LEAD AND SULPHUR ISOTOPE RATIOS OF SOME GALENA SPECIMENS FROM THE SOUTH PENNINES AND NORTH MIDLANDS

by

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Summary

The isotope composition of lead, extracted from 17 samples of galena from the South Pennines, and 6 from small deposits in Triassic rocks to the south and east of the South Pennines, has been analysed. The analyses include recalculated figures from those analysed by Moorbath in 1962. The results show that the leads are extremely homogeneous and also anomalous (averages: $^{206}\text{Pb}/^{204}\text{Pb} = 18.45 \pm 0.02$; $^{207}\text{Pb}/^{204}\text{Pb}$ 15.62 ± 0.01; $^{208}\text{Pb}/^{204}\text{Pb} = 38.32 \pm 0.03$). Lead samples from Triassic rocks are isotopically similar to those of the South Pennines. Taken as a whole, lead analyses in the South and North Pennines define a significant isotopic trend similar to that defined by isotopically more heterogeneous Mississippi Valley lead samples.

The lead isotope homogeneity indicates an opportunity for the lead to mix prior to mineralisation and thus be derived from a distant source. Isotopic studies of trace quantities of lead extracted from potential host rocks are needed to understand further Pennine lead isotope abundances.

Analyses are available on the isotope composition of sulphur, extracted from 5 samples of South Pennine galena. The δ^{34} S-values range from +3.2 to -8.4 per mil. The very limited sulphur isotope evidence indicates that the sulphides formed at low temperatures, and that further study has great potential.

Introduction

In the years since Moorbath's (1962) survey of British isotope abundance studies, which included only 4 analyses from the South Pennine Orefield, considerable advances have been made both in the understanding and in the measurement precision of lead isotopes.

Only one isotope study of Pennine lead has been published since Moorbath's paper, that of Mitchell and Krouse (1971), who carried out a detailed study on galena from the relatively small Greenhow - Skyreholme area of the North Pennine Orefield. These authors concluded that the lead isotope compositions of the specimens are anomalous, when interpreted using simple models, in that the compositions yield meaningless negative model ages, suggesting that the ore deposit has not yet formed. Mitchell and Krouse (1971) compared their results with those of Moorbath, for other Pennine areas, and concluded that all such lead isotope compositions are anomalous and that those from the different areas of the Pennines may form part of a common isotopic trend. If this trend could be better established by using high precision analytical techniques not available to previous authors, for Pennine lead isotopes, it could yield useful geochronological information about the source rocks.

This contribution presents new lead isotope evidence obtained as a result of the new analytical techniques on galena samples from the South Pennines and North Midlands, and presents a theory to explain the significance of the results in the context of existing knowledge

of lead isotopes. The methods of analysis and the criteria for interpretation are discussed.

Sulphur isotopes have proved themselves to have great potential in constraining theoretical models of ore formation. Reconnaissance sulphur isotope measurements on several South Pennine galena samples are presented and the results discussed.

Geology

The South Pennine Orefield in common with the other Pennine Orefields, is characterized by galena-fluorite-barite veins in Lower Carboniferous limestones.

A series of approximately east-west striking mineral veins with closely associated pipes and flats occur within massive limestones interbedded with occasional basalts and tuffs (Ford, 1969). The chief vein minerals are galena, fluorite, barite and calcite. The history of mineralization is complex, with five or more distinct mineralization pulses having been recognized in some veins (Ineson and Al-Kufaishi, 1970; Firman and Bagshaw, 1974).

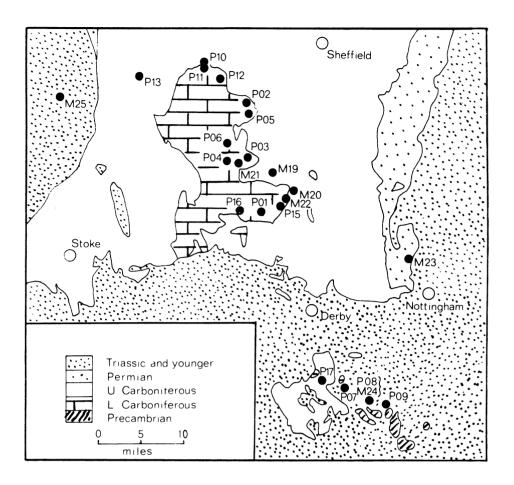
Moorbath (1962) analysed four South Pennine galenas and obtained lead model ages ranging from 150 ± 80 to 220 ± 70 m.y., but, as will be discussed in the next section, little value can be attached to these ages, because the model used has since been shown to be invalid

