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March 15, 1967

To: All Members of the Lead Program Review Committee
All Members of the Lead Environmental Health Committee

Re: "Isotope Studies of Ancient Lead"

We have secured for you a copy of an article from the American Journal of Archeology, which article is attached. You will note that the technique of measuring isotopic ratios has been expanded here to a new use and came out eminently successful. Besides learning where a lead source originated the technique also positively excludes certain other sources which are questionable in origin.

There is also an interesting discussion of the possible pitfalls and precautions necessary in using the technique of isotopic ratio measurements. Dr. Wampler, one of the co-authors, is a special consultant for our contractor Isotopes, Inc.

Very truly yours,

Don C. Fowler
Project Manager

DCF:sw
attachment

American Journal of Archeology January 1967

Isotope Studies of Ancient Lead

ROBERT H. BRILL AND J. M. WAMPLER

Most archaeologists are familiar with the term *isotope* from their knowledge of radiocarbon dating. Isotopes are simply atoms of the same chemical element which have slightly different masses. Lead, for example, has four stable isotopes, designated by the symbols Pb^{204}, Pb^{206}, Pb^{207}, and Pb^{208}, which represent atoms weighing, respectively, 204, 206, 207 and 208 atomic mass units.

For the great majority of chemical elements, the relative proportions of their isotopes are the same regardless of where the elements are found in the earth's crust. For instance, copper in natural minerals always contains the same proportions of Cu^{63} and Cu^{65}; and silver always contains the same proportions of Ag^{107} and Ag^{109}. Lead, however, is unusual, in that the relative proportions of its isotopes vary somewhat among ores occurring in different geographical areas. These variations reflect differences in the geological ages of the ore deposits and stem from the fact that Pb^{206}, Pb^{207} and Pb^{208} are formed as end products of the radioactive decay series of uranium and thorium.

The isotope composition[1] of a specimen of lead can be determined with a mass spectrometer, by techniques which consume only very minute samples. Over the past thirty years many hundreds of such determinations have been made for leads taken from naturally occurring galena ores (lead sulfide). These are very useful for interpreting the geological histories of ore deposits and the rocks with which they are associated.[2] A survey of such data in the geochemical literature a few years ago suggested to one of the authors (BRILL) that the differences between the isotope compositions of lead in galena ores should be sufficient to allow one to distinguish between leads mined in Greece, England, and Spain. This in turn suggested that measurements of the isotopic compositions of archaeological objects made of lead would be of help in tracing probable locations of the mines from which the leads could have been taken. Therefore, we have collected a group of 230 samples, consisting of 70 specimens of ores from ancient mining areas and 160 samples of lead from archaeological objects. Measurements of the isotopic compositions of 60 of these samples were made during the period 1963–1965 at the Chemistry Department of Brookhaven National Laboratory using a thermal-emission mass spectrometer.[3,4]

The measurements made on these samples are only the first stage of this study. The findings are being reported now because it is likely to be a few years before the remaining work can be completed, and we hope that this publication might aid in the collection of additional samples.

This stage of the work was limited to two objectives. The first was to make a survey of ancient leads from a deliberately broad sampling of places, periods, and materials, in order to see what range of isotope compositions exists and what sort of groupings might emerge. The second objective was to confirm experimentally that the isotopic compositions of archaeological objects known to have been made from lead mined in specific areas could be matched to those of ores from these same areas.

In brief, the results of these early experiments have been very encouraging. They have shown that the isotope compositions of the archaeological objects do vary over a considerable range, but that many of them fall into one of three definite groups. These same groups also include galena ores from three of the best known ancient lead-mining areas, Laurion, Southern Spain, and Roman Britain.[5]

[1] By isotope compositions we mean the percentage compositions of a lead sample in terms of the four isotopes. Isotope ratios refer to the relative proportions of any two isotopes.

[2] R. D. Russell and R. M. Farquhar, *Lead Isotopes in Geology* (New York, Interscience, 1960). See especially pp. 201, 202.

[3] The experimental part of this work, monitored by Dr. Wampler, was performed under the auspices of the United States Atomic Energy Commission.

[4] A preliminary report on this project was presented at the Sixty-ninth General Meeting of the Archaeological Institute of America in Boston in December 1966. *AJA* 69 (1965) 165, 166.

[5] Although references to ancient lead mines are numerous, we have selected a few which were particularly useful in connection with this study: O. Davies, *Roman Mines in Europe* (Oxford, Clarendon Press, 1935); B. J. Parker, *Metallurgy in Antiquity* (London, B. J. Brill, 1950); R. F. Tylecote, *Metallurgy in Archaeology* (London, Edward Arnold Ltd., 1962); W. Greenwell, "The Early Metallurgy of Lead and Silver (Part I. Lead)," *Archaeologia* 37 (1901) 330–360; R. Archib-

Moreover, the isotopic compositions of several objects made of lead known to have come from specific mining areas were found to match those of ores from the same areas. As was anticipated, a number of samples fell between the groups or near the borderlines, which are not as yet well defined. Because the experiments were designed to reveal the broad picture rather than to define accurately any one area, definite and useful conclusions about all of the samples studied cannot be expected at present. The final interpretation of the results for many of these samples must be reserved until additional leads and ores have been studied, and the groupings and relationships can be established more firmly.

In order to explain the findings in as clear a way as possible, the data are presented in a series of four graphs. Before discussing the results, however, it will be helpful to outline how the graphs were constructed and how they are to be read.

The mass spectrometric measurements provide three experimentally independent ratios which represent the isotopic abundances relative to one another.[6] The data are plotted in figs. 1–4 as the ratios Pb^{206}/Pb^{204} vs. Pb^{208}/Pb^{204} and Pb^{207}/Pb^{204} vs. Pb^{206}/Pb^{204}. To save space two separate graphs are plotted in each illustration. In each case the upper series is the Pb^{208}/Pb^{204} data and the lower is the Pb^{207}/Pb^{204} data. The points on the upper series are plotted against the scales to the left of the graphs and those on the lower series against the scales to the right. The horizontal scale at the bottom, showing Pb^{206}/Pb^{204} ratios, is common to both plots. The points are numbered according to the catalogue of samples at the end of the article. When comparing two leads, the data must be in agreement on both of these plots if the leads are to be considered isotopically similar.[7]

Fig. 1 shows the range of experimental values determined for a selection of fourteen galena ores. Since later mining operations have obliterated all traces of many of the ancient mines it is very difficult to obtain samples which can definitely be said to be representative of ancient workings. In most cases it is necessary to study ores from nearby regions worked more recently and assume that they are representative of ores from the ancient mines. As will be seen from the results presented below, this assumption seems to be generally valid.

The group of six ores toward the right of the graph came from Laurion, Turkey, Iran, and Italy. The differences between the ores from Laurion (77) and those from Turkey and Italy (85, 113 and 193) are greater than the estimated analytical error, so there are some significant variations among ores from these regions. However, the analytical uncertainty does not permit us to distinguish between the Iranian ores (111 and 112) and that from Laurion, even though it is unlikely that any of these samples are truly identical in isotopic composition. Three of the four points from the central group are ores from England, while the fourth (118) is from northeastern Turkey. This is an excellent illustration of the way in which ores from widely separated geographical regions can be indistinguishable as far as their isotopic compositions are concerned, and serves as a clear warning that one must use considerable caution in interpreting this kind of data. The same warning is inherent in the plots of points 83 and 32. These are isotopically similar (except for a possible difference in Pb^{206}/Pb^{204}), but one comes from Spain and the other from Wales. A further complication, the converse of that above, is seen in the series 136, 137, 139 and 175. Although these leads are all from the Eastern Desert in Egypt (except for 175 which is from Saudi Arabia), they are of widely divergent isotopic compositions. These leads came from ores either emplaced in or derived from rocks of the Precambrian Era, the earliest of the geological eras. Precambrian rocks occur on both sides of the Red Sea, and possibly in

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ium, *Les Mines du Laurion dans l'Antiquité* (Paris 1897). References to more specific topics are included in the catalogue section.

