



**Geochemical and lead isotopic data from sediment
cores, fluvial tailings, iron bogs, and pre-mining terrace
deposits,
Animas River watershed, Colorado, 1995-1999**

by

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**U.S. DEPARTMENT OF THE INTERIOR
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Denver, Colorado

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INTRODUCTION

The USDA-DOI Abandoned Mine Lands (AML) Initiative (USGS, 1999) is focused on the evaluation of the effect of past mining practices on the water quality and the riparian and aquatic habitats of impacted stream reaches downstream from historical mining districts located primarily on federal lands. This problem is manifest in the eleven western states (i.e., west of 102°) where the majority of hardrock mines having past production are located on federal lands (Ferderer, 1996). In areas of temperate climate and moderate to heavy precipitation, the effect of rapid chemical and physical weathering of sulfides exposed on mine waste dumps and the acidic drainage from mines has resulted in elevated metal concentrations in the stream waters and streambed sediment. The result of these processes is an unquantified impact on the quality of the water and the aquatic and riparian habitats, which may limit their recreational resource value. One of the confounding factors in these studies is the determination of the component of metals derived from hydrothermally altered but unmined portions of these drainage basins (Runnells and others, 1992). Several watersheds have been or are being actively studied to evaluate the effects of acid mine drainage (AMD) and acid rock drainage (ARD) on the near-surface environment (USGS, 1997) (<http://amli.usgs.gov/amli/5year/>).

The Animas River watershed in southwestern Colorado contains a large number of past-producing metal mines that have affected the watershed. Beginning in October 1996, the USGS began a collaborative study of these impacts under the USGS-AML Initiative (Buxton and others, 1997).

In this report, we present the geochemical and lead isotopic results of sediment coring work done between 1995 and 1999 in the Animas River watershed. The goals of this overall effort are: 1) to sample pre-mining streambed sediment to define the pre-mining geochemical baseline, 2) to examine existing iron bogs and beaver ponds as possible traps for metals, and 3) to document the geochemical and lead isotopic character of fluvial mill tailings deposits. Many of the samples we collected fall into one of the three categories above, whereas others capture the transition between streambed sedimentary deposits containing fluvial tailings and those unimpacted by historical mining activity. This transition zone usually is not sharp, so making calculations of the mean and median values for pre-mining streambed sediment requires some interpretation of the data (Church and others, 2000a). This work was greatly enhanced by consultations with our colleagues on the project and individual members of the Animas River Stakeholders Group ARSG (1997) (<http://www.waterinfo.org/arsg/>).

All the data tables in this report are presented as Excel (v. 7) spreadsheet files to facilitate digital transfer and analysis of the data.

THE ANIMAS RIVER WATERSHED

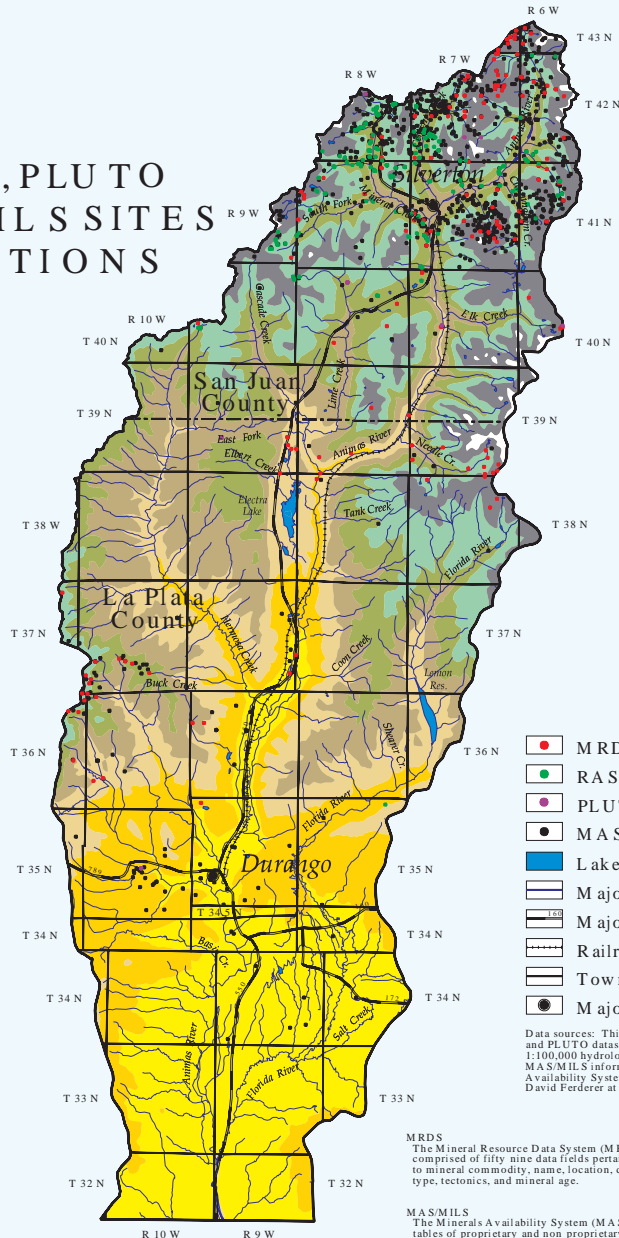
The Animas River watershed (fig. 1) has its headwaters in the mountainous terrain above Silverton, Colorado and drains south into the San Juan River in northern New Mexico. Elevations range from more than 13,000 ft. at the headwaters to less than 6,000 ft. at the confluence with the San Juan River near Aztec, New Mexico. The major population center in the basin is the city of Durango, Colo. The geology exposed at the surface and underlying the basin is varied. Precambrian rocks are exposed in the eastern part of the drainage basin south of Silverton (fig. 2), forming the high rugged mountainous area of the Animas Canyon. Paleozoic, Mesozoic, and Cenozoic sedimentary rocks are exposed in the southern part of the drainage basin. The headwaters of the Animas River watershed are underlain by the Tertiary igneous intrusive and volcanic rocks

