Issues affecting heap biooxidation of low-grade refractory gold ore: Formation of secondary sulfates, ore lithology, alteration and sulfide mineralogy at Gold Quarry, Carlin, Nevada

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in Geology

by

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August, 2010
THE GRADUATE SCHOOL

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Issues affecting heap biooxidation of low-grade refractory gold ore: Formation of secondary sulfates, ore lithology, alteration and sulfide mineralogy at Gold Quarry, Carlin, Nevada

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Abstract

The Gold Quarry mine is located in the Maggie Creek District in the northern section of the southern half of the Carlin Trend, 11 km north of the town of Carlin, Eureka County, Nevada. The primary metal of interest is gold. The majority of the ore is sulfidic-refractory consisting primarily of homogenously distributed extremely small gold particles, generally ranging in size from colloidal to approximately 50Å, hosted primarily as a solid solution within the structural lattice of arsenian pyrite rims which surround some pre-ore pyrite crystals (Arehart et al., 1993). Arsenian pyrite is also commonly found as discrete fine-grain disseminated crystals or in local fine-grain masses or clouds.

High-grade sulfidic refractory ore is processed via roasting methods where as, the low-grade ore cannot be economically processed through the roaster and is instead oxidized by a cost mediated biological heap method on three nominal 800,000 ton pads. The oxidized low-grade ore is then utilized as supplementary mill feed.

Recycling of the biooxidation fluid over time has resulted in a solution highly saturated in sulfate and various metals of which iron and aluminum are the largest contributors to sulfate formation. Consequently, local areas within the heap pad that experience dehydration may experience substantial secondary hydrous sulfate mineral accumulations. These sulfate mineral accumulations locally reduce permeability and may channel the oxidizing solution and induced airflow as well as limiting diffusion within the aggregate itself resulting zones of reduced oxidation and therefore reduced gold recovery. A significant percentage of the heap, visually estimated during visits to unloading pads in 2005 and 2006 at 25 to 30%, was locally isolated from a balanced biosolution and air flow mix due to channelization or the formation of overlying sulfate umbrellas and was consequently only minimally oxidized.

There are many complex factors affecting the efficiency and effectiveness of the heap style biooxidation process. It is clear however, that sulfate formation throughout heap plays a very significant negative roll in the oxidation process. Elevated heap core temperatures, large fluxuations in peripheral heap zone temperatures, and extreme low pH values may temporarily serve to decimate local populations of iron oxidizing microbes. The temperature in the core area of the heap periodically exceeds 87°C. This
is well above the maximum temperature survivable by most bacteria and may also be above the upper limits for the archaea strains present.

Both in-situ columns and laboratory columns were utilized for the study. The laboratory columns were operated under parameters intended to simulate extreme conditions present locally within the heap both spatially and temporally rather than under optimal conditions as is normally done in process labs. This allowed for phenomena such as sulfate channeling of air and fluid to be replicated. Many sulfates were identified including several iron sulfates thought to be the primary contributors to the sulfate formation. Aluminum was found to also be an important element in the formation of sulfate second only to iron. Aluminum occurs as aluminum sulfate (alunogen) as well as in several aluminum-iron sulfates. This complicates solution management as aluminum precipitation requires a higher solution pH than iron.

Lithology, specifically as related to permeability and alteration primarily as related to silicification and argillization, are also major controlling factors regulating overall sulfide oxidation percentage within the heap. Given certain lithologic parameters, oxidizing fluids may only penetrate those sulfides directly exposed at the surface of the aggregate or along fractures. The average sulfide size rarely exceeds 1 mm resulting in a very narrow oxidation halo in low permeability lithologies. Petrographic examination of thin sections from the biooxidized samples commonly display little or no oxidation below the aggregate surface. The most porous and least silicified samples collected rarely display oxidation deeper than 2 to 3 mm below the aggregate surface.

A detailed understanding of sulfide mineral paragenesis and corresponding gold mineralizing events is important in determining the optimum duration of the biooxidation cycle for a specific lithologic host and its associated ore mineral assemblage. Multiple arsenian mineralization events may not all coincide with the gold mineralizing event and therefore may require more or less complete oxidation than currently perceived. Increasing silicification in conjunction with waning ore mineralization may also have caused varying degrees of late stage silica encapsulation or entrainment thereby locally inhibiting biooxidation. This study has also recognized the apparent coprecipitation of silica with the arsenian pyrite rims. Scanning electron microscope and microprobe analysis of arsenian pyrite rims revealed that some of the rims contain silica within the
arsenian pyrite matrix. Locally extensive extremely fine-grain arsenian pyrite is common in certain lithologies or structural zones such as breccias or debris flows and may also be more easily isolated due to silica encapsulation.
Acknowledgements

The assembly and implementation of both the in-situ and laboratory columns required the assistance of many people. I would like to recognize and thank the following people for the assistance with this research project: Tommy Thompson and Peggy Arps for their guidance in formulating and maintaining the focus of the project; Keith Campbell and Matt Hastings for their physical support in building and loading the columns and Keith for assistance with the XRD scans; Carl Marsh for providing his time, parts, and personal tools to aid in the construction of the columns; Jim Murphy for allowing unlimited access to the equipment in his lab; John McCormack for his training and assistance with the SEM and microprobe and XRD; Nelson Publicover for the use of his lab and polisher while making the polished sections; Thom Seal and Jennifer Kerner of the metallurgy department at Gold Quarry for their assistance with the in-situ columns and laboratory analysis; Newmont Mining for supporting the project and supplying the people and equipment for on-site operations; and the CREG sponsors for making this research opportunity possible.
# Table of Contents

List of tables...........................................................................................viii
List of figures...........................................................................................ix
Introduction..............................................................................................1
Scope of project.......................................................................................5
Geology.....................................................................................................6
  - Stratigraphy.....................................................................................6
  - Structure.......................................................................................10
Mineralization..........................................................................................14
Alteration..................................................................................................21
Metallurgy...............................................................................................24
  - Biooxidation facility design ..............................................................24
  - Ore classifications ...........................................................................26
    - Oxide ore ............................................................................26
    - Refractory ore types .............................................................27
Geochemistry..........................................................................................29
  - Sulfide oxidation ............................................................................30
  - Secondary sulfate minerals ...........................................................32
Microbial mineral oxidation mechanisms..............................................36
Biooxidation of refractory gold-bearing ores.........................................39
Procedures...............................................................................................49
  - In-situ columns ..............................................................................49
  - Laboratory columns .......................................................................50
  - Analytical .....................................................................................54
Results........................................................................................................................................58