[6] The instrument used was a Consolidated thermal emission mass spectrometer, Model 302. Samples were introduced either in the form of precipitated lead sulfide or by reduction of a lead oxide mixture. A full description of the experimental procedures, an error analysis, and the corrections factors applied to the raw results will be published in the near future in an appropriate journal, together with geological information on the lead ore samples.

[7] On each plot an ellipse has been drawn showing our estimate of the analytical error limits for the individual points; that is to say, the true isotopic ratios for a particular sample are expected to fall within the limits of uncertainty indicated by the experimental values. It will be noted that the Pb^{206}/Pb^{204} ratios are measured with greatest accuracy and the Pb^{208}/Pb^{204} ratios with least accuracy.

The ellipses are tilted because isotopic fractionation may occur during the mass spectrometric analysis. This effect produces correlations which are strongly manifested along diagonal axes. The variation among the samples from Greece, and part of that among the samples from England, is thought to be due to this effect. (See also footnote 20.)

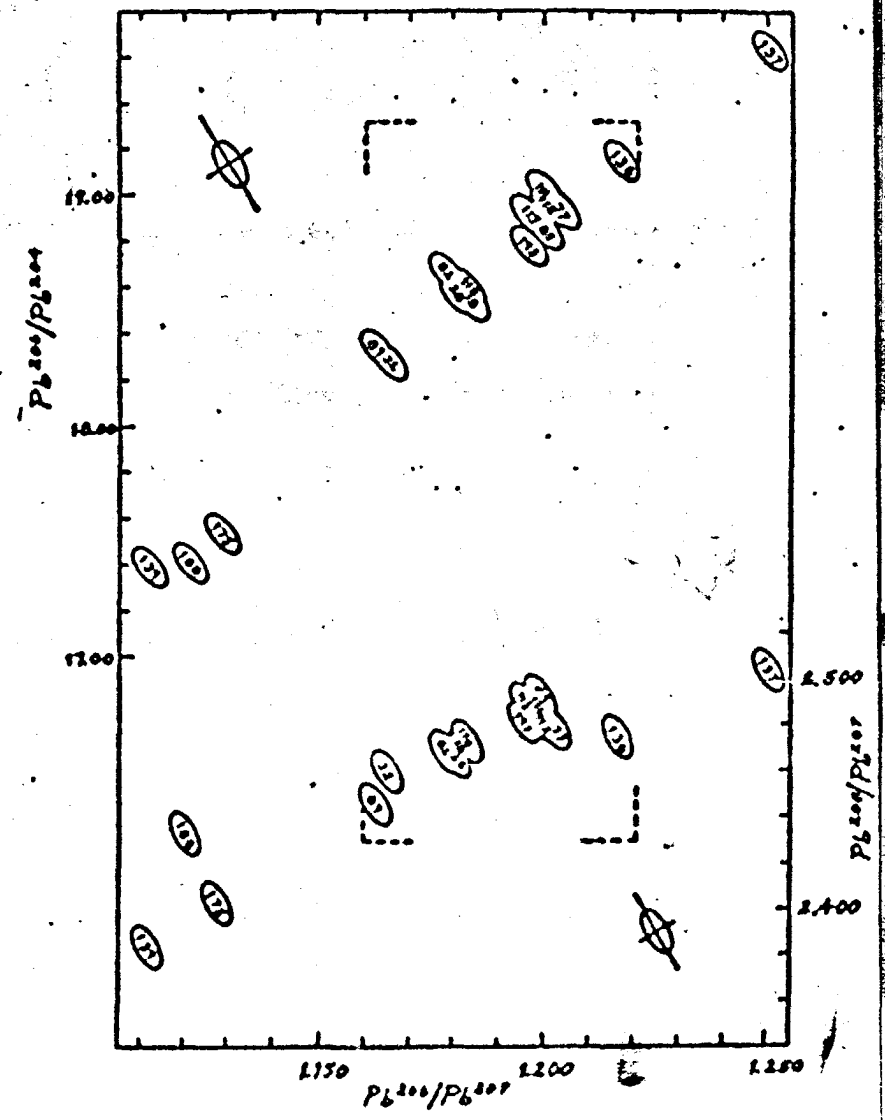
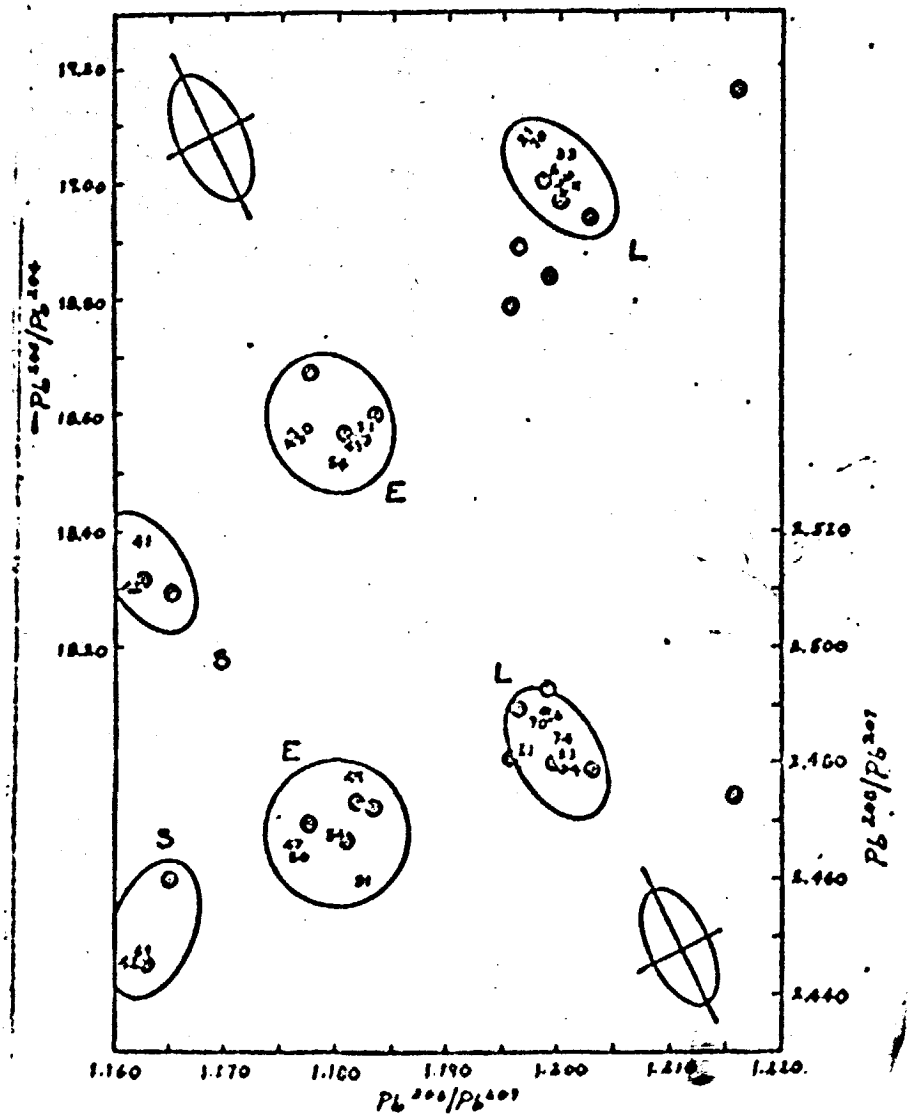
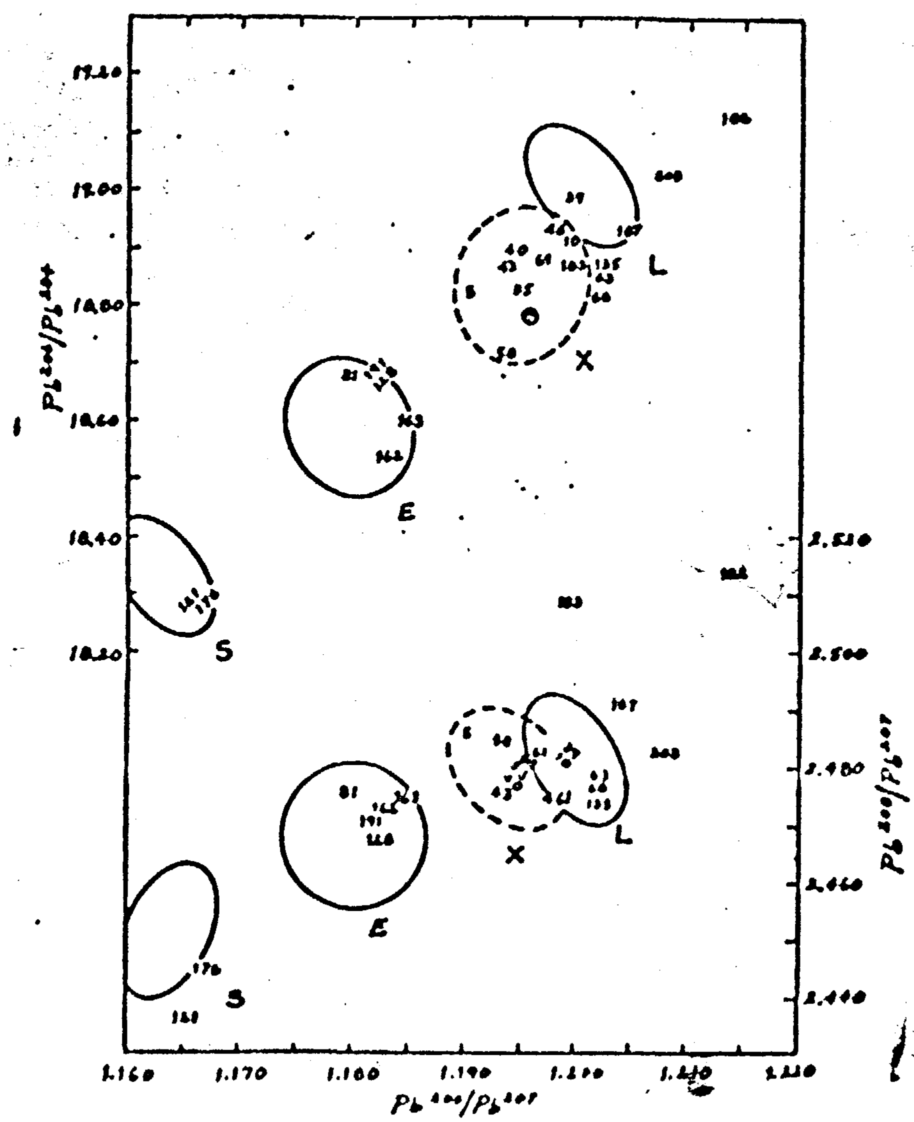