Figure 1. (page 2) Map of the Animas River watershed (from Church and others, 1997) showing elevation and mine localities from the USGS MRDS, RASS, and PLUTO databases (Ryder, 1994) and the USGS MAS database (Babitzke and others, 1982; McFaul and others, 2000). The digital elevation model data are from USGS (1990), towns, railroads, and roads are from ESRI (1992), and the hydrology data are from USGS (1989).

Figure 2. (page 3) Geologic map (from Church and others, 1997) of the Animas River watershed area (after Tweto, 1979). The digital geology is from Green (1992); hydrology data are from USGS (1989); the towns, railroads, and roads are from ESRI (1992).

A N I M A S R I V E R W A T E R S H E D S O U T H W E S T E R N C O L O R A D O

M R D S , R A S S , P L U T O A N D M A S / M I L S S I T E S W I T H E L E V A T I O N S



Explanation

- | | | | |
|--|----------------|--|---------------------|
| | MRDS Sites | | > 13,000 ft. |
| | RASS Sites | | 12,000 - 13,000 ft. |
| | PLUTO Sites | | 11,000 - 12,000 ft. |
| | MAS/MILS Sites | | 10,000 - 11,000 ft. |
| | Lakes | | 9,000 - 10,000 ft. |
| | Major Rivers | | 8,000 - 9,000 ft. |
| | Major Roads | | 7,000 - 8,000 ft. |
| | Railroads | | 6,000 - 7,000 ft. |
| | Township Lines | | < 6,000 ft. |
| | Major Towns | | |

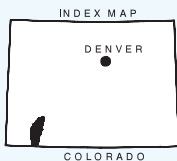
Data sources: This map was compiled using USGS's MRDS, RASS, and PLUTO datasets; 1:250,000 hydrologic units boundaries, 1:100,000 hydrology, 1:250,000 DEM, and ESRI's ArcUSA data. MAS/MILS information was derived from the Bureau of Mines Minerals Availability System. For additional information please contact: David Ferderer at the U.S. Geological Survey, (303) 236 3611.

MRDS
The Mineral Resource Data System (MRDS) is comprised of fifty nine data fields pertaining to mineral commodity, name, location, deposit type, tectonics, and mineral age.

MAS/MILS
The Minerals Availability System (MAS) contains tables of proprietary and non proprietary mineral information. The Mineral Industry Location System (MILS) is a non proprietary table in MAS comprised of sixty four fields including: deposit name, type, status, and location.

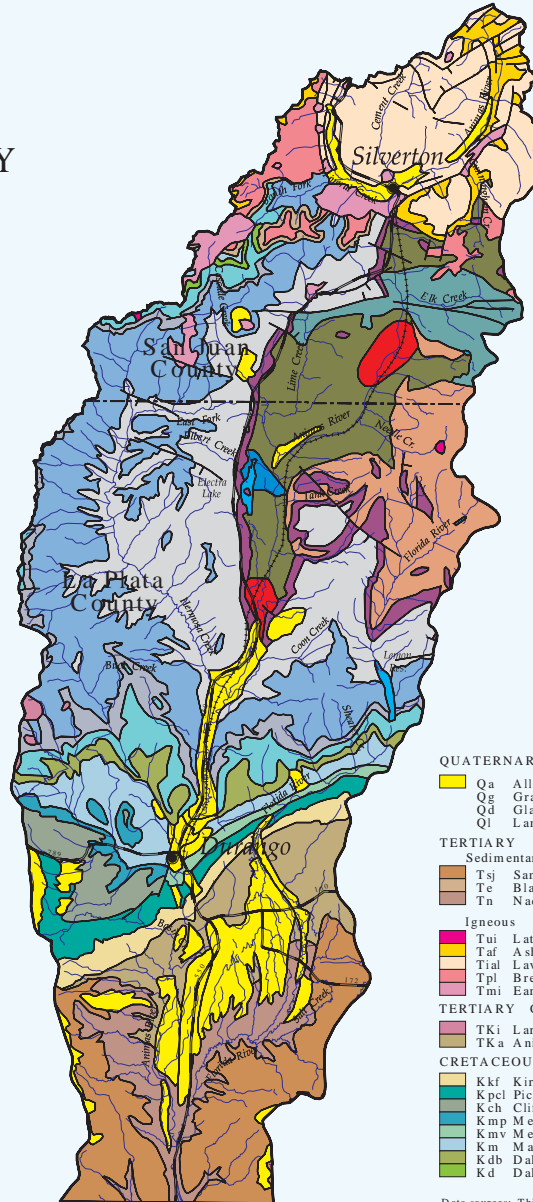
RASS
Rock Analysis Storage System (RASS) is a collection of geochemical analyses from the National Geochemical Database. It contains data on fifty elements and various analytical methods.