Ineson and Mitchell (1973), have recently dated clay minerals separated from volcanic rocks adjacent to mineralized zones using the potassium/argon method. They interpret their results to indicate that repeated pulses of mineralization probably occurred within the South Pennine Orefield, with at least two main episodes at about 270 m.y. and about 235 m.y. respectively.

Mineralization is not confined entirely to the eastern margin of the South Pennines Lower Carboniferous outcrop. An important pipe deposit, containing copper and zinc, as well as lead, occurs further west in Lower Carboniferous limestones at Ecton. Smaller scale but widespread mineralization occurs at horizons within the Permian Magnesian Limestones (Deans 1961; Aldred 1969; Taylor and Elliott 1971) and at the Triassic/Lower Carboniferous unconformity to the south of the main orefield (Ford and King 1968). Ford (1969) suggested that the persistent mineralisation at this unconformity could be explained if the South Pennine mineralization, or a late pulse of it, occurred during Triassic times.

The ultimate source of the South Pennine lead ore has not yet been proven, but the lack of any known igneous body beneath the ore-field, combined with the recent discovery of evaporite beds at the base of the Lower Carboniferous in some parts of the area (Llewellyn and Stabbins, 1969; Dunham, 1973) points towards the metals probably having been leached by brines from buried Carboniferous rocks further to the east and migrating westwards. Preliminary fluid inclusion results by Smith (1973) are compatible with such a hypothesis.

For this study 17 galena samples were added to the analyses available from Moorbath's (1962) data (recalculated). 15 of these came from various types of mineralization widely scattered throughout the South Pennines Orefield. One sample came from the Ecton copper deposit and another from the unusual vein at Whaley Bridge; four further samples were from small deposits at or close to the Triassic/Lower Carboniferous unconformity situated in the North Midlands. Two further samples are from localities within the Permo-Triassic rocks of the same area. All the localities are shown on text-fig.1.



Text-fig. 1. Sample Locations

Lead Isotopes

Lead isotopes vary in natural abundance because radioactive decay of uranium and thorium isotopes has progressively formed new "radiogenic" lead. Because lead in galena is effectively isolated from uranium and thorium at the time of formation, the present isotopic composition is controlled by what happened <u>before</u> precipitation.

The amount of radiogenic lead in the earth as a whole has progressively increased with time, so more recently mineralized galena deposits, by and large, have more radiogenic lead than older ones.

Lead has four stable isotopes: ^{204}Pb ; ^{206}Pb ; ^{207}Pb ; and ^{208}Pb . Of these only ^{204}Pb is unaltered by uranium and thorium decay, that is, its abundance is constant so lead isotope variations are usually expressed relative to ^{204}Pb , as $^{206}\text{Pb}/^{204}\text{Pb}$ and so on.

The isotope ratios of a specific suite of galena samples can be interpreted only within the context of existing <u>models</u>, which have been evolved to explain lead isotope observations in general. Surveys of lead isotope models have been given by Russell and Farquhar (1960) and Doe (1970).

All lead isotope models make the basic assumption that at the time of formation of the earth all lead had the same "primeval" isotopic composition, and that natural abundance variations have been caused solely by the addition of radiogenic lead since that time.

The isotopic composition of primeval lead has been obtained by analysing lead extracted from the uranium-and thorium-free troilite phase of meteorites.

The first model to be used widely was the so-called Holmes-Houtermans model. The assumption is made that the lead of any galena has spent the whole period between the formation of the earth and the precipitation of the galena in an environment whose U/Pb ratio was effectively constant in time. Lead in other galena samples evolved in source environments with a different U/Pb ratios. These assumptions allow a "model age" and a theoretical source U/Pb ratio to be calculated for lead in any single galena. The model was "calibrated" so that it gave reasonable ages on galena specimens of independently known age by adjusting the parameters, in particular the "age of the earth", within the then known uncertainties.