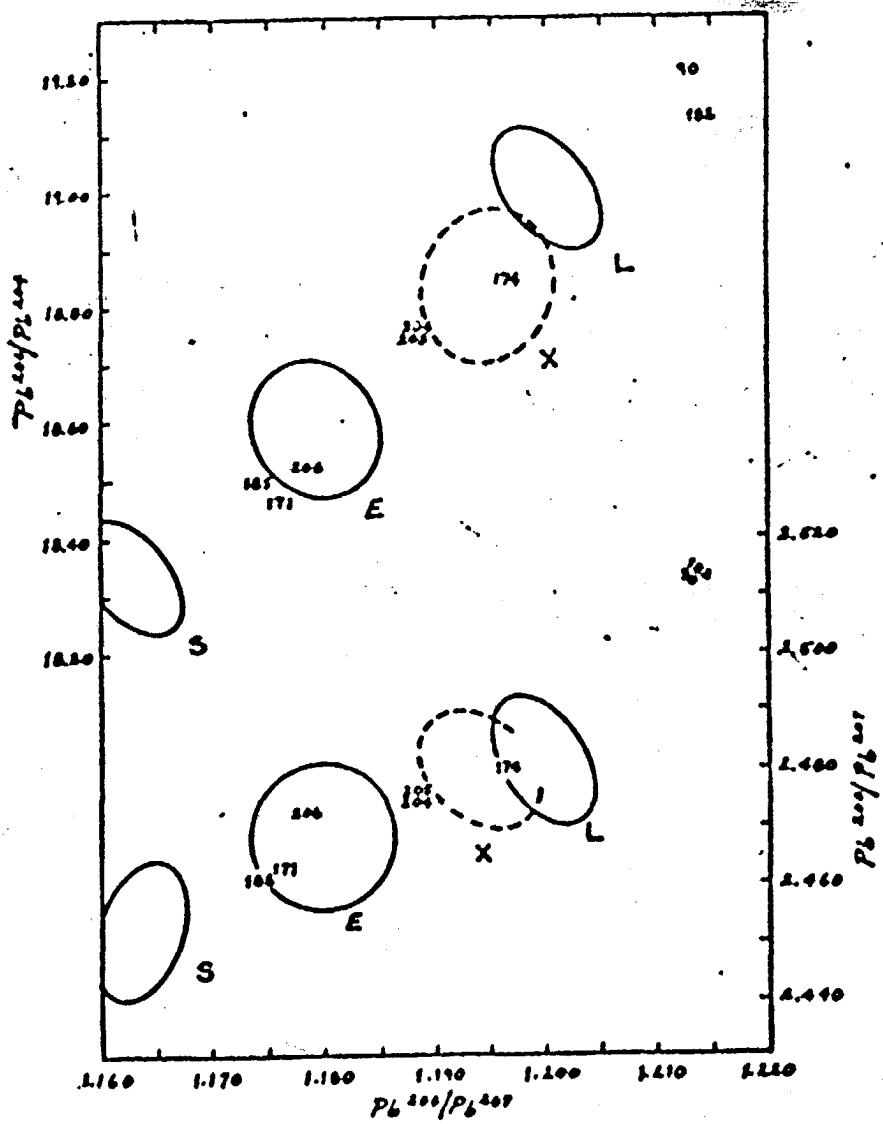
Fig. 3 Isotopic compositions of lead ores from ancient mining regions. Upper half is a plot of the Pb^{206}/Pb^{204} ratio vs the Pb^{206}/Pb^{207} ratio, lower half is Pb^{208}/Pb^{204} vs Pb^{206}/Pb^{207}. Upper points to be read against scales to left, lower points against scales to right. Numbers correspond to sample descriptions in catalogue. Broken lines enclose portion of graph enlarged in following illustrations



III. 3 Isotopic compositions of lead from excavated objects. Lead was assumed to have come from known mining regions. Loops are all three-sigma ellipses of samples, labeled L, E, and S. Cross-hatched data are were replotted from ill. 1



Ill. 3. Isotopic compositions of lead from excavated objects. Locations of mines which provided the lead are unknown. Loops set off the same three major groups drawn in ill. 2, and in addition an apparently broad group, X. Ore 193 is replotted as cross-hatched dot.