PLUTO
This is another analytical subset from the National Geochemical Database. It contains data and analyses on 80 elements and sample source categories including: open pits, mills, prospects, mines, and mine dumps.



ANIMAS RIVER WATERSHED SOUTHWESTERN COLORADO

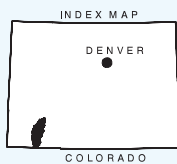
GEOLOGY



Explanation

- | | |
|----------------------------|------------------------------|
| QUATERNARY | JURASSIC |
| Qa Alluvium | Jmw Morrison/Entrada Fms. |
| Qg Gravel | TRIASSIC |
| Qd Glacial drift | Trd Dolores Fm. |
| Ql Landslides | PALEOZOIC |
| TERTIARY | Pc Cutler Fm. |
| Sedimentary | PPrh Rico/Hermosa Fms. |
| Tsj San Jose Fm. | MD Leadville/Dyer Fms. |
| Te Blanco Basin Fm. | PRECAMBRIAN |
| Tn Nacimiento Fm. | Yg Granitic Rocks (1,400 MY) |
| Igneous | Yam Alkalic and Mafic |
| Tui Late Intrusives | YXu Uncompagre Fm. |
| Taf Ash flow | Xg Granitic Rocks (1,700 MY) |
| Tial Lavas | Xfh Gneisses |
| Tpl Breccias | |
| Tmi Early Intrusives | |
| TERTIARY CRETACEOUS | Lakes |
| TKi Laramide Intrusive | Major Rivers |
| TKa Animas Fm. | Major Roads |
| CRETACEOUS | Railroads |
| Kkf Kirtland/Fruitland Fm. | Faults |
| Kpcl Pictured Cliff Fm. | Major Towns |
| Kch Cliff House Fm. | |
| Kmp Menefee Fm. | |
| Kmv Mesaverde Group | |
| Km Mancos Fm. | |
| Kdb Dakota/Burro Fms. | |
| Kd Dakota Fm. | |

Data sources: This map was compiled using USGS 1:250,000 hydrologic unit boundaries and 1:100,000 hydrology, ESRI's ArcUSA, and Ogden Tweto's 1:500,000 Geologic Map of Colorado. For additional information please contact: David Ferderer at the U.S. Geological Survey, (303) 236 3611.



SCALE 1:250,000

(1 inch represents approximately 4 miles)



that formed as a result of a late Tertiary age episode of andesitic to dacitic volcanism followed by a later episode of ash-flows, lava flows and intrusions of dacitic to rhyolitic composition (Lipman and others, 1976). During this later episode of volcanism, the Silverton caldera was formed. Pervasive and intense hydrothermal alteration and mineralization events postdate the formation of the Silverton caldera by several million years (Casadevall and Ohmoto, 1977). This area of the Animas River watershed above Silverton has been extensively fractured, hydrothermally altered, and mineralized by Miocene hydrothermal activity.

Gold deposits were discovered in 1871 on Arrastra Creek above Silverton by prospectors who followed the occurrence of placer gold upstream. Following the signing of a treaty with the Ute Indians in 1873, between 1,000 and 1,500 mining claims were staked in the Animas River watershed upstream from Silverton. Mining activity then spread rapidly throughout the area. Chimney deposits (mineralized breccia pipes) in the headwaters of Mineral Creek (fig. 3) were discovered in 1881 (see fig 4, Church and others, 1997). A railroad was completed from Durango to Silverton in 1882, providing cheap transportation to the smelters in Durango (Sloan and Skowronski, 1975). Mining continued in the Animas River watershed at various levels of activity until 1991 when the Sunnyside Mine was closed. The extent of mining activity within various portions of the Animas River watershed can be estimated from the distribution of mining claims and from Minerals Availability System (MAS) records (Babitzke and others, 1982; McFaul and others, 2000) within the basin (fig. 1). Mineral deposits in several major mining districts have recorded production. Deposits in the Red Mountain district in the northwestern part of the Silverton caldera, the Eureka district in the Eureka graben within the Silverton caldera, and the South Silverton district along the southern margin of the Silverton caldera east of the town of Silverton (Burbank and Luedke, 1968; Leedy, 1971; Casadevall and Ohmoto, 1977) comprised the majority of the mineral production. There are also several porphyry molybdenum deposits that were discovered by drilling in the Mineral Creek area (oral commun., Tom Casadevall, 1996). Some of the porphyry molybdenum deposits are surrounded by large iron bogs at the surface. Iron bogs are found elsewhere within the basin, associated with springs flowing from mineralized and altered areas within the basin (Mast and others, 2000).