Despite the simplicity of its assumptions, the Holmes-Houtermans model allowed approximate, but useful ages to be assigned to a wide variety of galena specimens. Lead samples that yielded unreasonable or negative model ages were termed "anomalous". The model is no longer used, however, because its simple assumptions have been shown to be dubious. This is why Moorbath's (1962) model ages on British lead ores can no longer be accepted.

The next model to be used widely was the so-called Russell-Stanton-Farquhar model. These authors noted that many lead specimens, in particular those from conformable ore deposits closely associated with volcanic rocks, all fall on a single "ordinary growth curve" (text-fig. 2). The simplest explanation of this curve is that all these lead samples were tapped off at different times from an environment whose U/Pb (Th/Pb) ratio changed only as a result of the radioactive decay. The authors of this model further argued that this homogeneous source environment was the upper mantle. Lead samples that fall on this curve were termed "ordinary", whilst those that did not were termed "anomalous" and were assumed to have evolved for a considerable period within the earth's crust.

Kanasewich (1968) summarized models that could make sense of many suites of anomalous leads, which very often fall on lines that intersect the ordinary growth curve in text.fig. 2. These models assume that anomalous leads evolved in more than the two stages envisaged for "ordinary" leads, and hence were termed "multi-stage" models.

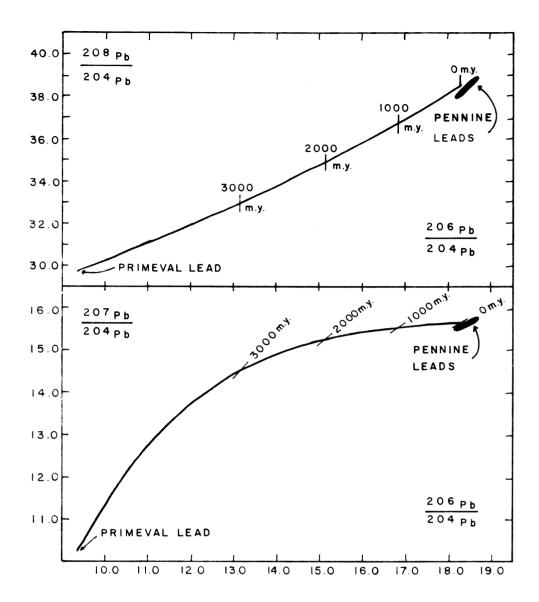
Richards (1971) recently summarized evidence which shows that, although the isotopic distribution represented by the curve in text-fig. 2, remains a fact, these ordinary leads cannot have originated in the upper mantle. This is because modern studies have shown that the mantle is not geochemically homogeneous and because leads extracted from modern midoceanic tholeites, which almost certainly came from the mantle, are themselves anomalous.

As a consequence, emphasis has recently shifted away from using galena lead isotopes as a dating technique (except in cases where an uncertainty of up to ± 200 m.y. is still tolerable, as in the Pre-Cambrian), and into using them more empirically as natural tracers, isotopically comparing lead from ore deposits with the lead extracted from various potential source rocks. An example of this type of application is that of Doe and Delevaux (1972).

Sulphur isotopes

In contrast to lead isotopes, sulphur isotopes vary in nature because small differences in the properties of the various isotopes result in their becoming fractionated during many geochemical reactions. Sulphur also has four stable isotopes but only 32 S and 34 S are present in sufficient abundance to be measured accurately.

Because sulphur from the sulphides in meteorites is very constant in isotopic composition, the Canon Diablo Troilite (CDT) has been accepted as the international standard, and all sulphur isotope measurements are quoted as δ values in per mil units (%0) relative to this standard:



Text-fig. 2. "Ordinary" lead isotope growth curve showing position of South Pennine leads

$$\delta^{34}S = \left(\frac{(^{34}S/^{32}S)sample}{(^{34}S/^{32}S)CDT} - 1\right) \times 1000(\%_0)$$

A positive δ value indicates that the sample is enriched in ^{34}S relative to CDT; a zero δ value that it is isotopically identical to CDT, and so on.