Ill. 4 Isotopic compositions of lead extracted chemically from specimens of ancient glass. Sample 185 has been replaced for comparison with sample 90

some parts of Turkey, but nowhere else in the Mediterranean region.

Having seen the picture of the ores, the prospects brighten considerably when the leads from archaeological objects are examined. Ill. 2, in which the scales have been expanded so as to show more clearly the region of most interest, contains data for thirteen samples of ancient leads. These are all samples which can safely be assumed to have been made from lead which came from specific mining areas. The six samples to the right were all assumed to have been made of lead mined at Laurion or in its immediate vicinity; the five in the middle are from lead pigs which bear inscriptions giving the names of the mines they came from in Roman Britain; and samples 41 and 42 were excavated at Sassari, in a lead mining region of Sardinia.[9] When these points are compared to the data for the corresponding ores, replotted here as shaded dots, the agreement is very close. In fact, within the English leads alone one can almost associate the appropriate pigs with the ores found in the same counties.

On the basis of this close agreement, supported by data for other ore samples in the geological literature, three separate types of lead have been set off by the loops drawn around the points. These boundaries are arbitrary and should be considered as only provisional. It may appear that these groups have been defined on rather scarce evidence, since so few ores have been used. Therefore, it must be emphasized that the reality of these groups is consistent with both the geochemical literature on lead isotopic compositions[9] and the known geological histories of these areas. The leads which fall in the group to the left, those lowest in Pb^{206} and Pb^{208}, are from rocks formed early in the Paleozoic Era. The leads from England are from later Paleozoic rocks. In the central and eastern Mediterranean region, where the samples in the group to the right originated, the predominant rocks were formed during more recent geological eras.

For the present, these groups have been designated as Group L (for Laurion like), Group E (for England), and Group S (for Spain), although it must always be kept in mind that ores from other geographical areas are also included in these groups.[10] Even with isotopic analysis of only moderate precision, it is possible to distinguish between leads from these three major groups.

Ill. 3 shows data for twenty-three other samples from archaeological objects. Although these objects came from known contexts, it was not possible to specify just where the leads themselves had been mined. The provisional boundaries for the three groups in Ill. 2 have been redrawn on this graph and it can be seen that many of the new set of points fall either within the groups or near their boundaries. In particular, there is one cluster of points falling just to the left of Group L which appear to differ significantly from the Laurion-like leads. These points have been enclosed by dashed boundaries to indicate a tentative fourth group of leads, Group X.[11]

Since each sample among these twenty-three presents an interesting case considered either by itself or in relation to others, it is difficult to discuss the results without the risk of rambling. Therefore, in order to simplify the discussion, only a few remarks will be made here under group headings. Concise statements regarding each sample are included in the catalogue descriptions.

LEADS OF GROUP L

Several samples containing lead of unknown origin were found to resemble the known Laurion leads. These include sample 135, from a curse tablet excavated at Antioch-on-the-Orontes, sample 19 from Cyprus, and sample 39 from the Bronze Age shipwreck off Cape Gelidonya.

Two samples from Sardis (63 and 66), separated by nearly a millennium in time, are virtually identical, and very probably came from the same mine. The differences between the Pb^{206}/Pb^{204} values for these samples and the known Laurion samples could be the result of analytical error, since different methods of analysis were used. On the other

[9] At least some leads from Sardinia are known to have isotopic compositions similar to those from Southern Spain. See Barnett and Pamplonet, *op. cit.* (supra n. 2) 178, 205.

[9] See R. Moorbath, "Lead Isotope Abundance Studies on Mineral Occurrences in the British Isles and Their Geological Significance," *Phil. Trans. of the Royal Soc. of London* 254, series A (1962) 295–360, as well as Barnett and Pamplonet, *op. cit.* (supra n. 2) and many others.

[10] For the moment the designations L, E, and S seem as convenient as any, but it may be that a different system will prove more reasonable as further results become available.

[11] It is worth pointing out that there is a distinction between Group X and the other groups. The first three groups were established by samples which were known or are at present known to have originated in known mining areas, while Group X emerged empirically from the study of samples from unknown mining regions.

hand, the differences could be real. We might note here too that the lead in the Sardis samples also differs isotopically from sample 113, an ore from Balya. Balya is only about eighty miles north of Sardis.

LEADS OF GROUP X

The leads in this group show a definite trend away from Group L. Since most of these samples came from objects excavated in Italy or Sicily this could be considered an indication that there is an "Italian" type of lead which is distinguishable from the Laurion-like leads. Such a group could prove to be very useful, but the data at present are too few to be conclusive. The evidence for an "Italian" type includes samples 55, 58 and 61 from Morgantina, sample 40 from the Temple of Segni, sample 46 from an Etruscan tomb and a single ore from Campiglia Marittima (193). Since there is a considerable spread among the Group X samples themselves, they are not necessarily identical in isotopic composition. The presence of a very early sample from Nippur (43) among these leads points out that Group X also contains leads from Middle Eastern sources and perhaps other regions as well. In the geological literature one lead from Italy has been reported which has an isotopic composition within this range, but such ores appear to be rare in other parts of Europe. A few have also been reported from localities in Yugoslavia and North Africa.

LEADS OF GROUP Z

A number of interesting samples were added to this group. Sample 81 consisted of a few flakes of glaze removed from a potsherd of the first century A.D. found at Caerleon on the Bristol Channel. The lead from this sample was extracted chemically and used for isotope measurements.[10] The isotopic composition resembles that of an ore believed to have come from Machen a few miles away and makes it almost certain that this pot was glazed locally and was not an import.

Sample 162 was from a lead cistern of the first century A.D. at Pompeii. Unless there are native Italian leads which are of this type[11] it can be reasoned that the lead might have been imported from England, for the Roman lead mines there were becoming active at that time. If this were so, the date of import would be narrowed down to between the beginning of Roman working of the mines in the Mendip Hills (ca. A.D. 49) and the destruction of Pompeii in A.D. 79.[12]

LEADS OF GROUP B

Two additional leads were found to be of this type. Sample 121 is from a lead pot cover excavated at Ur. Since its date is so early (ca. 3000 B.C.) it could hardly be expected to have been made from lead mined in any of the regions previously included in this group (Spain, Wales, and Sardinia). This is more likely an indication that lead deposits of this type exist somewhere in the vicinity of Ur, possibly in the Zagros Mountains, and illustrates the need for investigation of ores from that region. To date, however, we have been unable to obtain any specimens for study. Sample 176 is a lead droplet removed from a piece of slag found in the old workings of the Samrah silver mine in Saudi Arabia. It is very rich in silver. The lead is quite different from the sample of lead ore (175) found at the same site, indicating that ores from more than one source were brought to this site for smelting or cupellation.

THE MIDDLE EASTERN LEADS

Ten lead samples from several Middle Eastern sites were studied. The samples came from Nippur (43), Ur (121), al Rimah (203), Gurob (167), El Amarna (163), Hazanlu (123), Nimrud (182 and 183), Antioch (135), and Beth She'arim (191). They range in date (in the above order) from ca. 3500 B.C. to ca. A.D. 300. The isotopic compositions spread over a wide range of values, with some points falling within the established groups, while others do not. This is ample proof that a number of lead deposits from different geological environments were worked throughout the Middle East in ancient times.

