

## THE NATURAL OCCURRENCE OF GALENA-CLAUSTHALITE SOLID SOLUTION SERIES\*

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### ABSTRACT

A study of the sulfides associated with the vanadium-uranium deposits of the Colorado Plateau has revealed that a complete natural solid solution series exists between galena (PbS) and clausthalite (PbSe). Twenty analyzed samples range in composition from 0.04 to 93.7 molecular per cent PbSe in PbS with a concomitant variation of the unit cell from 5.930 Å to 6.127 Å. A plot of these values using the  $a_0 = 5.936$  Å for PbS and  $a_0 = 6.140$  Å for PbSe indicates an almost straight line variation of the cell edge between galena and clausthalite. Mineragraphic and x-ray study shows these minerals to be true solid-solution phases; exsolution of one end member in the other was not found. Semiquantitative spectrographic analyses indicate that the minor elements in these sulfides are similar to those found in galena with some variations resulting from the environment of deposition.

### INTRODUCTION

Recent studies (Coleman and Delevaux, 1957) of the sulfides associated with sedimentary-type uranium deposits from the western United States indicate that selenium is commonly present in the sulfide structures substituting for sulfur. A systematic study of these various sulfides shows that galena usually contains more selenium than the associated sulfides, particularly where these minerals have crystallized from selenium-rich solutions.

Earley (1950) has shown that a complete solid solution series, PbS-PbSe, can be produced by pyrosyntheses. The fusions were made at intervals of 20 atomic per cent between the end members, galena and clausthalite. A plot of the unit cell dimension against composition produces an almost linear variation illustrating that the two isostructural compounds could form a continuous series.

The presence of selenium has often been reported in galena (Fleischer, 1955); however, the combined analytical and x-ray data were insufficient to definitely establish the natural occurrence of the PbS-PbSe solid solution series.

Bergenfelt (1953) has reported 50 ppm to 1.5 per cent Se in galena from the Boliden mine, Sweden, but the unit cell was not determined on these samples. Heier (1953) has used the unit cell size of galena to determine its selenium content from Earley's synthetic data. He determined the molecular per cent of PbSe as 80, 79, and 93 per cent on three samples from Norway. Since chemical analyses were not made on Heier's samples, these may not be used to further illustrate the series as other elements may have been substituting in the PbS structure.

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Within the collection of sulfides from the Colorado Plateau uranium-vanadium deposits analyzed for selenium it was found that 19 samples ranged in composition between galena and clausthalite. Fortunately, these specimens exhibited enough range in selenium content to illustrate the existence of a galena-clausthalite series in nature.

#### LOCATION AND DESCRIPTION OF SAMPLES

The majority of the galena-clausthalite samples were obtained from the vanadium deposits in the Entrada sandstone of Jurassic age (Table 1). In most of these layered deposits thin sulfide bands asymmetrically border the tabular ore bodies. These bands may be continuous along strike for several hundred feet.

The galena-clausthalite within the thin sulfide bands forms as interstitial grains concentrated in a thin zone to produce a continuous banding effect (Coleman and Delevaux, 1957). The bands are well developed in the Rifle and Garfield mines, Garfield County, Colorado, where the majority of the samples were obtained through the cooperation of R. P. Fischer and Theodore Botinelly, U. S. Geological Survey. Similar galena-clausthalite bands are developed in the Bear Creek mine, San Miguel County, and Good Hope mine, La Plata County, Colorado (Table 1).

The almost pure clausthalite was obtained by A. D. Weeks and Lee Eicher, U. S. Geological Survey, from the Corvusite mine, Grand County, Utah, a uranium-vanadium deposit in the Morrison formation of Jurassic age. Here the clausthalite formed a quarter-inch layer bordering an uranium-rich coalified log. The seleniferous galena, from the Darwin mine, California, was collected by Wayne E. Hall, U. S. Geological Survey.

#### ANALYTICAL PROCEDURES

Each sample was treated in a similar manner in its purification. The rock containing the sulfide-selenide was crushed to pass through a 100-mesh sieve and concentrated by the use of a superpanner. The superpanned concentrate was further purified by centrifuging in methylene iodide. The abundance of heavy minerals in these sulfide-bearing sandstones along with alteration of the sulfide-selenide to secondary lead minerals contribute impurities to the concentrates.