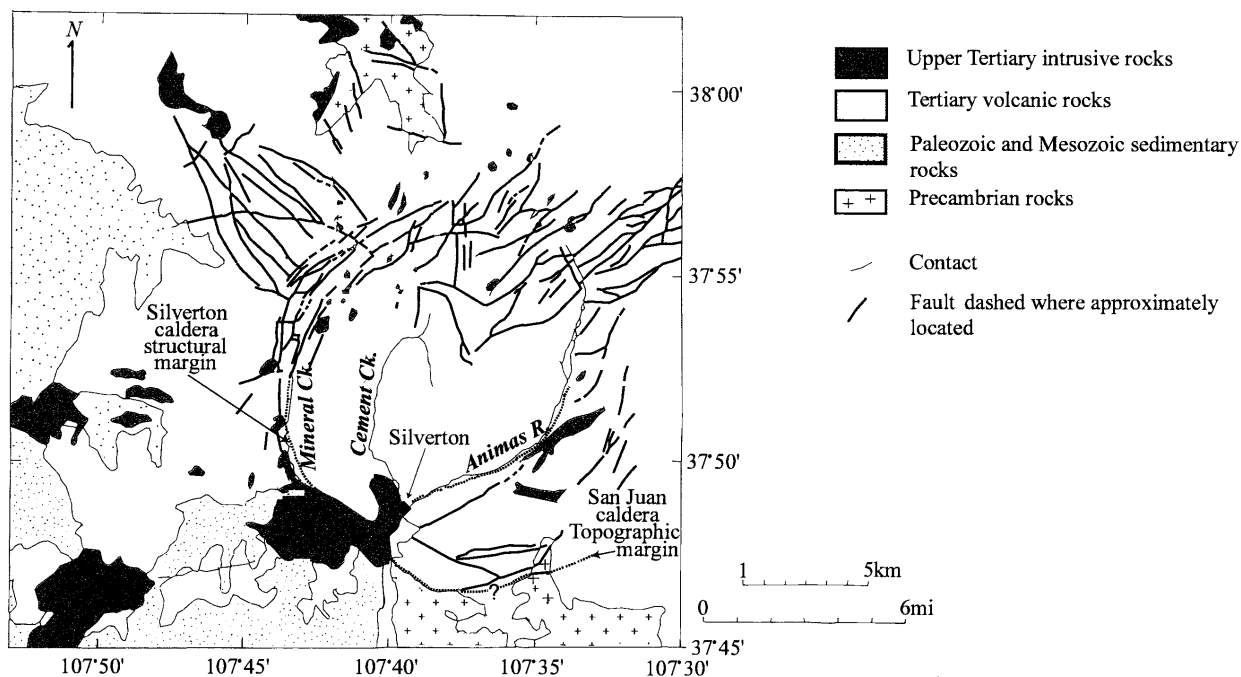


Figure 3. Generalized geologic map of the upper Animas River watershed. The Animas River and Mineral Creek follow the structural margin of the Silverton caldera. In addition to the ring-fractures that were created when the Silverton and the earlier San Juan calderas formed, radial and graben faults, which host much of the subsequent vein mineralization, are shown schematically (modified from Casadevall and Ohmoto, 1977).

METHODS OF STUDY

Sample Collection

Numerous samples were collected to address the three goals outlined above. The sample sites are indicated in figures 4 and 5, and individual sample localities and sample types are listed in table 1. Whereas we have referred to the above samples as cores, some are actually samples of streambed gravels that were dry-sieved to minus-2 mm in the field, and the fine-grained fraction retained, providing a sample directly analogous to the streambed-sediment samples in Church and others (2000b). Dendrochronology (tree ring) samples were taken at several sites to provide minimum ages of the terraces from which pre-mining streambed samples were obtained. Sample descriptions are in tables 2-5. Table 2 contains descriptions for samples from the Mineral Creek drainage, table 3 contains descriptions for samples from Cement Creek, table 4 contains descriptions for samples taken from the Animas River above Silverton, and table 5 contains descriptions for samples taken from the Animas River below Silverton. The geochemical data for the samples are in tables 6-9, in an analogous sequence. Note that the description tables (2-5) contain more subsamples than the analytical data tables (6-9). This is because we provide the descriptions and depths for all subsamples from all core (or lift) samples, while only a percentage of subsamples was actually analyzed and reported.