The only Mesopotamian site from which more than one sample has yet been taken is Nimrud. Two samples from there were found to be quite different from one another isotopically. Sample 183 is similar to the Group L leads, except for a distinct en-

[10] This illustrates that even extremely small samples can be used for isotope studies.

[11] None have been reported in the literature.

[12] In this case such reasoning might be open to question but it is mentioned here merely to show the kind of information that might theoretically be obtained from lead isotope studies.

ores of Pb^{200}, while sample 182 is different from anything else measured, except for a most significant sample of glass also from Nimrud. (See sample 90 below.) Only one lead, sample 203 from al-Rimah, falls between the Group L leads and these two unusual samples from Nimrud.

The Middle Eastern samples do show some similarities. The samples from El Amarna (163), Hasanlu (128), and Beth She'arim (191), although of very different dates, all came from ores which must have been similar. They fall within Group E and, in fact, somewhat resemble sample 118, the ore from northeastern Turkey. The leads from Gezer and Antioch (167 and 135) both fall within or on the borderline of Group L, but seem to differ from one another isotopically as well as in date. Sample 43 from Nippur is a Group X lead, and the sample from Ur (121) falls within Group S.

LEAD EXTRACTED FROM ANCIENT GLASSES

Lead is present as an intentional ingredient in only a small fraction of ancient glasses, but these glasses are usually of special significance. Lead was often added to help the precipitation of cuprous oxide for making bright red opaque glasses, or in the form of lead antimonate ($Pb_2Sb_2O_7$) as a yellow opacifier.[10] We extracted lead chemically from seven specimens of ancient opaque glasses and used it for isotope measurements. The results are plotted in ill. 4.

Sample 90 was from a cake of bright red opaque glass excavated at Nimrud, and suspected to have been made there. It has recently been redated to ca. 210 B.C. or later. The isotopic composition of its lead matches that of one of the pieces of metallic lead from Nimrud (182). That piece of lead and the lead in the red glass very probably came from the same mine. This can be taken as evidence that either they were imported from the same place, or a more likely possibility, that the red glass was definitely made at Nimrud, where this type of lead was available. The lead in these two samples is completely different from anything else we have yet encountered.

Samples 204 and 205 are from green and red opaque tesserae from Shavei Zion. The leads in the two tesserae are identical and fall between Group L and Group F. They may represent an ore type not seen before in this study, or they may be a mixture of two or more types of lead. It is especially noteworthy that the lead was introduced into these glasses by two different chemical processes. It was added to the green glass in the form of a yellow pigment which was probably prepared separately beforehand, and not struck in the glass. In the red glass the lead must have been added as a separate ingredient either in the form of litharge, or possibly in combination with the copper. Since the leads are identical isotopically, the yellow pigment and other basic lead ingredient were very probably prepared in the same workshop.

There has always been some question as to where the well known red opaque glasses of the Roman period were made. Eventually, lead isotope studies may provide an answer to this question. The two areas which have been given most consideration are Italy, in the environs of Rome or Naples, and Alexandria. The latter is sometimes favored because the glasses so often occur in objects containing traditional Egyptian motifs.

The results we have obtained for lead extracted from three red opaque glasses are quite similar. Sample 171 was removed from a miniature mosaic plaque of unknown provenience, but dating from the first century B.C. or the first century A.D. Sample 206, a red opaque tessera from Pompeii, is probably of a similar date, and sample 185, which was excavated in London, is of unknown date but believed to be Roman. The results resemble one another closely enough to suggest a common source of manufacture, which is not at all unlikely. All three are included in Group F, but sample 185 in particular seems to be somewhat different from the English leads we have analyzed.[11] Although it has already been pointed out that the presence of a lead in Group F does not mean that the lead came from England (only 171 was excavated there) such a theory would not be unreasonable in this instance because of the known Roman lead mining throughout England. If this should be substantiated by further data or linked to independent information, it would be a valuable finding, since it would establish

[10] See ref. 11 following paragraph and also W. B. S. Turner and H. P. Rooksby, "A Study of Opacifying Agents in Ancient Glasses Throughout Seven Years," *Glastechnische Berichte* 32K, VIII (1959) 17–28; H. P. Rooksby, "A Yellow Cubic Lead Tin Oxide Opacifier in Ancient Glasses," *Physics and Chemistry of Glasses*, 3, no. 1 (Feb. 1962) 30–39.

[11] Manuelsson, ref. 10 (supra n. 3), reported English leads similar to our sample 185.

lish a *terminus post quem*, ca. A.D. 43, for red glasses containing that type of lead and for the objects in which they were incorporated.

A fourth piece of red opaque glass (174) from Tara Hill in Ireland is of unknown date and place of manufacture. The lead in this glass seems to be of the "Italian" type, and does not at all resemble any of the English or Welsh leads that have been studied so far. Although there are a number of sources of lead in Ireland, it is uncertain if they were worked in ancient times[11] and unless there is some source of lead in Ireland that has this same isotopic composition, it is doubtful that the glass could be of local manufacture. No such leads were observed by Moorbath,[9] who reported data for thirteen Irish leads. Among his samples the highest Pb^{206}/Pb^{204} ratio found was 1.178, which falls within our Group E.

MISCELLANEOUS LEADS

Sample 188 was a small quantity of lead recovered electrolytically from a wet chemical analysis of a Chinese bronze ceremonial vessel of the late eleventh to early tenth century B.C. The isotopic composition of this lead is so different from the rest of the objects studied that it falls beyond the scale of the graphs in ills. 3, 4. The point is plotted in the lower left corner of ill. 1.

Sample 5 is from the solder holding an Attic column to its pedestal. The column dates from about 535–525 B.C. and is now in the Metropolitan Museum of Art. The lead is definitely not like the Laurion leads, which is quite surprising considering the date and provenience of the column. The lead may represent an ancient repair (it does not look modern) and might have been made with an "Italian" lead, or with a mixture of leads of two or more types.

Four other samples (1, 4, 12 and 17) were studied before the main experiments were under way.[10] Since these were only preliminary experiments of uncertain accuracy, the data have not been plotted on the graphs. It was apparent, however, that sample 1 (a lead pottery clamp from Kea, sixteenth to fifteenth century B.C.) and sample 4 (a lead solder from an Attic grave stele, ca. 550–535 B.C.) were Laurion like leads. Sample 12 (a Roman lead figurine from Chesterholme) and sample 17 (a Roman lead slag from nearby Green Ore) both seem to be of Group F, but tend toward the direction of Group S. All of these isotope findings are consistent with the archaeological attributions.

EVALUATION OF THE TECHNIQUE

The most restrictive limitation of the technique is of a logical nature. While it would be very convenient if each lead mine had its own unique isotopic composition, this is far from being true. Isotopic compositions of leads from widely separated sources may be indistinguishable if the deposits are geologically similar. Therefore, one cannot hope to use isotope evidence alone to prove conclusively that the lead from some given object was taken from some given mining area. In making attributions one can only say that the lead from a particular object came from either one particular mining area or from some other regions (possibly unknown) which are geochemically similar. For practical purposes, however, especially where independent evidence is available, such results can still be useful. For example, although the lead in the glaze on the pottery sherd from Caerleon (81) could theoretically have come from any of several different places, it probably really did come from nearby and the sherd probably really is of local manufacture.