Broadly, sulphur originating from deep in the earth's crust or upper mantle has δ values near to zero per mil, whereas sulphur that has been involved in low temperature reactions at the earth's surface usually exhibits a broad scatter of δ values (Jensen, 1967).

Analytical Methods

The lead was purified by lead iodide precipitation followed by two dithizone extractions (Tilton et al., 1955). Isotopic analysis was performed using an A.E.I.MS5 mass spectrometer, an ion beam being obtained from lead sulphide on a rhenium filament (Doe et al., 1967).

The mass spectrum was scanned in a series of continuous sweeps, the isotope ratios being obtained from the digitalised output using a polynomial curve-fitting program (Robertson, 1969) on a KDF9 computer. The Broken Hill standard lead (1003B) was analysed regularly, and correction factors, obtained by dividing the results for each ratio of the standard by the accepted value published by Cooper *et al.* (1969), were used to normalize our ratios. Replicate analyses of the standard, together with duplicate analyses on several samples, indicate our precision to be ± 0.05 for $^{206}\text{Pb}/^{204}\text{Pb}$ and $^{207}\text{Pb}/^{204}\text{Pb}$ and ± 0.15 for $^{208}\text{Pb}/^{204}\text{Pb}$. Uncertainty in measurement of the small ^{204}Pb peak was the most important source of error. This causes the preferred scatter indicated by the error bars shown in text-figs 3 and 4.

Sulphur was converted from galena to silver sulphide and then oxidized to sulphur dioxide in a stream of pure oxygen at 1200°C (Rafter, 1957). It was analysed on an AEI MS20 double-collecting mass spectrometer. The precision of the analyses, obtained by comparing replicate analyses of the standard and duplicate analyses of other samples, was about $\pm 1\%$. This is below that typically achieved by established laboratories (\pm 0.2%)

Results and Discussion

All lead isotope results now available for the South Pennines and adjacent areas, including those of Moorbath (1962), are presented in text-fig. 3 and table 1 (p.300). Sample locations are shown in text-fig. 1. Moorbath's results have been corrected so as to be directly comparable with the new ones, using a correction factor independently obtained by Mitchell and Krouse (1971).

Lead specimens from the South Pennines are isotopically extremely homogeneous, the total range in the ratio $^{206}\text{Pb}/^{204}\text{Pb}$ being barely larger than the analytical variation. Such isotopic homogeneity indicates that the lead in the various galena samples had every opportunity to mix before it was precipitated, and so was almost certainly derived from sources some distance from the present location.

The more extensive and precise results essentially confirm Moorbath's results (1962) for the isotopic compositions of galena deposits in the South Pennine Orefield.

Although no detailed study of a single vein has been undertaken, the homogeneity of the results over a wide range of ore types and deposits indicates that, at this level of precision, it will probably not be possible to distinguish different mineralization pulses using lead isotopes. Also, no district-wide isotopic zonations are observed.

Lead isotope compositions from the Ecton Copper Mine, the vein in Coal Measures at Whaley Bridge, and from the Permian Magnesian Limestone at Bulwell are indistinguishable from those in the main orefield. Lead samples from the Triassic deposits in Leicestershire are isotopically slightly more heterogeneous than those from the South Pennine Orefield, but have similar average composition.

On present evidence, therefore, lead isotopes indicate that the Permian and Triassic galena either had the same source as, or were eroded from, the South Pennine ores. There is no way of distinguishing between these possibilities using lead isotopes.

Text-fig. 2 shows that the lead isotope analyses fall mainly to the right of the ordinary growth curve of the Russell-Stanton-Farquhar model. They are therefore anomalous, and must have evolved in some crustal environment for a significant period before being mineral-

Taken alone, the South Pennine results are too homogeneous to be interpretable using an anomalous lead model such as that proposed by Kanasewich (1968), but taken together with results from the North Pennines and adjacent areas (Mitchell and Krouse, 1971), there is evidence for a real isotopic trend (text-fig. 4) even though more data for the Alston Block are needed.

