	0.8143 ± 0.001	2.0029 ± 0.003
Box 10	1	0.8172 ± 0.001	2.0112 ± 0.003
	2	0.8168 ± 0.001	2.0093 ± 0.003
Box 11	1	0.8151 ± 0.001	2.0069 ± 0.003
	2	0.8137 ± 0.001	2.0029 ± 0.003
Box 12	1	0.8234 ± 0.001	2.0236 ± 0.003
	2	0.8231 ± 0.001	2.0212 ± 0.003
Box 13	1	0.8229 ± 0.001	2.0196 ± 0.003
	2	0.8229 ± 0.001	2.0196 ± 0.003

mation about the production habits of various manufacturers of cartridges. Many manufacturers stockpile lead bullets in a bin before they are assembled to produce the cartridges of a particular production lot. Thus, there is not a direct correlation between the production lots of the bullets and those of the cartridges themselves.

Bullets from Different Production Lots—The isotope ratio measurements were carried out on .357 Magnum bullets from a number of ammunition boxes manufactured by Winchester. More than 90 bullets were analyzed in this study, taken mostly from boxes purchased at various ammunition shops in Europe. The time of production is known from the code on the boxes. There was no possibility of obtaining ammunition boxes from selected production lots. The boxes were supplied to us by the police and the distribution in the time of production is random. A total of 43 ammunition boxes were supplied to our laboratory. Some of the boxes were of older date (produced during the 1960s), the majority was produced during the period between 1976 and 1982. The production of this specific kind of ammunition was stopped in 1982.

Our collection of ammunition boxes manufactured by Winchester contains boxes produced shortly after each other and also boxes produced several months apart. The number of ammunition boxes manufactured within the same month ranged from one to six. The determinations of lead isotope ratios could not distinguish between all samples from different ammunition boxes. The measurements could be used to divide the bullets into several groups. Not only the bullets produced the same day but also those produced the same month showed indistinguishable lead isotope ratios. Some results obtained for bullets produced within a few days are presented in Table 5.

To present our results on more than 90 different bullets in a diagram, we assumed the same isotopic composition for bullets produced the same month (or, more exactly, within 16 days in our actual collection). We cannot confirm the relevance of this assumption because the details of the production and the production capacity of the Winchester company is not available to us. With this assumption, the mean values and the mean standard deviations were calculated for each month's production for the material in our collection. The results are depicted in Fig. 4. This figure corresponds to an expansion of the middle part of Fig. 3. The circles in Fig. 4 illustrate roughly the uncertainties in the

TABLE 5—*Lead isotope ratios in Winchester bullets produced within several days.*

			Isotope ratios	
Year of production	Month of production	Day of production	^{207}Pb/^{206}Pb (±0.001)	^{208}Pb/^{206}Pb (±0.003)
1979	April	02	0.8144	2.0049
		05	0.8143	2.0029
		07	0.8147	2.0054
1980	January	02	0.8228	2.0192
		09[a]	0.8225	2.0205
		09[a]	0.8223	2.0182
		09[a]	0.821[b]	2.018[b]
		09[a]	0.8215	2.0190
		10	0.8218	2.0182

[a] Different ammunition boxes produced the same day.

[b] The measurement with lower precision (2 × standard deviation ±0.002 and ±0.006 respectively).

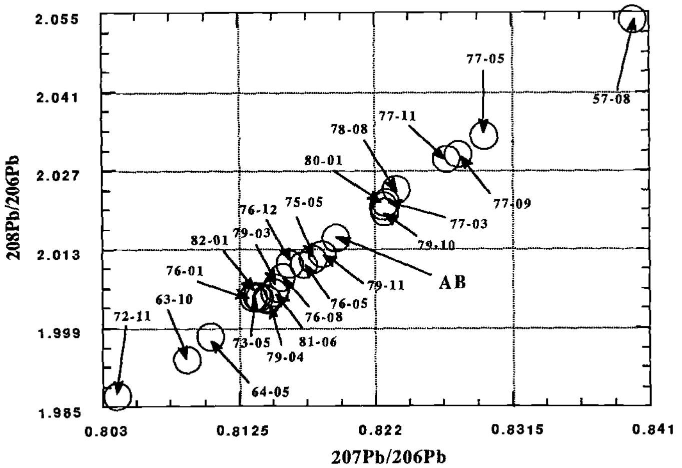

FIG. 4—*Lead isotope ratios in .357 Magnum Metal Piercing bullets manufactured by Winchester. The figure shows the mean values calculated for different months of production (79-03 means March 1979, etc.). The uncertainties in the mean values are shown roughly as circles and are represented by the uncertainties in single measurements. AB indicates the mean value obtained for the bullets A and B.*

mean values for each group of ammunition boxes. These uncertainties were put equal to those for single measurements of lead isotope ratios. The month of production for the various ammunition boxes is indicated in this figure (79/03 means March 1979, etc.). Only 23 of the 27 months of total production are represented in Fig. 4. The remaining bullets showed lead isotope ratios far outside this range.

The mean value of the isotope ratios measured for bullets A and B are also shown in Fig. 4. One of the mean values presented in Fig. 4 overlap within the limits of uncertainty with the bullets A and B. All the other samples—bullets from 26 different months of production—show an isotopic composition that is distinguishable from that of the evidence bullets.

The lead isotope ratios do not seem to have a simple correlation with the time of production. The values change randomly with time. Several examples of boxes manufactured at widely separated points in time having indistinguishable lead isotope ratios are apparent from an examination of this figure. It is interesting to note that the isotope ratios for boxes produced at two subsequent months overlap, falling within the same limit of uncertainties (79/03 and 79/04). This may be an accident or an indication that the lead smelts used in manufacturing Winchester ammunition are very large. Selected boxes produced within two months showed close agreement (77/09 and 77/11) or large difference (77/03 and 77/05). The material is too limited to make conclusions about the variations of isotope ratios with the time of production for Winchester ammunition.

Conclusions

The measurement of lead isotope ratios has good potential for distinguishing between bullets. Bullets produced by different manufacturers generally show clear differences in the lead isotopic composition (see Fig. 3 and Refs *11* and *12*). Bullets of the same caliber and produced by the same manufacturer (within several years) show less variation in the lead isotope composition. Nevertheless, these variations are of such a magnitude that this method can distinguish between many different lots produced by the same manufacturer. High precision and high accuracy measurements of the lead isotope ratios are needed to achieve reliable results. The results of this study of .357 Magnum ammunition produced by Winchester (shown in Fig. 4) confirm this. Only the bullets produced in 1 of 27 different months of production showed an isotopic composition indistinguishable from that obtained for lead in the evidence bullets A and B. From an examination of Fig. 4 it can be seen that for bullets with other lead isotope ratios the discrimination could be quite a bit better or could be worse.

The advantage of this method is its potential to analyze small amounts of sample without loss of accuracy. Thus small lead fragments or smears found in a victim's clothing can be analyzed and compared with lead from a gun barrel or in ammunition found in the suspect's possession. This method is also less sensitive to contamination than the traditional trace element analysis, since lead is the major element in bullets.

When a sufficient amount of material is available, the measurements of lead isotope ratios can be combined with other methods such as trace element analysis. The combination of these two independent methods might increase the possibility of differentiating between lead in bullets.

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