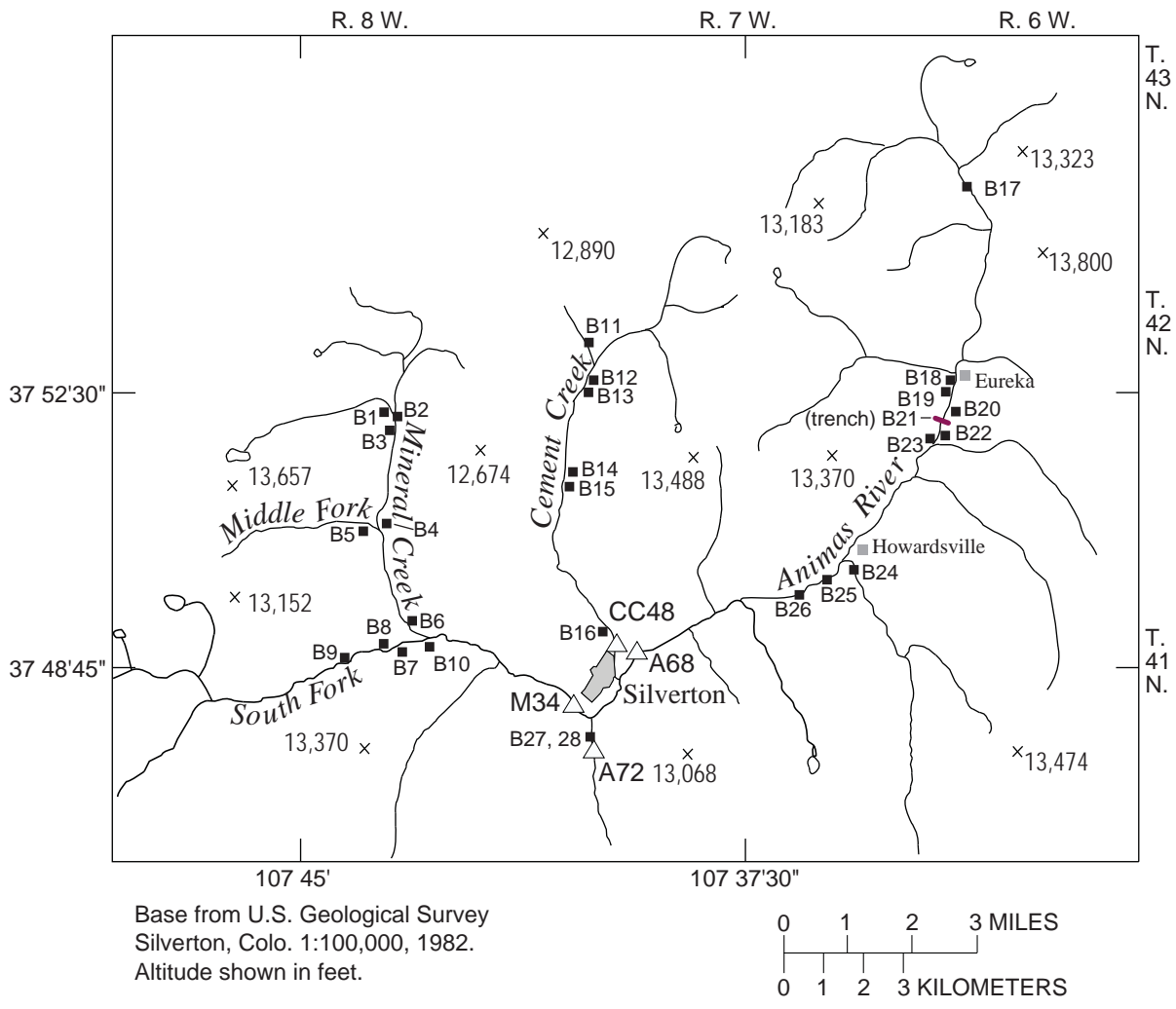
Stream Terraces

Samples of stream terraces were collected and given letter designations, with “a” being the highest sample stratigraphically (tables 2-9). In general, stratigraphy could not be discerned in these gravel deposits, so each sample is really a vertical subsample of the gravel deposit. Each subsample was dry-sieved through a 2-mm (10 mesh) stainless-steel screen in the field. The minus-2-mm fraction for each subsample was retained separately; the larger size fractions were discarded. Samples collected by this method are designated as “lifts” in the data tables. Each subsample was processed separately and the resulting geochemical data evaluated for homogeneous distributions (with depth) of ore-related trace elements (As, Ag, Cd, Cu, Pb, Zn) throughout the terrace gravel deposit. Samples from terrace sediment intervals having a uniform trace elemental distribution were used to calculate the pre-mining geochemical baseline concentrations reported in Church and others (2000a). Selected samples were also used to determine the lead isotopic composition of the pre-mining bed sediments.

Sediment Cores

Sediment cores were collected by driving and subsequently extracting acid-cleaned sections of 2-inch (5-cm) or 4-inch (10-cm) diameter PVC pipe into suitable terrace sites. These cores' material was then divided into subsamples based on stratigraphic (where applicable), color, or grain size differences; the subsamples were analyzed for metal content and lead isotopic signatures. All of the cores through iron bogs and modern beaver ponds were 2-inch cores. Some of the cores through fluvial tailings deposits in the upper Animas River basin below the townsite of Eureka were 4-inch diameter cores (sites B-18, B-19 and B-23). Most of the cores through pre-mining sediment terraces were 2-inch cores, with core 98ABB235 at site B-15 on Cement Creek being the exception.