- In-situ columns .....................................................................................................................58
- Laboratory columns ..........................................................................................................68
- Sulfate analysis ..................................................................................................................81
- Solution chemistry ............................................................................................................85
- Microbial populations ......................................................................................................86
- Aggregate sample acquisition ..........................................................................................90
  - Laboratory columns .......................................................................................................90
  - In-situ columns ...............................................................................................................91
- Sample preparation ............................................................................................................92
- Petrology .............................................................................................................................93
  - Transmitted light ...........................................................................................................93
  - Reflected light .................................................................................................................95
    - Iron sulfides .................................................................................................................95
    - Base metal sulfides and sulfosalts .............................................................................98
  - Textures .........................................................................................................................99
- Microbeam analysis ..........................................................................................................101
  - Iron sulfides ..................................................................................................................101
  - Base metal sulfides and sulfosalts ..............................................................................114
Conclusions................................................................................................................................121
List of tables

Table I. Comparison of original design parameters versus actual initial commercial operational parameters ........................................25

Table II. Comparison of original operational criteria versus initial commercial operational criteria ...........................................25

Table III. Comparison of biooxidation efficiency in the demonstration heap related to ore type and delivery method ..............................26
List of figures

Figure 1. Location of the Gold Quarry Mine ..............................................................2
Figure 2. Stratigraphic column for the Maggie Creek District ...............................7
Figure 3. Structural geology of the Gold Quarry Mine ...........................................12
Figure 4. Location of structural controls for Gold Quarry ......................................16
Figure 5. Cross-section of the Gold Quarry pit .........................................................20
Figure 6. Photo of precipitate “umbrellas” ...............................................................35
Figure 7. Representation of bacterial cell held in place by a biofilm .......................39
Figure 8. Photo of Newmont biooxidation pads at Gold Quarry .........................43
Figure 9. Biooxidation process flow diagram ..........................................................44
Figure 10. Photo of the removal of column 1 at Gold Quarry ...............................50
Figure 11. Photo of eight lab columns .................................................................50
Figure 12. Temperature profile fir in-situ column 4 over time ...............................59
Figure 13. Photo of vent site on top of the heap .....................................................61
Figure 14. Photo of hand sample of sulfate-cemented aggregate .........................62
Figure 15. View from base of heap during unloading ..........................................63
Figure 16. Photo of a small puddle of biosolution with green melanterite crystals ....64
Figure 17. Photo of mass sulfate formed on the base layer of the heap ...............65
Figure 18. Photo of exposed wall of the hole, in-situ column 4 .........................66
Figure 19. Photos of all four in-situ columns following their removal ..................67
Figure 20. Temperature profile of unheated columns over time .........................69
Figure 21. Temperature profile of heated columns over time ..............................70
Figure 22. Column effluent pH over time ..............................................................72
Figure 23. Photos of sulfate cemented ore forming an air channel .....................73
Figure 24. Photos of melanterite crystals..............................................................74
Figure 25. Photo of melanterite stalactite forming from column drain....................75
Figure 26. Melanterite forming on the drain of column by vapor transport.............75
Figure 27. Photo of sulfate-cemented aggregate from the bottom of a column.......75
Figure 28. Column effluent Eh over time..............................................................77
Figure 29. Photos of sulfate minerals on reservoir walls.....................................79
Figure 30. Photos of melanterite and rozenite.......................................................79
Figure 31. Photos of sulfate channel boundary....................................................81
Figure 32. SEM photomicrograph of Magnesiocopiapite.......................................82
Figure 33. SEM photomicrograph of halotrichite ................................................82
Figure 34. SEM photomicrographs of halotrichite needles.....................................83
Figure 35. SEM photomicrograph of alunogen......................................................83
Figure 36. SEM photomicrograph of a spherical vanadium-rich sulfate.................84
Figure 37. Photo of sericite quartz vein cutting and offsetting a quartz vein...........94
Figure 38. Photo of quartz vein cutting a sericite filled stylolite.............................94
Figure 39. Photo of a euhedral pyrite with arsenian rim......................................97
Figure 40. Photo of an anhedral pyrite with arsenian rim....................................97
Figure 41. Photo of pyrite with arsenian rim and disseminated arsenian pyrite........97
Figure 42. Photo of vein marcasite displaying two generations.............................97
Figure 43. Photo of anhedral pyrite with a marcasite overgrowth...........................98
Figure 44. Photo of anhedral arsenopyrite with arsenopyrite overgrowth...............98
Figure 45. Photo of euhedral arsenopyrite with pyrite rim....................................98
Figure 46. Photo of euhedral arsenopyrite with arsenian rim...............................98
Figure 47. Photo of sphalerite replacement of euhedral pyrite.........................................99
Figure 48. Photo of stannite core with sphalerite overgrowth..........................................99
Figure 49. Photo of radial platy pyrite after marcasite...................................................100
Figure 50. Photo of radial platy pyrite after marcasite...................................................100
Figure 51. Photo of framboidal pyrite.............................................................................101
Figure 52. Photo of two euhedral elongate arsenopyrite crystals.....................................101
Figure 53. SEM/BSE image of euhedral pyrite with a very narrow arsenian rim .........103
Figure 54. SEM/BSE image of a very small euhedral pyrite with arsenian rim.........103
Figure 55. SEM/BSE of pyrite with arsenian core........................................................104
Figure 56. SEM/BSE of arsenopyrite overgrowth on arsenopyrite...............................104
Figure 57. SEM/BSE of small pyrite with multiple gradational arsenian rims.........105
Figure 58. SEM/BSE image of pyrite with multiple generations of arsenian rims......105
Figure 59. SEM/BSE image of pyrite with arsenian rim and overgrown by pyrite.......106
Figure 60. Photo of pyrite with arsenian rim and overgrown by pyrite......................106
Figure 61. SEM/BSE image of pyrite with arsenian rim overgrown by marcasite......106
Figure 62. SEM/BSE image of euhedral pyrite with gradational arsenian pyrite ......106
Figure 63. SEM/BSE image of arsenian rim and infill in eroded pre-ore pyrite...........108
Figure 64. SEM/BSE image of fractured pyrite with arsenian pyrite fracture fill.......108
Figure 65. Closer view of center top of Figure 64........................................................108
Figure 66. Closer view of figure 65 showing greater detail...........................................108
Figure 67. SEM/BSE image of euhedral pyrite with gradational arsenian rim........109
Figure 68. SEM/BSE image of zoned pyrite preferentially oxidized.........................109
Figure 69. SEM/EDS elemental spectrum for arsenian rim on pyrite.......................111
Figure 70. Microprobe linear track from arsenian rim to quartz matrix..................112
Figure 71. Microprobe linear track from arsenian rim to quartz matrix..................113
Figure 72. SEM/BSE oblique view of a two generation arsenian rim.....................114
Figure 73. SEM/BSE image of pyrite with pre-arsenian pyrite surface erosion.........114
Figure 74. SEM/BSE image of stannite core with sphalerite overgrowth...............115
Figure 75. TSEM/BSE image of tetrahedrite-tennantite solid solution series.........115
Figure 76. SEM/BSE image of arsenian sphalerite replacing pyrite......................116
Figure 77. SEM/BSE image of pyrite with rare tiemannite rim............................116
Figure 78. SEM/BSE image of pyrite with a new Fe-Ni-As sulfide mineral.............117
Figure 79. SEM/BSE image of euhedral unknown sulfide from Figure 78..............117
Figure 80. SEM/EDS spectrum of unknown new sulfide....................................118
Figure 81. SEM/BSE image of pyrite with epitaxial arsenopyrite overgrowths.......119
Figure 82. Photo of anastomosing pyrite microveinlets in quartz......................119
Figure 83. SEM/BSE images of compositional zoning in epitaxial arsenopyrite......120
Introduction