Each purified sample was then analyzed for selenium by the distillation method described by Robinson and others (1934). The selenium content was determined gravimetrically except in the one sample (74-D) where selenium was determined colorimetrically. The analytical error estimated for the gravimetric determination of selenium by this method is  $\pm 5$

TABLE 1. LOCATION AND DESCRIPTION OF SAMPLES<sup>1</sup>


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AW-165-52	—Corvusite mine, Grand County, Utah; selenide rimming coalified log associated with uranium ore. Morrison formation. U. S. National Museum No. 112933.
306-RGC-56	—Rifle mine, Garfield County, Colorado; single sulfide band in No. 1 vein. Navajo (?) sandstone.
303-RGC-56	—Rifle mine, Garfield County, Colorado; single sulfide band in No. 1 vein. Navajo (?) sandstone.
302-RGC-56	—Rifle mine, Garfield County, Colorado; single sulfide band in No. 1 vein. Navajo (?) sandstone.
305-RGC-56	—Rifle mine, Garfield County, Colorado; single sulfide band in No. 1 vein. Navajo (?) sandstone.
73A-RGC-55	—Rifle mine, Garfield County, Colorado; single sulfide band in No. 2 vein. Entrada sandstone.
74-RGC-55	—Rifle mine, Garfield County, Colorado; single sulfide band in No. 1 vein. Navajo (?) sandstone.
300-RGC-56	—Rifle mine, Garfield County, Colorado; single sulfide band near junction of No. 1 and No. 2 veins. Entrada sandstone.
304-RGC-56	—Rifle mine, Garfield County, Colorado; multiple sulfide bands in No. 1 vein. Navajo (?) sandstone.
74C-RGC-55	—Rifle mine, Garfield County, Colorado; single sulfide band in No. 1 vein. Navajo (?) sandstone.
69-RGC-55	—Rifle mine, Garfield County, Colorado; thin multiple bands in No. 1 vein. Navajo (?) sandstone.
72-RGC-55	—Rifle mine, Garfield County, Colorado; single broad band in No. 2 vein. Entrada sandstone.
452-RGC-56	—Good Hope mine, La Plata County, Colorado; multiple fine bands in Entrada sandstone.
75-RGC-55	—Bear Creek mine, San Miguel County, Colorado; broad zone of disseminated sulfide in Entrada sandstone.
65-RGC-55	—Garfield mine, Garfield County, Colorado; single sulfide band in No. 2 vein. Entrada sandstone.
301-RGC-56	—Rifle mine, Garfield County, Colorado; extremely finely disseminated band in No. 1 vein. Navajo (?) sandstone.
66-RGC-55	—Garfield mine, Garfield County, Colorado; double sulfide band in No. 3 vein. Entrada sandstone.
68-RGC-55	—Garfield mine, Garfield County, Colorado; broad double banding in No. 2 vein. Entrada sandstone.
H-2	—Darwin mine, Inyo County, California; sulfide in lead-zinc vein type deposit, massive galena in Essex vein.
74D-RGC-55	—Rifle mine, Garfield County; galena in brecciated fracture zone, probably hydrothermal in origin. Entrada sandstone.

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<sup>1</sup> Exact locations for samples from the Rifle and Garfield mines are plotted on mine maps compiled by R. P. Fischer (personal communication), U. S. Geological Survey.

per cent of the reported value. When large amounts of selenium are present ( $> 20$  per cent Se) the error may be higher, possibly  $\pm 10$  per cent.

The unit cell dimensions were determined from  $x$ -ray diffractometer charts, using the method described by Parrish, *et al.* (1953).

A split of the original sample was then analyzed by the semiquantitative spectrographic method described by Waring and Annell (1953).

Each sample was carefully checked by mineragraphic techniques to establish the presence of a single phase. Exsolution of PbSe in PbS or PbS in PbSe was not observed in any of the samples. This was verified by the  $x$ -ray examination as all the peaks of the important reflections are well defined; no doublets were observed. To check the possibility that the diffractometer was not sensitive enough to distinguish exsolved phases, several mechanical mixtures of galena and clausthalite were made. In all these mixtures it was possible to distinguish the individual peaks of the two end members.

#### RESULTS

The 20 analyzed samples range from 0.04 to 93.7 molecular per cent PbSe in PbS with a variation in the unit cell from 5.930 Å to 6.127 Å (Table 2). To establish the ideal linear curve between the two end members, the precise unit cell of pure galena (PbS) and clausthalite (PbSe) should be known. Earley (1950) used 5.923 kX for pure galena and 6.110 kX for pure clausthalite. These values (converted from kX to Å) along with those determined for the intermediate synthetic members produce an almost linear variation. However, it is not clear on what type of material Earley determined the unit cell of galena PbS—synthetic or natural. The value used for clausthalite is somewhat open to question as it was not analyzed and the density is low. A small amount of sulfur substituting for selenium in clausthalite would produce a marked change in the unit cell determination.