On the other hand, isotope measurements do permit one to make conclusive negative judgments. The leads from El Amarna, Ur, and Hasanlu definitely did not come from Laurion or any other Group L mine and the leads from Morgantina could not have come from Southern Spain or Sardinia.[10] Such negative evidence was also useful for showing that the red glass from Tara Hill is probably not of local manufacture. In fact, results need not always involve particular mines to be of significance. It may be helpful for the archaeologist to know simply whether two or more leads could have come from a common source or must have come from different sources.

Another limitation of the method is that in ancient times, just as at present, lead must often have been salvaged and reused time and time again. There is always a possibility that leads from different sources might have been remelted together. In such instances, the resulting isotope ratios would

[9] Private communication: M. J. O'Kelly, University College, Cork, and C. M. L. Bouska, R.T.É. Services, Ltd., Dublin.

[10] These measurements were made by Dr. Jerome Cobb of Brookhaven National Laboratory.

[11] There is a possibility that Group B lead could be present if mixed with a much greater quantity of Group L lead.

be intermediate between those of the individual leads. It is probable that some of the leads reported here are such mixtures; for example, possibly sample 5, sample 58, or samples 204 and 205.

In order to gain full benefit of the isotope data on ancient leads, mass-spectrometry of the highest precision is required because we know now that small differences in isotopic composition can be significant. This does not constitute a real drawback because the current trends in instrumentation are now making it more convenient to get very precise data on thermal emission instruments. Moreover, by using duplicate or triplicate analyses it should be possible to distinguish between samples which differ by no more than a few tenths of a per cent in their isotope ratios.[20] It is important, if samples are to be compared to one another, that they be prepared similarly beforehand and run on the same instrument. Mass spectrometric data at present are notoriously sensitive to the characteristics of individual instruments, and to different methods of introducing the samples into the instrument.

There are several very advantageous features to this isotope technique, the first being that only very small samples are required. Literally only a few micrograms of sample are consumed in the analysis, but for practical handling purposes one needs a few milligrams at least. In addition, the lead need not be a pure metal. Corrosion products, pigments, or lead extracted from materials such as alloys, glazes, or glasses, have all been studied successfully.

The technique has one important advantage which is missing in chemical analyses. The most frustrating part of trying to characterize metals by their chemical compositions (for instance, by comparing their trace element contents) is that every process to which a metal is subjected, from mining to smelting, melting, forming, and subsequent corrosion, can alter its chemical composition. The isotopic compositions of lead samples remain essentially constant, however, no matter what one does, short of mixing together leads from different sources. An ore can be mixed with other minerals, crushed, smelted, and the metal melted, remelted, oxidized, reduced, formed, corroded, or reacted to form chemical compounds, and the resulting lead will still retain its original isotopic composition within the measurable limits of interest here.[21] The reason for this is that the isotopes of a given element differ only in mass, and their chemical properties are virtually identical. It is only through painstaking chemical or physical procedures that isotope separations can be effected. The fact is that if isotopes could be separated by simple chemical operations, the whole history of chemistry would probably have been very different.

CHEMICAL ANALYSES

As a supporting experiment, quantitative spectrographic chemical analyses have been made of all the samples studied, and of some hundred others in addition. Particular efforts were made to obtain accurate silver values. In this way it has been possible further to characterize the leads as being made from ores smelted for lead, or leads that were desilvered. It is well known that in ancient times lead was often just a useful by-product, and that ancient miners were really more interested in the silver that could be extracted from the lead. The results of the silver analyses are included in the catalogue of sample descriptions.[22] There are some indications that other chemical elements present as impurities, sometimes only as traces, may serve to tie together some of the samples and ores studied.

[20] At present our estimated analytical errors, referred to single determinations, are as follows: [illegible]. The error refers to the accuracy of our determinations, that is, the comparison of the experimental values to the "true" values. For those samples introduced in [illegible], the precision of our determinations, or the reproducibility of the measurements, seems considerably better. Actually, for the comparison of leads from different samples run under a given set of conditions in one laboratory, high precision is more important than high accuracy. For comparing results between different laboratories, however, high accuracy becomes just as important. Our estimated accuracies are based on comparisons of samples run by both the lead sulfide and [illegible] techniques and upon several analyses of lead isotope reference samples ([illegible]) which have been analyzed by many other laboratories.

[21] Although a slight isotope fractionation is likely to occur during the vaporization of lead, it is difficult to picture a practical situation where such a process might alter the isotope ratios by as much as one-tenth per cent.

[22] Various authors have discussed the extent to which ancient metallurgists were capable of extracting silver from lead. We hope that eventually our chemical analyses will provide some additional useful information on this subject. For the moment, however, we shall only mention that the Romans, and probably some Hellenistic metallurgists, were capable of extracting silver from lead down to a level of about [illegible] per cent residual silver. Our data do not yet allow us to place a similar level for earlier metallurgists. We would like to repeat here, for emphasis, that there were galena deposits worked in ancient times, such as some in England, which had natural silver contents of less than [illegible] per cent. Therefore, it is not valid to assume that all archaeological leads containing little silver (of the order of [illegible] per cent) must necessarily have been desilvered.

PROSPECTS FOR FURTHER WORK

In conclusion, these experiments provide encouragement that the measurement of isotope ratios in archaeological objects made of lead can provide useful information to archaeologists concerning possible sources of the lead used for making the objects. It is also clear, however, that considerably more experimental data are required before the technique can be applied generally. Further studies may establish new isotope groups in addition to the four provisional groups outlined here, but it is also likely that the groups as set out now will have to be broadened geographically to include other mining areas which are geologically similar. Further work is anticipated on ores from other areas, particularly from the Middle East in such places as the Zagros Mountains, which might have supplied lead to the early Mesopotamian civilizations. The Italian ores require more study to see if a discrete Italian group can be defined, and work is anticipated with Macedonian ores whose lead might be different from Laurion lead. It will also be necessary to make a more comprehensive comparison with isotope data in the literature concerning leads from Central Europe and the Balkans. These have been studied by a number of other investigators for geological purposes, and it is evident that there is overlapping in isotopic composition among these lead ores and the major groups outlined in this study. Leads from the Far East have been neglected here because the initial project had to be confined to a manageable scale, but work is planned on ores from India, Afghanistan, and Japan. Because of the widespread occurrence of Precambrian rocks in the Far East, the range of isotope variation will probably be expanded by future studies.

It also remains to be established just how much variability exists within ores from certain mining areas. We have assumed (but not without reason) that this variability is less in general than the variation between different regions. The mines of Sardinia may prove to be an interesting case study in this connection.[10]

One of the most promising applications to emerge from this study is the comparison of the isotope compositions of leads in lead-containing ancient glasses. As pointed out earlier, these glasses amount to only a small proportion of ancient glasses, but they are found in widely separated places and span a period of several centuries. We plan to make a thorough investigation of such glasses as our work continues.

It does not seem likely that isotope analysis will prove helpful in the same way for the study of other metallic elements, but isotope studies of some of the non-metallic elements are potentially useful. With the continuing accumulation of geochemical data and rapid refinements in instrumentation the possibilities should be reviewed from time to time.

We acknowledge most gratefully the assistance of numerous individuals and institutions in supplying information and samples for study. This includes not only those named in the catalogue which follows, but also those who for one reason or another remain anonymous; and equally the more than a hundred other persons who have written long, patient letters or provided us with samples which have not yet been studied.