The Gold Quarry Mine is a Carlin-type ore deposit operated by Newmont Mining Corporation utilizing standard open-pit mining methods. The Gold Quarry Mine is within the Maggie Creek Mining District, located in the northern section of the southern half of the Carlin Trend, 11 km north of the town of Carlin, Eureka County, Nevada (Fig. 1). The primary metal of interest at Gold Quarry is gold. The majority of the ore is sulfidic-refractory consisting primarily of homogenously distributed, extremely small, gold particles generally ranging in size from colloidal to approximately 50Å. The gold is hosted primarily as a solid solution within the structural lattice of arsenian pyrite rims which surround some pre-ore pyrite crystals (Arehart et al., 1993a). Arsenian pyrite is also found as discrete fine-grain disseminated crystals or in local fine-grain masses or clouds.

High-grade sulfidic refractory ore is processed using a large single stage roaster. Since the low-grade ore cannot be economically processed through the roaster, it is instead oxidized by a cost-mediated biological heap method on three nominal 800,000-ton pads. The oxidized low-grade ore is then utilized as supplementary mill feed for the carbon in leach (CIL) circuit and to achieve an optimum blend for the roaster feed (Seal, T., personal communication). The biooxidation heap process, unlike roasting, is very time consuming and typically requires a five-month oxidation period, plus approximately another two to three months for loading and unloading each pad. Currently, as of April 2008, the biooxidation circuit is being indefinitely mothballed and replaced by a flotation circuit.
The goal of this research was to improve the effectiveness and efficiency of the heap style biooxidation process in order to increase low-grade gold recovery percentages and overall heap efficiency. Initial research has endeavored to identify specific local environments and mechanisms within the biooxidation heap that could

Figure 1. Location of the Gold Quarry Mine, Carlin Trend, Eureka County, Nevada (From Norby and Orobona, 2002).
result in reduced oxidation potentials and therefore reduced levels of gold recovery. Lower recovery is due in part to locally decreased heap permeability caused by the precipitation of various secondary sulfates and also to compaction issues. Recycling of the biooxidation fluid over time has resulted in a solution highly saturated in sulfate and various metals of which iron and aluminum are the largest contributors to sulfate formation. This problem was recognized during pilot scale testing and a solution treatment plant was incorporated to alleviate the metal loading issue. However, due to cost constraints, the solution treatment plant was not included in the final biooxidation project design (Temple, 2003). Periodic diversion of a small percentage of the biosolution from the biosolution pond to the tailings dam and replenishment with fresh water has not significantly reduced the metal loadings due to continued replenishment from the active heaps. Consequently, local areas within the heap pad that become dehydrated may experience the accumulation of substantial amounts of secondary hydrous sulfate minerals. These sulfate mineral accumulations locally reduce permeability and may channel the oxidizing solution and induced air flow creating zones of reduced oxidation and therefore reduced gold recovery. Site observations (2005-2006) during heap unloading indicated that a significant percentage of the heap (25 to 30%) was locally isolated from a balanced biosolution and air flow mix due to channelization or the formation of overlying sulfate umbrellas, and was consequently only minimally oxidized. Elevated heap core temperatures, large fluctuations in peripheral heap zone temperatures, extremely low pH values, and desiccation zones may temporarily or permanently serve to reduce local populations of biooxidizing microbes.
Lithology, specifically as related to permeability and degree of silicification, is also one of the major controlling factors regulating overall sulfide oxidation percentage within the heap. Decreased permeability and or increased silicification results in decreased depth of oxidation below the aggregate surface. Given certain lithologic parameters, oxidizing fluids may only penetrate those sulfides directly exposed at the surface of the aggregate or along fractures. The average sulfide size rarely exceeds 1 mm resulting in a very narrow oxidation halo in low permeability lithologies. The unscreened crushing process employed at Gold Quarry dictates a nominal size parameter of 2.5 cm minus. Depending upon lithology, this process also realizes a common bypass fraction of an estimated one to five percent (visually estimated during lab column loading in 2006) that meets or rarely exceeds 8 cm in long dimension, allowing for significant subsurface unoxidized potential. Generally, less permeable more highly silicified ore constitutes a higher proportion of the oversize fraction. The high clay content of the more intensely argillized lithologies also serves to create local zones within the heap of substantially reduced permeability and consequent channelizing or umbrella shielding effects.

Certain aspects of ore mineralogy and petrology may also play an important role in the biooxidation equation. A through understanding of sulfide mineral paragenesis and corresponding gold mineralizing events is important in determining the optimum duration of the biooxidation cycle for a specific lithologic host and its associated ore mineral assemblage. Multiple arsenian mineralization events may not all coincide with the gold mineralizing event and therefore may require more or less complete oxidation than currently perceived. Increasing silicification in conjunction with waning ore mineralization may also have caused varying degrees of late stage silica encapsulation or
entrainment thereby locally inhibiting biooxidation. This study has also recognized the apparent coprecipitation of silica with the arsenian pyrite rims. Certain polygons have realized abnormally low flotation recoveries of 30 to 40 percent (Becker, J., personal communication) which may be the result of this style of arsenian pyrite rim silicification. Locally extensive extremely fine-grain arsenian pyrite is common in certain lithologies or structural zones such as breccias or debris flows and may also be more easily isolated due to silica encapsulation.

**Scope of project**

The scope of the project covered two primary aspects of biooxidation. The first examined the effects of various environmental conditions including temperature, pH, and fluid and air flow cycles on bacterial populations and oxidation efficiency. The second investigated the types of secondary sulfate minerals formed, relative environmental conditions promoting their precipitation, and their local effect on the biooxidation process including permeability and oxidation efficiency.

Results from four in-situ columns installed within the biooxidation heap on pad 8-5C at Gold Quarry and eight environmentally controlled and monitored columns in a laboratory at the University of Nevada, Reno were used to compare simulated laboratory conditions to actual site conditions. Data collected from the laboratory columns included the data recorded from temperature probes in the columns, pH/Eh measurements of drain effluent and reservoir solution, as well as inductive coupled plasma (ICP) analysis and viable microbial counts of the biosolution from the column drains and reservoirs at regular intervals. Biosolution and aggregate samples were collected and preserved for
future RNA/DNA analysis. Secondary sulfate minerals were identified by x-ray
diffraction (XRD), ICP, and scanning electron microscopy (SEM) analysis.