The value used in this study for galena (PbS) is  $a_0 = 5.924$  kX, determined by Wasserstein (1951) on pure analyzed material from Joplin, Missouri. As pointed out by Wasserstein, bismuth may enter the galena and proxy for Pb producing a concomitant decrease in cell size, thus unit cell parameters on unanalyzed material are of no value in establishing the cell size of end members.

The true cell size of PbSe is difficult to establish as there are many conflicting values reported in the literature. Unit cell measurements for synthetic PbSe have been reported as  $a_0 = 6.14$  Å (Ramsdell, 1925),  $a_0 = 6.128$  Å (Nozato and Igaki, 1955),  $a_0 = 6.135$  Å (Goldschmidt, 1926), and for natural clausthalite the values reported are  $a_0 = 6.162$  Å (Ols-hausen, 1925),  $a_0 = 6.124$  Å (Swanson and others, 1955).

The values given by Ramsdell and Goldschmidt are in fairly close

TABLE 2. UNIT CELL AND SELENIUM DETERMINATIONS OF THE GALENA-CLAUSTHALITE SERIES

Sample No.	Per cent Se <sup>1</sup>	Molecular per cent PbSe in PbS	Unit cell in Å <sup>2</sup>
AW-165-52	25.6	93.7	6.127 ± .003
306-RGC-56	25.1	89.4	6.099 ± .003
303-RGC-56	22.5	78.8	6.120 ± .003
302-RGC-56	21.9	76.4	6.116 ± .003
305-RGC-56	21.1	73.0	6.103 ± .006
73A-RGC-55	18.0	61.1	6.048 ± .008
74-RGC-55	17.0	56.7	6.099 ± .008
300-RGC-56	16.1	54.0	6.043 ± .003
304-RGC-56	15.9	53.2	6.068 ± .005
74C-RGC-55	15.1	50.3	6.048 ± .008
69-RGC-55	14.2	47.6	6.044 ± .008
72-RGC-55	11.8	38.5	6.033 ± .008
452-RGC-56	11.7	38.1	6.029 ± .003
75-RGC-55	10.0	32.2	6.003 ± .008
65-RGC-55	8.66	27.7	5.996 ± .008
301-RGC-56	8.24	26.2	5.981 ± .008
66-RGC-55	8.10	25.8	5.996 ± .008
68-RGC-55	7.09	22.4	5.981 ± .008
H-2	2.11	6.5	5.924 ± .005
74D-RGC-55	0.009	0.04	5.930 ± .008

<sup>1</sup> Relative error of the Se analyses is approximately ± 5 per cent of the reported value. Analysts, Maryse Delevaux and Esma Campbell, U. S. Geological Survey.

<sup>2</sup> CuK $\alpha$  radiation; calculations from x-ray spectrometer charts. The estimated error of the unit cell measurement was established from the average of five  $a_0$  determinations from reflections in the high angle region. The extremes on either side of the average give the estimated error. Measurements by R. G. Coleman and J. R. Houston, U. S. Geological Survey.

agreement and since these were determined on synthetic PbSe, the cell size  $a_0 = 6.140$  Å seems to be the best figure.

Using  $a_0 = 5.936$  Å for galena and  $a_0 = 6.140$  Å for clausthalite as the best values for the end members, a graph was constructed with molecular per cent plotted against unit-cell size. The assumption was made that the relationship was linear. The naturally occurring galena-clausthalites were plotted on the same graph along with Earley's (1950) data on synthetic materials (Fig. 1). In the majority of cases the values determined seem to follow the hypothetical linear trend. The greatest divergence is found near PbSe where the analytical problem of accurate Se determinations becomes critical because of the great difference in atomic weight between sulfur and selenium. In addition to this, impure samples tend to decrease the amount of total selenium (for example, 74-RGC-55, Fig. 1). Only one complete analysis was made (AW-165-52, Table 3), and from