This trend is similar to that observed in the geologically similar Mississippi Valley deposits (Heyl, 1969), although the latter are isotopically much more heterogeneous, even on the scale of a single orefield. The isotopic heterogeneity in the Mississippi Valley has been ascribed to mixing of lead from different sources, and/or to derivation of lead from a geochemically variable source rock. However, until studies of the isotopic composition of trace leads in various potential source rocks for galena of the Pennine orefields are undertaken, as was done by Doe and Delevaux (1972) for southeast Missouri, further speculation on the significance of the observed isotopic trend is unwarranted.

Regional zonations have been observed across several Mississippi Valley Orefields (Heyl et al. (1971), where southerly ore deposits have slightly more radiogenic elements than the northerly ones. The effect is much smaller than observed in the Mississippi Valley districts, and is so near to the limit of significance at the level of precision achieved (similar to that achieved in this work) that it cannot yet be regarded as proven.

Although there is a hint of a similar zonation in galena in the Pennine orefields, the South Pennines having more radiogenic lead than the Alston Block, this cannot yet be proved on a wider basis, owing to insufficient analyses being available. There are only three analyses by Moorbath (1962) from veins in Lower Carboniferous limestones as far apart as the Mendip Hills, Halkyn Mountain and Hensingham in Cumberland which are isotopically indistinguishable from the South Pennine leads.

The five sulphur isotope analyses were made as a pilot study to test the potential of the method, and so firm conclusions cannot be drawn, especially as the precision obtained was not good.

The wide range (+3.2 to -8.4% $_{\circ}$) of $_{\delta}$ values for sulphur isotopes from the five South Pennine galena samples strongly indicates that further study has great potential. Such a wide range is indicative of sulphur that has fractionated at low temperatures. The heterogeneity of the sulphur isotopes contrasts sharply with the homogeneity of the lead isotopes. The apparent decrease in $_{\delta}^{34}$ S from north to south should be regarded as pure coincidence.

The spread of δ values lies within that observed by Solomon *et al* (1971) in the North Pennine (Alston Block) Orefield. It contrasts with although includes, the narrow scatter observed by Mitchell and Krouse (1971) at Greenhow-Skyreholme.

Further studies, especially the analysis of barite δ ³⁴S-values would be of great interest.

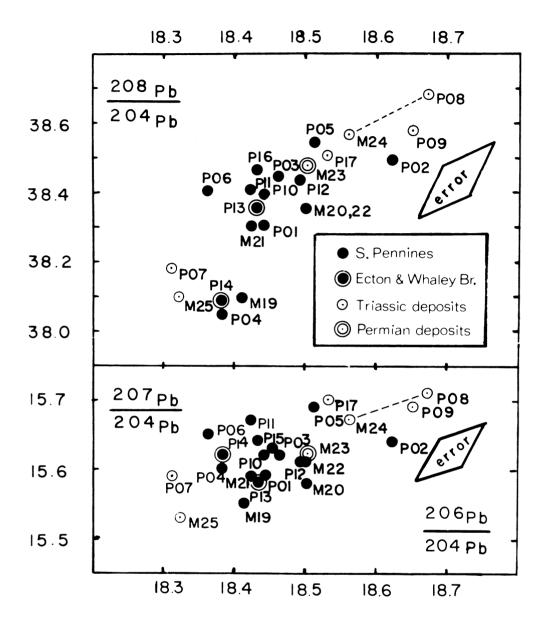
Conclusions

The homogeneity of lead isotopes in South Pennines galenas indicates that isotopic mixing must have occurred before deposition, and so the lead probably originated some distance away from its present location.