Scanning electron microscope (SEM) backscatter electron (BSE) and energy
dispersion spectroscopy (EDS) analyses of the sulfide and sulfosalt ore mineralogy of one
inch round polished sections and polished thin sections from selected aggregate samples
were also used in order to quantify the relative effectiveness of the oxidation process for
specific aggregate size ranges, lithologies, alteration styles and mineralogical variations.
Microprobe analysis was used in order to verify the style of arsenian pyrite rim
silicification.

**Geology**

**Stratigraphy**

The Gold Quarry Mine is located at the southeastern terminus of the Tusc corridor
in the Carlin window of the central Carlin trend, Maggie Creek Mining District (Norby
and Orobona, 2002) (Fig. 1). The Gold Quarry deposit was partially exposed by erosion
of the overlying Tertiary Carlin Formation revealing Paleozoic thinly bedded to massive
carbonate sequences (Rota and Hausen, 1991). Evans and Cress (1972) reported that only
10 percent of the deposit was not covered by the Carlin Formation. Present day expansion
of the deposit boundaries, primarily to the southeast, would serve to lower this
percentage. Two primary stratigraphic groupings are constrained within these sequences
in the Gold Quarry area: the lower plate section comprising the Hanson Creek, Roberts
Mountains, and Popovich Formations, and Rodeo Creek unit, and the upper plate section
comprising the Marys Mountain Sequence (Harlan et al., 2002) (Fig. 2). The upper-most
Western Siliceous assemblage is not present in the Gold Quarry area. Overlying the
Figure 2. Stratigraphic column for the Maggie Creek District showing gold host lithologies for the deposits in the District (From Harlan et al., 2002).

majority of the south central Carlin trend is the Miocene Carlin Formation and local Quaternary colluvium.

The Silurian-Ordovician Hanson Creek Formation occupies the base of the stratigraphic section in the Gold Quarry area. Only the upper two thirds of the Formation is exposed or has been drilled in the Maggie Creek district. It is described by Harlan et al. (2002) as light to dark gray dolomite that is massive and fine-grained and by Rota (1995) as a massive dolostone or dolomitic limestone with white quartz veins, black chert lenses and a tan-brown sandstone-dolostone cap. Little gold mineralization is associated with the Hanson Creek Formation in the Gold Quarry deposit (Harlan et al., 2002).

Conformably overlying the Hanson Creek Formation is the Devonian-Silurian Roberts Mountain Formation. The Roberts Mountain Formation has been drilled below the northwest wall of the Gold Quarry Mine in the footwall of the Chukar Gulch Fault. In the northeast wall of the Gold Quarry pit the Roberts Mountain Formation is exposed having been thrust over the Rodeo Creek Unit by the Schroeder thrust. The Roberts Mountain Formation is comprised of a coarse-grain silty limestone that displays planer lamination and interbedded calcarenite. The Roberts Mountain Formation consists of four members as defined by Sager and Johnston (2000). The basal DSr4 member is the thickest member at approximately 220 m and consists of silty limestone. DSr3 comprises a silty limestone with interbedded calcarenite accounting for greater than thirty percent of the volume at approximately 66 m thick. DSr2 is approximately 52 m thick and comprises a silty limestone which locally displays a wispy texture due to turbidite flows and contains only minor calcarenite beds. DSr1 at approximately 27 m thick is the thinnest member and consists of a silty limestone without calcarenite beds. Gold
mineralization is located in the footwall of the Chukar Fault primarily in the DSr2 (wispy) member and locally extending into the bounding DSr1 and DSr3 members at greater than 13.7 g/t. The upper portions of the DSr1 and DSr4 and the lower portion of the DSr3 locally host lower grade gold values. The Roberts Mountain Formation also hosts gold in the thrust area on the northeast wall above the Rodeo Creek unit (Harlan et al., 2002).

The Devonian Popovich Formation is exposed in the northwest and northeast walls of the Gold Quarry pit. The Popovich Formation conformably overlies the Roberts Mountain Formation and is comprised of limestones described as micrite, silty, bioclastic, debris-flow, and calcarenite. Where oxidized, it is light gray to brown and where unoxidized, it is light to dark gray. Local carbon rich zones are black. Within Gold Quarry, the Popovich Formation is segregated into three members by Sagar et al. (1999). The approximately 250 m thick basal member Dp3 consists primarily of micritic limestone with interbedded silty, bioclastic, debris-flow, and calcarenite. The approximately 75 m thick intermediate member Dp2 contains primarily thick-bedded calcarenite with lesser bioclastic and silty limestone horizons. The approximately 69 m thick upper member Dp1 of the Popovich Formation consists primarily of thin-bedded silty limestone with minor calcarenite and has locally undergone both brecciation and argillic alteration. The intermediate and upper members locally contain gold mineralization at Gold Quarry (Harlan et al., 2002).

The Devonian Rodeo Creek unit (Fig. 2) is exposed in the northeast wall of the Gold Quarry pit. The Rodeo Creek unit has multiple subdivisions with varying assemblages of limy siltstone, planar light gray to black siliceous mudstone, and gray to
green cherty siltstone with undulating black chert layers. Oxidized zones are light gray to brown and unoxidized zones are light to dark green-gray. Local carbon rich zones are black. Phosphate lenses are more prevalent in the lower 21 m of the unit. The gold grade is generally higher in the lower portion of the unit. The Rodeo Creek unit contains the majority of the gold at Gold Quarry as well as secondary zinc (Harlan et al., 2002).

The basal portion of the upper-plate unit is comprised of the Devonian Marys Mountain sequence and is exposed on the south wall of the Gold Quarry pit. The Marys Mountain sequence has alternating arenaceous limestone and cherty mudstone. Flasure texture is common in close proximity to the Robert Mountain thrust. The basal portion of the Marys Mountain sequence is locally mineralized at Gold Quarry (Harlan et al., 2002).

The Miocene Carlin Formation has a basal tuff that grades upward into sand, silt, and gravels (Harlan et al., 2002) covering approximately 90% of the Gold Quarry exposure (Evans and Cress, 1972). The basal tuff is exposed in the southeast pit wall (Kuiper-Creel, 1998), contains biotite and glass, and is fragmental in nature with scattered gravel lenses (Harlan et al., 2002). The intermediate section consists primarily of locally indurated sand and silt with lesser tuff and gravels. The upper section has debris flows of sand and gravel with variable carbonate cementation. The southeast rim of the Gold Quarry pit also exposes the unconsolidated Holocene Hewettite landslide.

**Structure**

The Carlin window is an area where lower plate rocks were uplifted with the southwest and southeast boundaries created by the Roberts Mountain thrust and the Gold Quarry fault system, respectively (Harlan et al., 2002). The Gold Quarry deposit is located at the southern terminus of the Carlin window and the southeast end of the Tusc
corridor. The primary faults controlling the structural fabric of the Gold Quarry area include the Good Hope fault, Gold Quarry fault system, and the Deep Sulfide Feeder fault zone (Fig. 3).