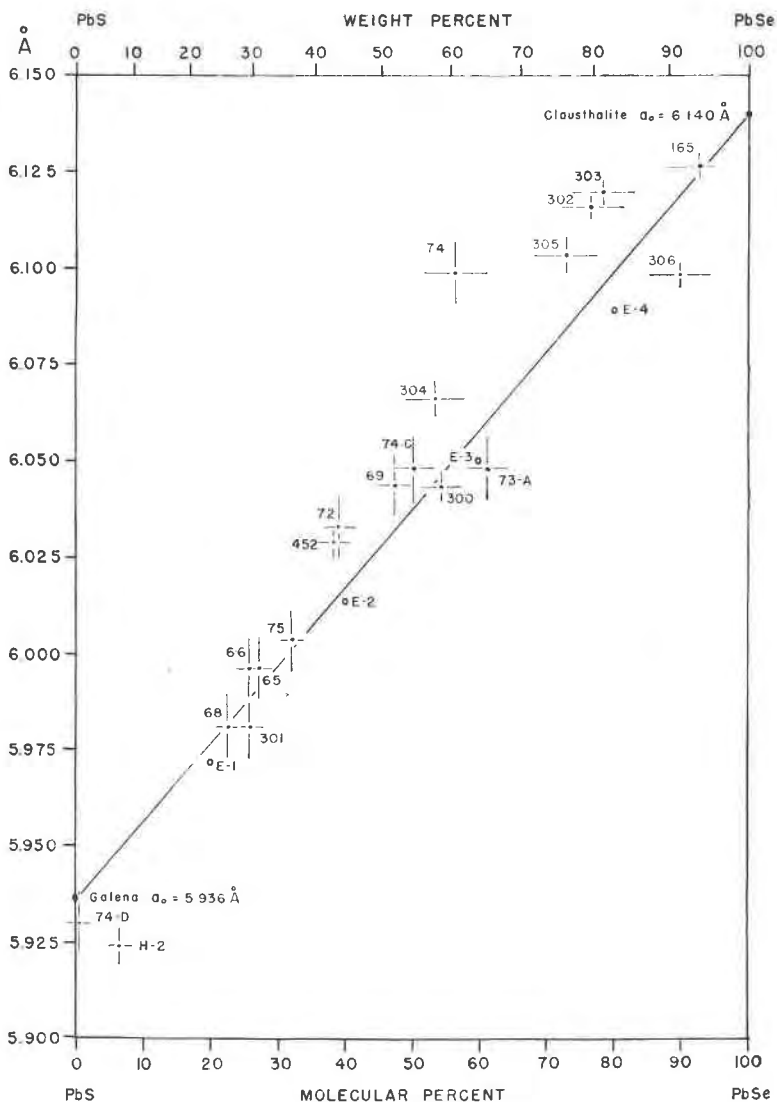


FIG. 1. Plot of unit cell against molecular per cent PbSe in PbS for galena-clausenthalite series. Relative error of the unit cell and selenium determinations are expressed as bars. The points labeled E-1 to E-4 represent the synthetic material made by Earley (1950). Weight per cent PbSe in PbS is given on the top abscissa.

this PbS and PbSe were calculated directly. For the other points in Fig. 1 the molecular per cent PbSe was calculated from the selenium analyses and the remainder was taken as PbS. When using this type of calculation to determine PbSe-PbS values, impurities will give a lower apparent weight per cent PbSe with the same  $a_0$ . The strong divergence of H-2 can be explained by its high bismuth content. X-ray spectrometer analysis on this sample indicates several per cent Bi; and as shown by Wasserstein (1951), substitution of Bi for Pb causes a decrease in cell size. A complete analysis of clausenthalite (AW-165-52) shows that small amounts of sulfur may greatly affect the unit-cell determination (Table 3).

The calculation of the molecular per cent of PbS and PbSe in the

TABLE 3. CHEMICAL ANALYSIS OF CLAUSTHALITE, (AW-165-52)<sup>1</sup>  
(Analyst, A. M. Sherwood, U. S. Geological Survey)

	Weight per cent	Molecular per cent clausenthalite-galena
Pb	72.0	PbSe—93.7
Se	25.6	PbS—5.9
S	0.4	Density 25° C. = 8.01 ± 0.005 (Berman balance)
As	0.6	Density = 8.09 (calculated)
Total	98.6	

<sup>1</sup> Corvusite mine, Grand County, Utah. Material used for analysis and x-ray determination is in the U. S. National Museum, No. 112933.

clausenthalite from the Corvusite mine illustrates that only 0.4 per cent S in clausenthalite accounts for 5.9 per cent galena (PbS). Thus, one must be cautious in selecting natural material for true end members unless a complete analysis has been made.