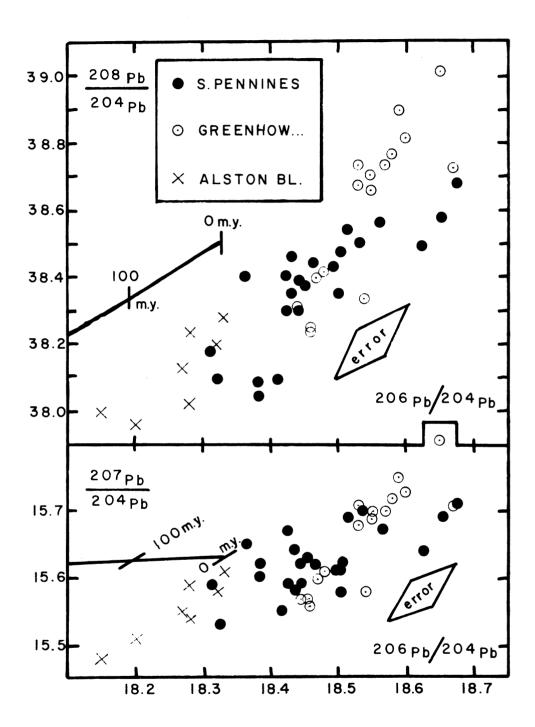
The lead isotopic compositions of galenas in small deposits in adjacent Permian and Triassic rocks are not significantly different from those of the South Pennines. Therefore, the former either had the same source as or were eroded from the latter.

A pilot sulphur isotope study points to wide δ S-values and indicates that further sulphur isotope study might have great potential. Precipitation of the ore minerals probably took place at low temperatures.

Taken together, the lead ores of the Pennines define a definite isotopic trend similar to that defined by the isotopically more heterogeneous Mississippi Valley deposits. Isotopic studies of trace leads extracted from various potential host rocks are required before the significance of this trend can be further elucidated.



Text-fig.3. South Pennines and adjacent areas lead isotope data



Text-fig.4. Combined Pennine lead isotope data

TABLE 1

LEAD AND SULPHUR ISOTOPE RESULTS

Location, O.S. Grid Reference, Collected by	SOUTH PENNINES OREFIELD ²	Odin Mine near Castleton, SK133835, D. K. Robertson.	Treak Cliff Cavern near Castleton, SK136832, G. Lane.	Prospect at Smalldale Head near Bradwell, SK165813, G. Lane.	Ladywash Mine near Eyam, SK218775, T.D. Ford.	Deep Rake on Longstone Edge, SK 218736, T.D. Ford.	Mogshaw Rake near Sheldon, SK183679, T.D. Ford.	Raper Mine, Long Rake, near Youlgreave, SK216653, T.D. Ford.	Spar Mine, Long Rake, near Youlgreave, SK186642, T.D. Ford.	Wheel's Rake, near Youlgreave Moorbath, (1962)	Millclose Mine, near Matlock Moorbath, (1962)	Riber Mine, near Matlock Moorbath, (1962)	Moletrap Mine, near Cromford Moorbath, (1962)	Dene Quarry near Cromford, SK289563, G. Lane.	Golconda Mine near Brassington, SK 249551, T.D. Ford	Minninglow near Parwich, e.l.u. G. Lane.
${\rm Type}^{-1}$	SOU TH	Λ	Ь	Ь	Λ	Λ	Λ	Λ	Λ	Λ	Λ	Λ	^	Ξŧ	Ĺτι	Ъ
$\delta^{34} { m S}(\%_{0})$		+3.2	+2.1	+0.2	- 2. 5										-8.4	
208 Pb 204 Pb		38.40	38.41	38.44	38.50	38.55	38.41	38.45	38.05	38.31	38.10	38.36	38.36	37.88	38.31	38.47
207 Pb 204 Pb		15.62	15,67	15.61	15.64	15.69	15,65	15.62	15.60	15,59	15, 55	15.57	15.61	15.63	15.59	15.64
206 Pb 204 Pb		18.44	18.42	18.49	18.62	18.51	18,36	18,46	18,38	18.42	18.41	18,50	18.50	18,45	18.44	18.43
Sample		P10	P11	P12	P02	P05	P 06	P03	P04	3 M21	3 M19	3 M20	3 M22	P15	P01	P16

DEPOSITS IN CARBONIFEROUS ROCKS OUTSIDE SOUTH PENNINES OREFIELD

¹ Type of deposit: V = vein (rake); P = pipe; F = flat.

 $^{^2}$ Deposits listed in order north to south.

³ All Moorbath's (1962) analyses have been corrected so as to be directly comparable with our data using a correction factor INDEPENDENTLY obtained by Mitchell and Krouse (1971).

⁴ See King and Ludlam (1969) for further details. e.l.u. = exact location unknown.

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