The Good Hope fault is termed the master fault in the Gold Quarry area due to its association with base metal mineralization and its close proximity to high-grade gold deposits (Harlan et al., 2002). The Good Hope fault traverses the full length of the Tusc corridor with open extension beneath cover at both ends. Multiple deposits are located along the Good Hope fault including Mike, Tusc, Mac and Gold Quarry. Historic mines along the Good Hope fault include the Copper King, Nevada Star, Schroeder Mountain and Good Hope #7 mines (Harlan et al., 2002). Gold mineralization is generally restricted to the footwall within a 600 to 1,500 m zone from the Good Hope fault. Locally, gold mineralization also occurs within a narrow zone of the bounding hanging wall. Mineralization tends to be locally concentrated where high-angle northeast-striking faults intersect the Good Hope fault (Norby and Orobona, 2002). The faults serving as the primary gold mineralization conduits throughout the Carlin Trend predominantly strike north to northwest (Teal and Jackson, 1997). The Good Hope fault is probably a strike-slip reverse fault (Dunbar, 1999) with a strike of N50-60° W and a dip of 45-75° NE. Stratigraphic sequences along the majority of the Good Hope fault exposures display a juxtaposition of the Silurian Roberts Mountain Formation over a folded Devonian Rodeo Creek unit attributed to the Roberts Mountain thrust (Harlan et al., 2002).
Figure 3. Structural geology of the Gold Quarry Mine (From Harlan et al., 2002).
The Gold Quarry fault system consists of numerous faults including the Chukar Gulch, Alunite, Midwest, Bad Attitude, and Rotator. The faults are generally normal, striking N10-35° E and dipping 25 to 80° SE with a 5 km lateral extent and an approximately 335 m cumulative throw. The Good Hope fault displays only minor plan view offset by the Gold Quarry fault system. The Good Hope fault was offset and later brought back to near alignment by left lateral faulting followed by normal faulting along the Gold Quarry fault system (Harlan et al., 2002).

The Deep Sulfide Feeder fault zone is roughly parallel to and approximately 300 m southeast of the Gold Quarry fault system. The Deep Sulfide Feeder fault zone may locally exceed 120 m in width with a strike exceeding 750 m at N20-40° E and a steeper dip to the southeast than the Gold Quarry fault system. Cumulative offset across the Deep Sulfide Feeder fault zone is considered normal but with a possible uplifted central block which may have resulted in a flower-type structure. Individual fault segments are probably discontinuous both along strike and dip. The Deep Sulfide Feeder fault zone apparently does not cut the Good Hope fault (Harlan et al., 2002).

The Roberts Mountain thrust generally separates the upper and lower plate assemblages. The Roberts Mountain thrust consists of a low angle fracture or shear zone and is responsible for the north to northeast striking folds in both the upper and lower plates (Cole, 1995). The Rodeo Creek unit and Popovich Formation likewise display low angle reverse faulting and folding related to the emplacement of the Roberts Mountain thrust (Harlan et al., 2002).

The Alta anticline strikes N60° W and due to its refolding of north to northeast striking folds associated with the Roberts Mountain thrust and similar strike to the Good
Hope fault, it is considered to be related to the compressional stress responsible for the reverse faulting of the Good Hope fault. The Alta anticline locally served as a controlling structure during gold mineralization at Gold Quarry (Harlan et al., 2002). Likewise, the northwest striking Snowbird anticline to the southwest of the Alta anticline also locally hosts gold mineralization.

The Chukar Gulch-Alunite fault zone is also related to the causative compression of the Good Hope fault. The Chukar-Alunite fault zone comprises a brecciated decalcified zone dipping 10-50º SE that is approximately 85 m thick. Rotation due to post-mineral normal faulting has steepened its dip relative to the Good Hope fault. The Chukar-Alunite fault zone served as a primary structural control for the gold mineralization of the Deep West orebody and secondarily for the Deep Sulfide Feeder orebody (Harlan et al., 2002).

Post-mineral faulting consists of generally north to northeast striking normal faults. The Hewettite-Ice and k-9 faults are northwest striking, but underwent post-mineralization reactivation resulting in the formation of the Hewettite graben located in the southeast area of the Gold Quarry pit. The Hewettite-Ice and k-9 faults may have contributed to the gold mineralization event prior to reactivation (Harlan et al., 2002).

**Mineralization**

The majority of the gold mineralization at Gold Quarry is bounded by Chukar Gulch and Alunite faults to the northwest, the Good Hope fault to the northeast, the Deep Sulfide Feeder fault zone to the southeast and the Hewettite-Ice fault to the southwest (Fig. 4). The general trend of the mineralization within the above-mentioned constraints
is N30º E and contains approximately 90% of the economically recoverable gold in the Gold Quarry deposit (Harlan et al., 2002).

The Gold Quarry deposit contains representative examples of the three primary disseminated gold deposit types found within the Carlin Trend. The Quarry Main deposit is a fracture-stockwork type hosted in the siliceous sedimentary Rodeo Creek unit. The Deep West deposit is structurally controlled by the Chukar-Alunite and Bad Attitude faults. Mineralization style consists of both breccia and stratigraphically controlled replacement. The majority of the gold mineralization is restricted to the shallower decalcified and argillized carbonate rocks with decreasing values found in the deeper siltstone and silicified zones. The Deep Sulfide Feeder may have served as the ore fluid conduit for both the Quarry Main and the Deep West deposits. The Deep Sulfide Feeder is near vertical, structurally controlled, and cuts lithologies (Harlan et al., 2002).

The Deep West deposit gold grades run greater than 3.4 to 5.1 g/t locally along the feeder faults. The Deep Sulfide Feeder deposit gold grades range from 3.4 to 34 g/t with an average of 10 g/t (Harlan et al., 2002). The majority of the oxide ore was contained in the shallow Quarry Main deposit and has been mined out. Oxide ore is still present in the southern zone of the Gold Quarry pit, but the bulk of the remaining ore is contained in the deeper Deep West and Deep Sulfide Feeder deposits and is generally sulfidic refractory in nature (Fig. 5).
Figure 4. Location of structural controls for Gold Quarry Main, Deep West, and Deep Sulfide Feeder deposits within the Gold Quarry Mine (From Harlan et al., 2002).
Numerous studies have been conducted on various mineralogical and geochemical aspects of the Carlin trend and Carlin-type deposits in general, as well as specific Carlin-type deposits. The term Carlin-type deposit was coined in the 1970's by F. W. Dickson and A. S. Radtke in reference to other deposits in the area displaying a similar geochemical and mineralogical signature to that of the Carlin Mine (Ferdock, 2004). Currently, nearly 300 mineral species have been recognized in Carlin-type deposits. As of 2004, nine new minerals have been identified from Carlin-type systems and more are under review. Gold Quarry boasts 112 documented minerals including two for which it is the type locality. The Carlin Mine initiated production in 1965, and by 1973 Wells and Mullens (1973) had recognized the relationship between gold and elevated arsenic concentrations associated with pyrite. Many other workers have since confirmed this relationship (Fleet et al., 1989; Cook and Chryssoulis, 1990; Bakken et al., 1991; Fleet et al., 1993; Arehart et al., 1993a; Mumin et al., 1994; Michel et al., 1994; Simon et al., 1999a, b; Savage et al., 2000; Cline, 2001; Emsbo et al., 2003; Palenik et al., 2004).