Recognizing pre-mining streambed sediment in the field based on physical characteristics was not easy; determination of pre-mining sediment was based largely on the geomorphological position of terrace deposits (Church and others, 2000a; K. Vincent, unpublished data, 2000) and then confirmed by subsequent geochemical analysis (based on low, consistent values of ore-related elements). Elevated and variable concentrations of zinc in the core sequence at a particular site in the absence of variation in the concentrations of the other ore-related trace elements listed above were interpreted to be possible groundwater contamination, particularly if associated with elevated manganese. The objective of our sampling strategy was to sample sediments deposited throughout the time period that includes the onset of historical mining activity. Thus, concentrations of ore-related trace elements in the cores or lift samples should decrease with depth or reach a consistent concentration, providing a clear indication of the pre-mining geochemical baseline in the streambed sediment.



- △ Streamflow gaging station
- Background (pre-mining or non-mineralized) sample locality
- x Mountain Peaks

Figure 4. Sample locality map for sediment cores (or lifts) collected in the Mineral Creek, Cement Creek, and upper Animas River basins.

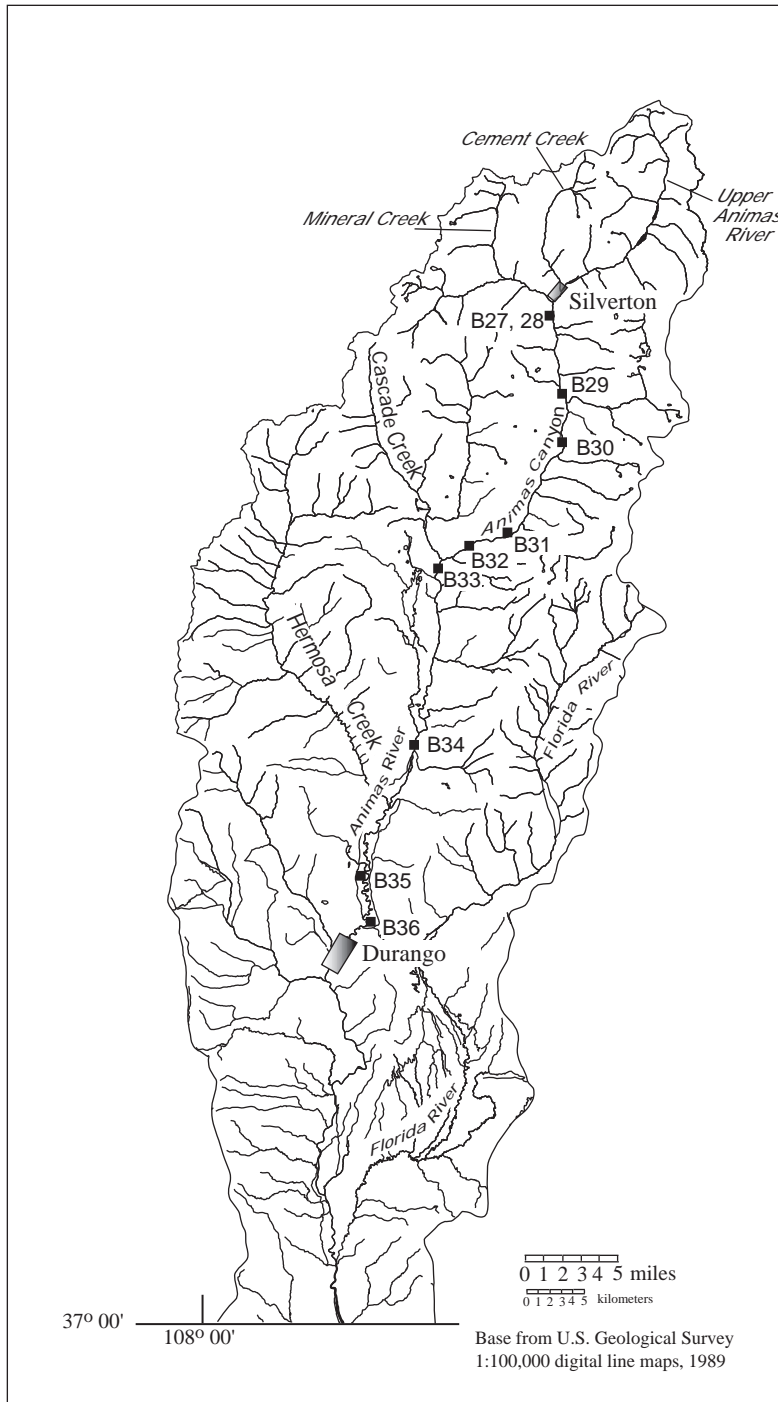


Figure 5. Sample locality map for sediment cores (and lifts) collected in the Animas River watershed below Silverton, Colo. (see fig. 4 for sample localities from the Mineral Creek, Cement Creek and upper Animas River basins).