The pyrosyntheses of the galena-clausenthalite solid solution series by Earley (1950) was accomplished at fusion temperatures; however, data on the PbS-PbSe system at various pressures and temperatures is not available. The majority of these samples were taken from sandstone-type uranium-vanadium deposits suggesting that the formation of this series can be accomplished at fairly low temperatures. Estimates of the temperature of formation of these particular deposits have shown that they may have formed at temperatures similar to that of the enclosing host rock (Coleman, 1957).

#### SPECTROGRAPHIC ANALYSES

Each sample was analyzed by a semiquantitative spectrographic method described by Waring and Annell (1953) to ascertain the minor element content of this group of specimens (Table 4).

TABLE 4. SEMIQUANTITATIVE SPECTROGRAPHIC ANALYSES OF GALENA-CLAUSTHALITE SERIES  
(Analysts: Charles Annell, Joseph Haffty, and Katherine V. Hazel, U. S. Geological Survey)

Sample No.	Element											Impurities <sup>1</sup>
	As	Ni	Co	Cu	Mn	Zn	Mo	Ag	Mg	Fe	Sr	
AW-165-52	0.5 -1	0.01 -0.05	0.01 -0.05	0.1 -0.5	0.001-0.005	—	0.001 -0.005	0.1 -0.5	0.05 -0.1	0.5 -1	—	V.
306-RGC-56	0.1 -0.5	0.01 -0.05	0.005 -0.01	0.01 -0.05	0.01 -0.05	0.05-0.1	0.005 -0.01	0.001-0.005	0.1 -0.5	1-5	0.01 -0.05	Py. dol.
303-RGC-56	—	0.001-0.005	0.001-0.005	0.005-0.01	0.01 -0.05	—	—	0.001-0.005	0.05 -0.1	1-5	0.005-0.01	Py. qtz.
302-RGC-56	—	0.01 -0.05	0.001-0.005	0.005-0.01	0.01 -0.05	—	0.005 -0.01	0.001-0.005	0.05 -0.1	1-5	0.001-0.005	Py. qtz.
305-RGC-56	0.1 -0.5	0.01 -0.05	0.0001-0.0005	1-5	0.01 -0.05	0.05-0.1	0.001-0.005	0.001-0.005	0.05 -0.1	0.5 -0.1	0.001-0.005	Ccp. qtz.
73A-RGC-55	—	—	0.001-0.005	0.001-0.005	0.001-0.005	—	0.001 -0.005	0.001-0.005	0.01 -0.05	0.01-0.05	—	Zr. qtz. clay
74-RGC-55	—	0.001-0.005	0.001-0.005	0.001-0.005	0.003-0.01	—	0.001 -0.005	0.001-0.005	0.1 -0.5	1-5	0.005-0.01	Py. dol. zr.
300-RGC-56	0.1 -0.5	0.01 -0.05	0.01 -0.05	0.005-0.01	0.001-0.005	—	0.01 -0.05	0.05 -0.1	0.05 -0.1	0.05-0.1	0.001-0.005	Qtz.
304-RGC-56	0.1 -0.5	0.01 -0.05	0.001-0.005	0.01 -0.05	0.05 -0.1	5-10	1-5	0.005-0.01	0.1 -0.5	0.5 -1	0.05 -0.1	Dol. v. sp.
74C-RGC-55	—	0.01 -0.05	0.01 -0.05	0.01 -0.05	0.01 -0.05	0.01-0.05	0.01 -0.05	0.01 -0.05	0.1 -0.5	0.5 -1	0.001-0.005	Dol. v. sp.
69-RGC-55	0.01-0.05	0.01 -0.05	0.01 -0.05	0.01 -0.05	0.005-0.01	0.01-0.05	0.001 -0.005	0.01 -0.05	0.1 -0.5	0.1 -0.5	0.001-0.005	Qtz. zr. dol.
72-RGC-55	—	0.005-0.01	0.001-0.005	0.005-0.01	0.01 -0.05	0.01-0.05	0.01 -0.05	0.01 -0.05	0.05 -0.1	1-5	—	Py. zr. qtz.
452-RGC-56	0.5 -1	0.01 -0.05	0.001-0.005	5-10	0.01 -0.05	1-5	0.001 -0.005	1-5	0.005-0.01	0.5 -1	0.01 -0.05	Ccp. sp.
75-RGC-55	—	0.001-0.005	0.001-0.005	0.001-0.005	0.005-0.01	—	—	0.1 -0.5	0.01 -0.05	0.1 -0.5	0.5 -1	Qtz. dol.
65-RGC-55	—	0.001-0.005	—	0.01 -0.05	0.005-0.01	—	0.0005-0.001	0.001-0.005	0.1 -0.5	0.1 -0.5	0.005-0.01	Zr. qtz. dol.
301-RGC-56	—	0.001-0.005	0.001-0.005	0.01 -0.05	0.01 -0.05	—	—	0.01 -0.05	0.05 -0.1	1-5	0.1 -0.5	Py.
66-RGC-55	0.01-0.05	0.01 -0.05	0.01 -0.05	0.01 -0.05	0.001-0.005	—	—	0.01 -0.05	0.05 -0.1	0.05-0.1	0.001-0.005	Qtz. dol.
68-RGC-55	—	0.001-0.005	—	0.01 -0.05	0.001-0.005	—	0.001 -0.005	0.001-0.005	0.05 -0.1	0.1 -0.5	0.005-0.01	Zr.
H-2	—	0.001	—	0.003	0.0003	0.01	—	1.0	0.00003	0.03	0.001	—
74D-RGC-55	1-5	0.1 -0.5	0.1 -0.5	0.01 -0.05	0.01 -0.05	0.1 -0.5	—	0.5 -1	0.01 -0.05	0.1 -0.5	0.001-0.005	Sp.
Sensitivity	0.05	0.001	0.001	0.0005	0.0005	0.02	0.0005	0.00001	0.001	0.001	0.001	—