Refractory gold is primarily associated with arsenian phases of pyrite. Other iron-containing sulfides, such as marcasite \((\text{FeS}_2)\), arsenopyrite \((\text{FeAsS})\), and chalcopyrite \((\text{CuFeS}_2)\) may locally contain trace to minor amounts of gold (Ferdock, 2004). A study by Arehart et al. (1993a) reported that arsenian marcasite with equal wt percent arsenic as comparable arsenian pyrite contained significantly less gold. The reason for the disparity is not known but may have to do with structural differences in the crystal lattices of pyrite and marcasite, assuming contemporaneous formation and identical ore fluid chemistry.
Arsenian pyrite commonly contains a wide array of trace elements including Sb, Hg, Ni, Co, Cu, Tl, Ag, Zn, W, U, Pb, Bi, Se, and Te (Cook and Chryssoulis, 1990; Fleet et al., 1989, 1993, 1997; Arehart et al., 1993a; Savage et al., 2000; Cline, 2001; Emsbo et al., 2003). However, the element of interest is gold. Pyrite is generally only capable of containing trace gold values of less than 10 ppm in the absence of arsenic (Cook and Chryssoulis, 1990). Arehart et al. (1993a) performed secondary ion mass spectroscopy (SIMS) analysis on five samples from the high-grade zones of Gold Quarry. They reported gold concentrations in pre-ore pyrite of 2.5-4.8 ppm and 45-62 ppm gold in arsenian pyrite rims with an average of 4.6 percent arsenic. Fleet and Mumin (1997) reported arsenian pyrite from Deep Star with gold values to 0.37 wt percent.

Experimental laboratory synthesized arsenopyrite, arsenian pyrite, and marcasite have realized up to 3.0, 0.64, and 0.35 wt percent gold respectively (Fleet and Mumin, 1997). Wu and Delbove (1989) reported much lower gold values for naturally occurring arsenopyrite of 1.5 and 1.7 wt percent. Fleet and Mumin (1997) also found that elevated gold values for arsenopyrite correlates with higher than normal arsenic concentrations and iron depletion from the idealized formula. Studies by Cook and Chryssoulis (1990), Simon et al. (1999a), and Savage et al (2000) verify that increasing arsenic correlates with decreasing sulfur in pyrite. Experimentally synthesized pyrite contains a maximum of 9.3 wt percent arsenic. Marcasite at 16.5 wt percent and arsenopyrite at 53.5 wt percent are capable of much higher arsenic values (Fleet and Mumin, 1997). Simon et al. (1999a) argue that some or most of the arsenic in arsenian pyrite may be contained within micro layers of marcasite or arsenopyrite 10 to 15 Å wide termed planer or growth stacking faults. Arehart et al. (1993a) reported that the arsenopyrite from the Carlin trend
is generally barren of gold. Fleet and Mumin (1997) also reported that the arsenopyrite was gold poor and displayed higher than optimum sulfur values. Low temperature of formation (<250° C) of arsenopyrite is consistent with elevated sulfur values (Kretschmar and Scott, 1976).

The gold contained within arsenian pyrite is very small, with individual grains generally undetectable. Previous studies utilizing high-resolution (~2 Å) transmission electron microscopy (HRTEM) failed to identify the majority of the gold particles in gold-rich arsenian rims (Yang et al., 1998). This suggests that much of the gold in the arsenian pyrite is in a solid solution.

Arehart et al. (1993a) argued that the gold was incorporated into the pyrite as a coupled substitution where gold oxidized to Au$^{3+}$ replaces iron and arsenic reduced to As$^{1+}$ replaces sulfur, resulting in a metastable auriferous arsenian pyrite. Some workers have argued for the replacement of iron by Au$^{1+}$ in the lattice of arsenopyrite (Wu and Delbove, 1989; Tarnocai et al., 1997; Cabri et al., 2000). Others suggest that the gold is sequestered in lattice defects (Fleet and Mumin, 1997). This process requires the presence of arsenic and a surface defect that was probably caused by the rapid crystal growth favored under relatively low temperatures (< 250° C) and nonequilibrium conditions (Reich et al., 2005).

Carlin-type systems are relatively low temperature with the low-grade periphery being even more so. The ore fluids passing through the low-grade horizons would have been under-saturated with respect to gold. The gold transported as a Au(HS)$^{0}_{(aq)}$ (Seward, 1973)(reaction 1) or HAsS$_2^{2-}(aq)$ (Spycher and Reed, 1989)(reaction 2) complex may have been chemically adsorbed from solution due to sulfidation and the
accompanying reduction in the activity of H₂S (Reich et al., 2005) by one of the following reactions.

\[
\text{Fe(S,As)₂ + 2Au(HS)₀} \rightarrow \text{Fe(S,As)₂ • Au₂S₀ + H₂S(aq) (Reaction 1)}
\]

\[
\text{Fe}^{2⁺} + \text{2HAS₂(aq)} + \text{2Au(HS)₀(aq)} + \text{2H₂(aq)} = \text{Fe(S,As)₂ • Au₂S₀ + 3H₂S(aq) + 2H⁺ (Reaction 2)}
\]

Figure 5. Cross-section of the Gold Quarry pit showing the relative positions of the Quarry Main, Deep West, and Deep Sulfide Feeder ore bodies and grade distribution (From Harlan et al., 2002).

Local nanoparticles of native gold contained within the arsenian pyrite rims are primarily restricted to the high-grade zones and feeders of the deposit. Palenik et al. (2002) and Reich et al. (2003) determined the maximum arsenic and gold values obtainable in arsenian pyrite as approximately 10 and 0.5 wt percent, respectively. Palenik et al. (2004) argues that if the solubility limit of the arsenian pyrite rim is exceeded locally, then any further gold precipitation in that area will occur as native gold. Native gold nanoparticles may also form by exsolution from the metastable arsenian...
pyrite-gold solid solution (Arehart et al., 1993a; Palenik et al., 2002; Reich et al., 2003). Palenik et al. (2004) speculate that the solubility limit may vary with temperature and that the lower temperatures experienced following mineralization and system cooling would result in the exsolution of nanoparticles of gold in areas that were previously at or slightly below the solubility limit and now exceed it. Elsewhere, the gold remains in solid solution.

There has been relatively little published work specifically on arsenic-gold bearing sulfides from Gold Quarry. Arehart et al. (1993a) studied five samples from Gold Quarry and another 20 samples from other Carlin trend deposits. Several other workers (Wells and Mullens, 1973; Bakken et al., 1991; Ramadorai et al., 1991; Fleet and Mumin, 1997; Palenik et al., 2002; Palenik et al., 2004) have concentrated on deposits located in the northern Carlin trend. The vast majority of previous work has also concentrated primarily on the high-grade zones and feeders rather than the peripheral low-grade ore. The low-grade periphery of the deposit may in fact provide a more complex mosaic of local variations in mineralogy and paragenesis due to locally weaker and less frequent overprinting.