Fluvial Mill-Tailings Deposits

Mill tailings deposits were sampled along the upper Animas River reach below Eureka. Deposits resulting from jig tailings (prior to 1915) and from flotation tailings (after 1915) occur downstream from the Eureka mill site. Tailings were also deposited on an island downstream from the Mayflower mill at site B-26 during the Gladstone flood of 1911 (Pruess, 1996). Tailings deposits also occur in fluvial deposits downstream from Silverton at site B29 (Elk Park) and at site B36 in an oxbow lake that was an active meander as shown on the 1898 topographic map of the Durango quadrangle (USGS, 1898).

Sample Preparation

Streambed-terrace gravel samples were dried at ambient room temperature (25°C) and sieved to minus-100-mesh (<0.18mm) prior to laboratory analyses.

Core samples were subdivided in the laboratory on the basis of mineralogy, organic content, and apparent oxidation zones. The depth assigned to each subsample is defined as the mid point for that subsample after taking core compaction into account (tables 2-5). Core compaction was determined by measuring in the field the depth of penetration of the core casing, and then measuring the length of the core itself. Individual cores were generally divided into two to ten subsamples, air dried, sieved if the grain size exceeded that of fine sand (0.125-0.25 mm) and the fines retained, and then ground in random order to minus 100 mesh in a vertical pulverizer.

ANALYTICAL METHODS AND RESULTS

Geochemical Analyses

Total Digestion

The samples were digested with a mixed-acid procedure consisting of hydrochloric, nitric, perchloric, and hydrofluoric acids (HCl, HNO₃, HClO₄, and HF) (Crock and others, 1983; Briggs, 1996). This procedure is effective in dissolving most minerals, including silicates, oxides and sulfides; resistant or refractory minerals such as zircon, chromite, and some tin oxides are only partially dissolved. Previous investigations using a variety of materials support the completeness of the digestion (Church, 1981, Church and others, 1987; Wilson and others, 1994).

Results are reported for 34 elements determined by ICP-AES (inductively coupled plasma-atomic emission spectroscopy; (Briggs, 1996) in tables 6-9. The percent ash contents of samples containing organic matter are also provided for samples that were ashed prior to analysis. For those samples, the reported elemental values have been recalculated to the original sample weight. Limits of determination for the ICP-AES total digestion method as well as a statistical summary of mean values, standard deviations, and median values for four National Institute of Standards and Technology (NIST) standard reference materials (SRM-2704, SRM-2709, SRM-2710, and SRM-2711) are given by Fey and others (1999). Limits of determination for the ICP-AES determination are also given below in table 10. Comparisons with certified values for these standards (NIST, 1993a; 1993b; 1993c; and 1993d) are also given by Fey and others (1999). Both analytical precision and accuracy are well within acceptable ranges. Analytical results for the samples reported here are directly comparable with results from the modern streambed-sediment samples reported in Church and others (2000b).

Warm 2M HCl-1 Percent H₂O₂ Partial Digestion

The use of a partial-digestion extraction enables one to determine concentrations of trace elements bound within different mineral phases, whereas a total digestion releases all trace elements in a sample (Chao, 1984). The leaching solution used in this study, a daily-prepared mixture of 2M HCL and 1 percent hydrogen peroxide (H₂O₂), dissolves known elemental sinks such as hydrous amorphous iron-, manganese-, and aluminum oxide minerals, as well as some crystalline iron- and manganese-oxides. (Church and others, 1993). This leach also dissolves water-soluble, ion-exchangeable, and carbonate species. We used a 2.0-g sample in 15 mL of reagent on selected samples to provide solutions for the lead isotopic determinations.

Table 10. Elements determined by ICP-AES (inductively coupled plasma-atomic emission spectroscopy) and their lower limits of determination. These limits apply to a standard sample weight of 0.2 g; when smaller weights are used, the determination limits are correspondingly higher. Analyses performed at the USGS laboratories in Denver, Colo.

Element	Symbol	Lower limit of determination	Element	Symbol	Lower limit of determination
Aluminum	Al	0.01%	Chromium	Cr	1 ppm
Calcium	Ca	0.01%	Copper	Cu	1 ppm
Iron	Fe	0.01%	Gallium	Ga	4 ppm
Potassium	K	0.01%	Lanthanum	La	2 ppm
Magnesium	Mg	0.01%	Lithium	Li	2 ppm
Sodium	Na	0.01%	Molybdenum	Mo	2 ppm
Phosphorous	P	0.01%	Niobium	Nb	4 ppm
Titanium	Ti	0.01%	Neodymium	Nd	4 ppm
Manganese	Mn	4 ppm	Nickel	Ni	2 ppm
Silver	Ag	2 ppm	Lead	Pb	4 ppm
Arsenic	As	10 ppm	Scandium	Sc	2 ppm
Barium	Ba	1 ppm	Strontium	Sr	2 ppm
Beryllium	Be	1 ppm	Thorium	Th	4 ppm
Bismuth	Bi	10 ppm	Vanadium	V	2 ppm
Cadmium	Cd	2 ppm	Yttrium	Y	2 ppm
Cerium	Ce	4 ppm	Ytterbium	Yb	1 ppm
Cobalt	Co	1 ppm	Zinc	Zn	2 ppm

The samples were placed in 90 mL Teflon FEP jars, sealed, and placed in an agitating waterbath at 50° C for three hours to ensure complete removal of the iron- and manganese-oxide coatings from the sediment grains. The leachates were subsequently analyzed by ICP-AES (Appendix III in Church and others, 1993). This partial extraction releases trace elements associated with hydrous amorphous iron- and manganese-oxide mineral coatings and colloidal particles. Mineral coatings such as those observed in the study area contain a significant percentage of the trace elements in a sample (Church and others, 1993, 1997). The lead concentration and isotopic data are reported in [table 11](#).