<sup>1</sup> Recognized impurities are listed without relative amounts; in most instances these do not exceed 10 per cent of the sample. V.—vanadium oxide, Sp.—sphaalerite, Py.—pyrite, Qtz.—quartz, Ccp.—chalcopyrite, Zr.—zircon, Dol.—dolomite.



In general, the minor element content of these galena-clausthalites is similar to those elements present in galenas as summarized by Fleischer (1955). However, several notable differences were observed and it is assumed that unique environment of deposition of the galena-clausthalites may account for these variances in minor element content. Bismuth and antimony are almost completely absent whereas both these elements are commonly reported in galenas from typical hydrothermal deposits. Antimony was found in the samples from the Good Hope and Darwin mines.

The iron, copper, and zinc reported are considered as impurities in the samples, as pyrite, chalcopyrite, and sphalerite were present in many of the sulfide concentrates. Nickel and cobalt are consistently present in about equal amounts ranging from 10 to 500 ppm and it is assumed that these elements must enter the galena-clausthalite structure. One might suspect that the small amount of iron sulfide present could contribute the necessary Ni and Co. However, the Ni-Co content does not vary sympathetically with the iron content.

The silver content of these specimens is similar to what has been reported in the literature (Fleischer, 1955) for galenas. The strong variations are probably related more to the local geochemistry of the sample rather than a function of temperature as suggested by Tischendorf (1955).

Molybdenum is a common element in these sediments and shows a marked affinity for sulfides. The amount of Mo reported is small in the samples but it undoubtedly has been captured in the sulfide-selenide lattice. Insufficient data are at hand to compare this with other galenas.

Magnesium and iron are probably present as impurities, the iron resulting from pyrite and chalcopyrite and the magnesium from dolomite.

Strontium has not been reported as a minor element in galena up to this time; however, in the galena-clausthalites from sedimentary rocks, Sr was found to be present in amounts up to 1 per cent. As the element is strongly lithophile one would not expect it to be present in sulfides. However, SrS has a NaCl type structure and  $\text{Sr}^{2+}$  has an ionic radius 1.27 Å which is very similar to the ionic radius of  $\text{Pb}^{2+}$  (1.32 Å). Strontium bearing minerals were not observed in the host rocks or in the purified concentrates; however, further analytical work is necessary to establish the substitution of Sr for Pb in the galena-clausthalite minerals.

There is no apparent difference in the kind of minor elements found in the PbS-PbSe series when compared to those characteristic of galena. It seems that the minor elements substituting for Pb are not strongly controlled by the sulfur-selenium ratio.

#### CONCLUSIONS

The close adherence of naturally occurring galena-clausthalites to the hypothetical linear variation of unit cell plotted against composition

strongly suggests that a complete isomorphous series PbS-PbSe does exist in nature.

The minor element content of these galena-clausthalites reflects, in part, their peculiar environment, but, in general, the minor element suites are comparable to those found in other galenas.

#### ACKNOWLEDGMENTS

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