**Alteration**

There are eight types of alteration recorded in the Gold Quarry deposit. Of these, decalcification, sericitization, silicification, and argillization are of the greatest relevance to gold mineralization. Late stage sulfidation was most important in the formation of the deposit as the gold is generally contained in late stage sulfidic overgrowths or rims. Early sulfide formation primarily through diagenesis had little bearing on gold deposition and is
generally barren. Dolomitization, alunitization, and supergene oxidation round out the alteration suite at the Gold Quarry deposit (Harlan et al., 2002).

Decalcification is structurally controlled and extensive throughout the Maggie Creek District and was the precursor to the other alteration types (Harlan et al., 2002). Decalcification promotes enhanced permeability and fault propagation through carbonate dissolution (Rota and Hausen, 1991). Sha (1993) estimated carbonate loss in the Maggie Creek District at approximately 40%. Decalcification led to the formation of significant collapse breccias in the upper Popovich Formation and lower Rodeo Creek unit and may have provided the carbonate for the massive calcite veins in the Gold Quarry fault system footwall (Williams, 1992).

Replacement hydrothermal dolomite with minor associated veining forms the basal zone of the decalcified zone. The dolomite zone ranges in thickness from 6 m to as much as 90 m. Narrow 2.5 cm milky dolomite veins and thin orpiment veinlets are more prevalent in the upper reaches of the zone. Gold mineralization to 3.4 g/t is associated with the alteration front and decreases with depth (Harlan et al., 2002).

Sericite-illite alteration is associated with decalcification and gold mineralization. Sericite replaces carbonate in areas of decalcification and is locally abundant in the southern and eastern areas of the Gold Quarry pit. Sericite-illite displays an association with silicification along the margins of dikes and veins (Harlan et al., 2002).

Silicic alteration over multiple generations led to the formation of large silicified replacement zones and breccias, small quartz veins, and fine quartz stockwork. The stockwork quartz was associated with gold mineralization within the Quarry Main deposit. The deeper deposits contain multistage collapse breccias where both the breccia
fragments and the matrix are silicified. These breccias, when displaying fluidized textures, are generally mineralized (Harlan et al., 2002).

Faults at Gold Quarry generally control argillization with the primary clays being kaolinite and montmorillonite. Main stage kaolinite contains up to 10% sulfide, is dark gray in color and is generally found in fluid conduits transecting silicified rock. Where the clay contains sooty black sulfide, the gold grades are generally high. White to light green kaolinite is late stage and unmineralized (Harlan et al., 2002).

Early stage sulfidation occurred prior to the primary gold mineralization event. Late stage sulfidation resulted in very fine generally submicron auriferous disseminated sulfide deposition along with auriferous arsenian rims forming on barren early stage diagenetic or hydrothermally recrystalized pyrite (Harlan et al., 2002). Sulfidation occurred when the ore fluid, enriched in H₂S, interacted with the iron in the wall rock, resulting in the precipitation of auriferous arsenian pyrite (Teal and Jackson, 1997).

Barite alteration resulted in the late deposition of barite crystals in fractures and voids throughout the Gold Quarry deposit. Crystalline alunite occurs in veins and breccias in the deeper parts of the deposit and is pink to pale purple. It is found in association with quartz, barite, and the late stage white to light green kaolinite. A gray to white, earthy and fibrous alunite is common in veins in the oxidized upper zone of the deposit (Heitt, 1992).

Oxidation of the upper zone of the Gold Quarry Main deposit allowed for the development of the mine using the less expensive cyanide heap leach methods. While oxidation localized along fracture and fault zones extends to depths in excess of 450 m,
the majority of the higher-grade ore at depth is refractory and requires more expensive processing methods.

**Metallurgy**

**Biooxidation Facility Design**

The following tables outline the differences between the original biooxidation facility design and the actual initial commercial facility design. Operating parameters were constantly modified until the program was terminated. Consequently, the operating parameters illustrated in these tables do not represent the conditions present at the time this study was conducted.

Table I illustrates heap design parameter changes that negatively affecting overall biooxidation efficiency. Fewer pads results in shorter reaction periods. Larger crush size results in lower reactive surface area. Truck loading and increased heap height lead to increased compressional loading and compaction. Final recovery through milling rather than heap leach increases overall cost.

Table II illustrates changes made to ore control and pad maintenance from original parameters. These changes again generally were represented by lower actual gold recovery. The combination of the changes from both tables I and II, resulted in gold recoveries that were 10% to 22% below original design expectations.
Table I. Comparison of original design parameters versus actual initial commercial operational parameters (modified from Tempel, 1999)

<table>
<thead>
<tr>
<th>Design Component</th>
<th>Original Design</th>
<th>As-Built Facility</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of pads</td>
<td>12</td>
<td>3</td>
</tr>
<tr>
<td>Crushing</td>
<td>Tertiary, P80 = 10 mm</td>
<td>Secondary, P80 = 19 mm</td>
</tr>
<tr>
<td>Inoculation</td>
<td>Drum</td>
<td>Belt Transfer</td>
</tr>
<tr>
<td>Transport</td>
<td>Overland Conveyer</td>
<td>Truck</td>
</tr>
<tr>
<td>Stacking</td>
<td>Radial Stacker</td>
<td>Haul Truck</td>
</tr>
<tr>
<td>Unloading</td>
<td>Bucket Wheel Excavator</td>
<td>Shovel &amp; Haul Truck</td>
</tr>
<tr>
<td>Biosolution</td>
<td>Lime Precipitation</td>
<td>None</td>
</tr>
<tr>
<td>Management</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Au Recovery</td>
<td>Heap Leach</td>
<td>Milling &amp; CIL</td>
</tr>
<tr>
<td>Heap Height</td>
<td>10.7 m</td>
<td>12.8 m</td>
</tr>
</tbody>
</table>

Table II. Comparison of original operational criteria versus initial commercial operational criteria (modified from Tempel, 1999)

<table>
<thead>
<tr>
<th>Operating Criteria</th>
<th>Original Design</th>
<th>Actual Operation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Biooxidation time (d)</td>
<td>270</td>
<td>150</td>
</tr>
<tr>
<td>Sulfide sulfur (%)</td>
<td>1.67 – 2.10</td>
<td>1.40 – 1.80</td>
</tr>
<tr>
<td>Max carbonate content (%)</td>
<td>2.50</td>
<td>1.25</td>
</tr>
<tr>
<td>Heap temperature (ºC)</td>
<td>38 – 60</td>
<td>11 – 81</td>
</tr>
<tr>
<td>Biosolution pond Eh (mv)</td>
<td>&gt; +550</td>
<td>385 – 745, avg. 519</td>
</tr>
<tr>
<td>Biosolution dissolved O2 (ppm)</td>
<td>4 – 6</td>
<td>0 – 7</td>
</tr>
<tr>
<td>Biosolution pond Fe (gpl)</td>
<td>13</td>
<td>8 – 36, avg. 18</td>
</tr>
<tr>
<td>Replace air piping</td>
<td>1 cycle</td>
<td>3 cycles</td>
</tr>
<tr>
<td>Au recovery (%)</td>
<td>71</td>
<td>49 - 61</td>
</tr>
</tbody>
</table>
Table III illustrates the importance of ore control (segregation) as well as the stacking method. SSR conveyer stacked ore achieved a substantially higher percent oxidation over the truck stacked ore of the same type in a much shorted period of time. CSR ore was also conveyer stacked and oxidized for a similar period of time as the SSR ore, but achieved a much lower percent oxidation.