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CATALOGUE OF SAMPLES

Galena Ores

18. Alston Moor, Cumberland Co., England. Robert Hogg, Museum and Art Gallery, Carlisle. (0.01% Ag) Group E.
19. Coal Measures Galena (Roman Smelt), Matlock, Derbyshire, England. Collected by R. H. Brill and Arthur Shoenhall, Matlock. (0.003% Ag) Group E.
32. Dylife (main section), Montgomeryshire, Wales. Collected by R. H. Brill with William Richards, Cecil Vaughn Owen and W. G. Putnam. (0.005% Ag) Group S.
77. Kamareza (near Laurion), Greece. Constantin Conophagos and Michel Privat, Athens. (0.3% Ag) Group L.
82. Corbridge, Roman Legionary Fortress, 1st and 2nd c. A.D. Probably from lead workings near Matlock, Monmouth. George C. Boon, National Museum of Wales, Cardiff. (0.001% Ag) Group E.
85. Akdag Maden, Yozgat Province, Turkey. R. F. Lethbridge and D. J. Small, RTZ Services, Ltd., London. (0.03% Ag) Probably Group L.
89. Rio Tinto, Spain, from North Lode near Roman workings. R. F. Lethbridge and D. J. Small, RTZ Services, Ltd., London. (0.11% Ag) Group S.
111. Nakhlak, Iran (near Anarak, ca. 125 mi. ne of Isfahan). Mohamad Oreimi, Persepolis College. (0.08% Ag) Group L.

[10] We have this suggestion as several very helpful and detailed private communications from Professor Cuthbert Desborough, University of Pisa.

112. Khana Surma, Iran (near Shahreza, ca. 50 mi. s of Esfahan). Mahmoud Ommi, Brooklyn College. (0.07% Ag) Group L.

113. Balya, Turkey (near Balikesir). Myles Walsh, New York City. (0.12% Ag) Probably Group L.

118. Yukari Maden Köyü, Turkey (near Artvin). Myles Walsh, New York City. (0.05% Ag) Group Z.

136. Gebel El Rusas, Egypt (Eastern Desert, 25° 11' N—34° 46' E). From Middle Miocene limestone, gypsum and evaporite series. A. M. Abdel-Gawad, Inchass, UAR. Anomalous content of radiogenic isotopes suggests ore was derived from the Precambrian basement rocks of this region.

137. Um Gheig, Egypt (Eastern Desert, 25° 30' N—34° 30' E). From Middle Miocene limestone, gypsum and evaporite series. A. M. Abdel-Gawad, Inchass, UAR. Anomalous content of radiogenic isotopes suggests ore was derived from the Precambrian basement rocks of this region.

139. Um Samuiki, Egypt (Eastern Desert, 24° 18' N—34° 45' E). From Precambrian basalt rocks; galena mixed with sphalerite, pyrite and chalcopyrite. A. M. Abdel-Gawad, Inchass, UAR. Isotopic composition is consistent with Precambrian age of host rocks.

173. Samuah (near Ad Dawadmi), Najd Prov. c. Saudi Arabia. From "ancient silver mine." Harold A. Quinn and F. K. Kaldass, Jeddah. Isotopic composition is consistent with Precambrian age of host rocks.

193. Campiglia Marittima, Italy (Refs. 1, 2). Gabor Desson, University of Pisa. (0.03% Ag) Group X.

Archaeological Objects

3. Lead soldier from base of marble stele of Antigonos, said to be from Amun, ca. 310-300 B.C. MMA 23.167 (Ref. 3). Metropolitan Museum of Art, New York City. (0.01% Ag) This sample does not closely resemble any of the other leads studied. Possibly Group X, or a mixture of two or more leads.

6. Molded lead bronze wreath from the Temple of Artemis Orthia, Sparta, probably 6th cent. B.C. MMA 24.135.163 (Ref. 4). Metropolitan Museum of Art, New York City. (0.01% Ag) Group L.

10. Lead mounting of bronze bowl or spout, from Cyprus (said to be Curium). Hellenistic. MMA 74.51.5673 (C.B. 451) (Ref. 5). Metropolitan Museum of Art, New York City. (0.01% Ag) Probably Group L.

22. Rounded bar of red wax and lead, from Carthage, ca. 146 B.C. This object is probably waste from a cosmetics process and may have been intended for use as a pigment (Refs. 6 and 7). Mme. Farnsworth, New York City. (Less than 0.01% Ag) Group L.

32. Small fragment of metallic lead from the Athenian Agora, Mediaeval. (0.01% Ag) Group L.

34. Small fragment of metallic lead from the Athenian Agora, ca. 300 B.C. (0.04% Ag) Group L.

39. Small fragment of lead disk from Bronze Age shipwreck at Cape Gelidonya, Turkey, 1200 B.C. ± 50 (Ref. 8). George Bass, University Museum, University of Pennsylvania. (0.04% Ag) Group L.

40. Small fragment of lead from votive refuse, Temple of Segni, Latium, 3rd or 2nd cent. B.C. A. Neppi Modona, Istituto di Studi Etruschi ed Italici, Florence. (0.01% Ag) Group X.

41. Roman sarcophagus from Tusi (Sassari, nw Sardinia), 3rd cent. A.D. A. Neppi Modona, Istituto di Studi Etruschi ed Italici, Florence. (0.01% Ag) Group S.

42. Fragment of reinforcing clamp from a large funerary vessel, necropolis of Florinas, Sassari, 2nd or 1st cent. B.C. A. Neppi Modona, Istituto di Studi Etruschi ed Italici, Florence. (0.01% Ag) Group S.

43. Fragment of heavily corroded lead from Nippur, Presidential Period, ca. 3500 B.C. Oriental Institute, University of Chicago. (0.04% Ag) Group X.

46. Small fragment of lead from Etruscan tomb "La Montagnola," near Florence, Central Papinia, 7th cent. B.C. A. Neppi Modona, Istituto di Studi Etruschi ed Italici, Florence. (0.003% Ag) Group X, or possibly Group L.

47. Lead pig from Ringdon, Mendip Hills, Somerset, England, Roman Period (Refs. 9, 10, 11 and 12). J. W. Bradford and H. Burker, British Museum. (0.05% Ag) Group E.

49. Lead pig from Haydon Moor, West Riding of Yorkshire, England, Roman Period (Refs. 10, 11 and 12). J. W. Bradford and H. Burker, British Museum. (0.008% Ag) Group F.

50. Lead pig from Wadsworth, Derbyshire, England. Lutudarum mines, Roman Period (Refs. 10, 11 and 12). J. W. Bradford and H. Burker, British Museum. (0.006% Ag) Group E.

51. Lead pig from Matlock Moor, Derbyshire, England. Lutudarum mines, Roman Period (Refs. 10, 11 and 12). J. W. Bradford and H. Burker, British Museum. (0.004% Ag) Group F.

54. Lead pig from Portland Grange, Tansley Moor, near Matlock, Derbyshire. Lutudarum mines, Roman Period (Refs. 10, 11 and 12). J. W. Bradford and H. Burker, British Museum. (0.003% Ag) Group F.

55. Lead bowl in hole on [illegible] sculpture site, Morgantina, Sicily, mid-5th cent. B.C. From Area III, 111. Erik Sjoqvist and Richard Stillwell, Princeton University. (0.03% Ag) Group Y.

58. Fragment of lead pipe from House of the Archaic Cornice, Morgantina, Sicily, ca. 250-230 B.C. Erik Sjoqvist and Richard Stillwell, Princeton University. (0.01% Ag) Group X.

61. Mending clamp of caponi in House of Tuscan Capitals, Morgantina, Sicily, latter half of 2nd cent. B.C. Erik Sjoqvist and Richard Stillwell, Princeton University. (0.01% Ag) Group X.

63. Lead clamp, Sardis, 4th cent. B.C. From House of Bronzes. George M. A. Hanfmann and David Mitten, Fogg Art Museum. (0.007% Ag) Probably Group L.