Lead Isotopic Analyses

The vast majority of the contaminant lead exists within the amorphous iron- and manganese-oxide mineral coatings and colloidal particles (Church and others, 1993, 1994, 1995, 1997); only the leachates from selected samples of the pre-mining streambed sediment were analyzed for lead isotopic compositions. These data are comparable to the lead isotopic data in tables 7 and 8 in Church and others (2000b), and provide a measure of the effect of exposed and near-surface mineralization on the pre-mining streambed sediment geochemical baseline within the Animas River watershed. Lead-isotopic analysis of the leachate solutions greatly reduces dilution effects from lead bound within major mineral phases such as potassium-feldspar and volcanic glassy rock matrix that would be encountered if we had used the total digestion solution from the samples.

The lead-isotopic work was carried out using a VG Sector-54 multi-collector mass spectrometer. Lead was separated using the procedure described below with minor variations (see appendix IV of Church and others, 1993; Tatsumoto and Unruh, 1976; and Unruh and others, 1979). The ICP-AES concentration value for lead ([table 11](#)) was used to calculate the volume of leachate needed to contain approximately 0.5 µg of lead; this aliquot was then evaporated to dryness in a teflon beaker. Then 0.5 to 1.0 mL of 1.0 N hydrobromic acid (HBr) was added to the sample and warmed gently for 5 to 10 minutes. The sample was allowed to cool, was centrifuged, and the supernatant loaded onto an anion-exchange column (0.8-1.0 mL resin volume) using Dowex AG1-x8 anion-exchange resin. The column was washed with 1.2 N HBr and water, and then the lead

was eluted with either 8N HCl or 0.5-1.0 N HNO₃. The eluted lead sample was again evaporated to dryness and then loaded onto a second anion-exchange column with a resin volume of 0.1-0.2 mL. The column was washed with 1.2 N HBr and water, and the lead was eluted with 0.5 N HNO₃. Two or three drops of dilute (0.25-0.5 percent) phosphoric acid (H₃PO₄) were added to the eluted lead sample, and then it was evaporated again to dryness.

Approximately one-half of the sample was taken up in approximately 10 µL of dilute colloidal silica gel, loaded onto a rhenium-ribbon filament, and evaporated to dryness. The filament was then loaded into a solid-source, thermal-ionization mass spectrometer and heated to 1150-1350°C for data acquisition. Most analyses were made using a VG Sector 54, 7-collector mass spectrometer run in “static” mode. A few samples were run on a VG Isomass 54R, single-collector mass spectrometer. No systematic biases have been observed between the two mass spectrometers (Taylor and others, 1999). Analyses of NIST SRM-981 were used to monitor mass fractionation during mass spectrometry (Cantanzaro and others, 1968; Todt and others, 1993). Methods of calculation of the analytical uncertainty and replicate analyses of the SRM-981 lead isotopic standard are presented in Unruh and others (2000). Analytical results are in [Table 11](#).

As demonstrated by Fey and others (1999), data from analyses of the leachates are inherently less reproducible than those from total digestion analyses. As a test of the leaching procedure for lead isotopic analyses, leachates of four NIST standards SRM-2704, SRM-2709, SRM-2710, and SRM-2711 were analyzed for lead isotopic ratios. The lead isotopic analyses of replicate solutions of the four NIST standards are in good agreement with previous work (Church and others, 1993).

Dendrochronology

Cores of live trees were sampled using a standard tree corer. The core was extracted and placed in a plastic sleeve, and sent to the Laboratory for Tree Ring Research at the University of Arizona for dendrochronological analysis. Slabs of dead trees were also sent for analysis in hopes that the year of death could be determined to date the tree stump. The age of the trees provides an estimate of the minimum age of the stream terrace (table 12). Cores or slabs from sample sites B-4, B-6, B-15, B-17, and B-24 were from Engelmann spruce; the slab from site B-30 was from a Mountain cottonwood.

Table 12. Dendrochronology dates from pre-mining background sites, Animas River watershed, Colo. [Dendrochronology done at the Laboratory for Tree Ring Research, University of Arizona, Tucson, Ariz.]

Site	Sample	Year (minimum age of terrace)
B-4	Live tree	1915 (85 years)
B-6	Dead tree stump; tree rings complacent, no chronological data available.	1876 (124 years)
B-15	Live tree.	1858 (142 years)
B-17	Dead tree stump; tree rings complacent, no chronological data available.	1922 (78 years)
B-24	Live tree.	1936 (64 years)
B-30	Dead tree stump; tree rings complacent, no chronological data available.	1957 (43 years)

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