66. Lead cladding over iron strap, Sardis, 6th cent. B.C. From antechamber of Lydian tomb at Bin Tepe. George M. A. Hanfmann and David Mitten, Fogg Art Museum. (0.12% Ag) Probably Group L.

70. Small fragment of metallic lead from Mycenae, Mycenaean Period. George Mylonas, Washington University. (0.03% Ag) Group L.

74. Small fragment of metallic lead from Mycenae, 3rd cent. B.C. George Mylonas, Washington University. (0.04% Ag) Group L.

121. Lead pottery from Ur, Jemdet Nasr, ca. 3000 B.C. University Museum no. 31-17-13, U20073 (Ref. 13). Robert Dyson, University Museum, University of Pennsylvania. (0.5% Ag) Group S.

128. Fine gray powder from core of a bronze kohl pot, from Hasanlu, ca. 850 B.C. HAS 59-861. This material was probably finely powdered galena ore (Ref. 14). Robert Dyson, University Museum, University of Pennsylvania. (0.03% Ag) On borderline of Group E.

135. Inscribed lead curse tablet from Amiens-on-the-Orontes, late 1st cent. A.D. or possibly 150 years later. No. 4758 [132 from 150, with nos. 2, 142, 435. Richard Stillwell, Princeton University. (0.02% Ag) Probably Group L.

162. Small fragment of large decorated lead cistern from Pompeii, probably 1st cent. B.C.—1st cent. A.D. Dr. Gordiano, Pompeii. (0.01% Ag) Group E.

163. Small lead net sinker from El Amrona, mid to late 19th cent. B.C. (Ref. 15). H. S. Smith, University College, London. (0.03% Ag) On borderline of Group E.

167. Small folded lead line sinker from Gurob, first half 2nd millennium B.C. (Ref. 16) H. S. Smith, University College, London. (0.03% Ag) Probably Group L.

176. Small metallic droplet in fragment of vitrified slag from base of ancient tailings at Sotiriadis silver mines, near Ad Dawadimi, Province of Nejd, Saudi Arabia. Date uncertain. Sample SQ 71-64, depth of 65 cm., sample no. 10. Found with small quantities of charcoal and bones. Harold A. Quinn and F. K. Kaldeen, Jeddah. (0.25% Ag) Group S.

182. Small flat bead of lead, heavily corroded, from the debris of building B 50 at Nimrud, not precisely stratified. The date of this object is doubtful. It could be as early as 700 or as late as about 500 B.C. M. E. L. Mallowan and B. Parker, Institute of Archaeology, London. (0.005% Ag) This lead does not match any of the other leads included in this study, with the possible exception of sample 90, which was recovered from a red opaque glass slag from Nimrud.

183. Folded lead sinker, corroded, from the debris of building B 50 at Nimrud, not precisely stratified. The date of this object is doubtful. It could be as early as 700 or as late as about 500 B.C. M. E. L. Mallowan and B. Parker, Institute of Archaeology, London. (0.005% Ag) Possibly Group L, but the high 208/207 ratio suggests an origin different from that of other leads in this group.

188. Small sample of lead chloride recovered from the analysis of the swing handle of a Chinese bronze ceremonial vessel dating from the early Chou dynasty, 11th cent. B.C. Similar to type 30, HGA 30-36. The alloy contains 11.6% Pb. Rutherford J. Gettens, Freer Gallery of Art. This lead was found to be entirely different from the leads found in any of the other archaeological objects studied. It was probably derived from an ore of Precambrian Age. Precambrian rocks are widespread in China.

191. Lead sarcophagus from Beth She'arim, A.D. 250-350 (Ref. 17). N. Avigad and D. Barag, Hebrew University. (0.01% Ag) Probably Group E.

203. Small piece of pyramidal shaped lead with white corrosion coating, from Tell al Rimah, ca. 1500 B.C. Sec A, above level 2, rm. 16. Theresa Carter, University Museum, University of Pennsylvania. (0.03% Ag) This lead might possibly be of Group L, but it seems more likely to be different.

Lead Recovered from Ancient Glasses and Glazes

81. Lead extracted from flakes of glaze removed from a pottery sherd of local manufacture. Carthon, Roman Legionary Fortress, 1st and 2nd cent. A.D. George C. Boon, National Museum of Wales, Cardiff. Group E.

90. Lead extracted from a fragment of bright red opaque glass from the north end of the Burnt Palace at Nimrud. This glass was once thought to date from the early 6th cent. B.C. (Refs. 18 and 19). More recent considerations of its stratigraphy, however (Ref. 20), have led to the conclusion that it probably does not date from before about 210 B.C. We have confirmed Pleodericah's and Bimson's chemical analyses (published by Turner) which showed that the glass contains about 22.8% PbO. (Probably devitrified.) This lead is distinctly different from any of the other leads studied, except sample 182, which is a piece of metallic lead from Nimrud.

171. Lead extracted from a flake of bright red opaque glass removed from a Roman miniature mosaic plaque, 1st cent. A.D.—1st cent. A.D. The Corning Museum of Glass no. 53.1.[illegible]. The plaque shows an adult eye and is of a type often said to have been made in Alexandria. The six different colored glasses making up the design have been analyzed and published (Ref. 21) and the red glass has been shown to contain nearly 30% PbO. The lead is probably of Group P.

174. Lead extracted from a large piece of red opaque glass found at Tara Hill, Ireland. The glass is of unknown date (Ref. 21). BM 91 5-15 1. Anthony Werner and Mavis Bimson, British

Museum. The lead is either of Group X, resembling the "Italian" leads, or possibly Group L, and is distinctly different from any of the leads in this study which came from England or Wales.

185. Lead extracted from a lump of red opaque glass said to have been found on Fish Street Hill, City of London. The glass is of uncertain date but is believed to be Roman, BM 1931 10-19 8. Anthony Werner and Mavis Bimson, British Museum. Probably Group E.

204. Lead extracted from yellow-green opaque glass tessera from Shavei Zion, 4th-5th cent. A.D., CMG anal. 482 (ca. 35% PbO, 0.007% Ag). M. W. Prausnitz, Jerusalem, and J. Ben Josef, Shavei Zion. Intermediate between Group E and Group X.

205. Lead extracted from red opaque glass tessera from Shavei Zion, 4th-5th cent. A.D., CMG anal. 481 (ca. 25% PbO, 0.010% Ag). M. W. Prausnitz, Jerusalem, and J. Ben Josef, Shavei Zion. Intermediate between Group E and Group X.

206. Lead extracted from yellow-green opaque glass tessera from Pompeii. 1st cent. B.C.–1st cent. A.D. Dr. Giordano, Pompeii. Group E.

Samples used for Preliminary Experiments

2. Lead pottery clamp from Kea, 16-15th cent. B.C. John Caskey, University of Cincinnati (0.03% Ag) Group L.
4. Lead solder from marble grave stele surmounted by a sphinx, said to be from Attica, ca. 540-535 B.C., MMA 11.185 (Ref. 23). Metropolitan Museum of Art, New York City (0.03% Ag) Group L.
12. Lead figurine from Charterhouse-on-Mendip, Somerset Co., England. Roman, F 2057, Mendip, A. C. Pass Collection. L. V. Grinsell, The City Museum, Bristol (0.05% Ag) Group F.
17. Lead slag from Green Ore, Somerset Co., England. Roman (Ref. 9). L. S. Palmer, Wells Museum, Wells. (0.002% Ag) Group F, or possibly Group S.

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