Table III. Comparison of biooxidation efficiency in the demonstration heap related to ore type and delivery method (from Shutey-McCann et al., 1997)

<table>
<thead>
<tr>
<th>Demonstration Heap</th>
<th>SSR ore Trucked</th>
<th>SSR Ore Conveyer</th>
<th>CSR Ore Conveyer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sulfide Sulfur (%)</td>
<td>3.83</td>
<td>3.29</td>
<td>1.57</td>
</tr>
<tr>
<td>Biooxidation Time (d)</td>
<td>240</td>
<td>180</td>
<td>160</td>
</tr>
<tr>
<td>Total Oxidation (%)</td>
<td>30</td>
<td>40</td>
<td>29</td>
</tr>
</tbody>
</table>

Ore Classifications

Oxide Ore

Gold ore from Gold Quarry is classified as either oxide or refractory. Harlan et al. (2002) separate oxidized from refractory ore by the percentage of total gold, as defined by fire assay, recoverable through conventional cyanide leach. Ore with greater than 40% gold recovery is considered oxide. Oxidation usually occurs in the shallow near-surface environment due to the downward percolation of oxidizing supergene meteoric fluids above the water table (Robb, 2005). Oxidation may also occur to greater depths localized along fault or fracture zones and at Gold Quarry reaches 450 m below the surface (Harlan et al., 2002). Romberger (1991) argues that hydrothermal oxidation
is responsible for the deposition of hematite, which is not stable below 55° C, along feeders and at shallow locations within the deposit.

The oxidation process liberates the gold that is encapsulated within sulfides and deactivates any preg-robbing properties of organic carbon that may be present, thereby facilitating gold recovery by cyanide heap-leach extraction methods. Silica encapsulation is not affected by oxidation and is generally the primary limiting factor in gold recovery from oxide ores by cyanidization. Within the Gold Quarry Mine, the majority of the oxide ore was contained in the relatively shallow Quarry Main deposit at 120 to 210 m below the surface and that material has been exhausted (Harlan et al., 2002).

Refractory Ore types

Refractory ores at Gold Quarry are below the oxide zone and comprise two general suites: carbon ± sulfide (CSR), and silica ± sulfide (SSR) (Harlan et al., 2002). The carbon is refractory when it is capable of preg-robbing and both silica and sulfide crystals may encapsulate gold particles during their formation and act to shield the gold from the cyanide solution.

The origin of the preg-robbing carbon within the Carlin trend is probably due to the presence and devolatization of petroleum into pyrobitumen (Hausen and Park, 1986). Preg-robbing occurs when the carbon cation possesses a sufficiently strong positive charge (activated carbon) that it adsorbs to the negatively charged gold-cyanide complex anion resulting in reduced recoveries (Ramadorai et al., 1991). Preg-robbing ore at Gold Quarry is currently stockpiled until cost effective metallurgical processes are derived to deactivate the carbon.
Sulfidic refractory ore is composed primarily of pyrite that has an arsenian rim or disseminated very fine-grain arsenian pyrite. Lesser amounts of arsenopyrite, arsenian marcasite, and other arsenic-bearing phases are also present and are only minor contributors to gold production (Arehart et al., 1993a). Pyrite and marcasite gold concentrations are directly related to crystal size, shape and arsenic content with the general parameters of less than 30-micron diameter framboidal or anhedral grains that are porous and contain 1 to 6 wt. % arsenic (Sha, 1993). Gold values are generally restricted to later stage arsenian rims that are up to 25 microns thick on otherwise gold-barren pre-ore pyrite. Euhedral pyrite crystals and arsenopyrite without arsenian pyrite rims contain very little or no gold (Arehart et al., 1993a). The majority of the gold occurs as colloidal to approximately 2 Å particles in solid solution in the structural lattice of the sulfide crystals (Arehart et al., 1993a; Sha, 1993). Rare gold inclusions up to 200 Å occur primarily in the high-grade feeders, probably due to exsolution (Arehart et al., 1993a). Due to the size and nature of the gold inclusions, fine grinding is not sufficient to enable cyanide recovery (Ramadorai et al., 1991).

Silicic refractory ore is generated when the gold is encased in quartz, chalcedony, chert, or opaline silica (Ramadorai et al., 1991). This may occur when fine gold is deposited prior to or contemporaneously with silicic alteration. Biooxidation has no effect on silicic encapsulation. Only fine grinding may expose the gold to cyanidization. Silicification is represented in Gold Quarry by multiple episodes of veins, breccias, and pervasive silicification. Early stage silicification is associated with low-grade gold mineralization in the deep sulfide feeder deposit and is followed by high-grade gold deposition during intense argillization and sulfidation (Harlan et al., 2002).
Geochemistry

Gold deposition generally correlates with arsenic, antimony, and mercury at Gold Quarry (Harlan et al., 2002). While arsenic is associated with arsenopyrite and arsenian pyrite and antimony is associated with stibnite, no mercury containing minerals were identified by Rota and Hausen (1991). Tetrahedrite is the primary antimony mineral recognized to date, since stibnite was not identified in any of the sections analyzed for this study. Other arsenic-bearing minerals present include gersdorffite and tennantite. Tiemannite is the only mercury mineral identified during this study. However, ongoing research beyond the scope of the current project has identified several other mercury bearing minerals. Silver is anomalous in conjunction with gold throughout the deposit and is substantially enriched in rare argentiferous galena veins and pods. Larger galena pods or veins were shallow, with silver values as high as 107 oz/ton, and have been mined out. Rare small occurrences of galena were also found in the Deep Sulfide Feeder zone (Harlan et al., 2002). Ongoing Ph.D. research has identified multiple silver-bearing minerals. No appreciable silver association with galena has been found; however, other lead minerals that locally are spatially associated with galena in veins such as lead-arsenic-antimony and lead-antimony sulfosalts are argentiferous (silver containing). Minor base metals at Gold Quarry include lead, zinc, copper, and nickel and are generally associated with the northwest striking Good Hope fault and related compressional structures. Continuing dissertation research has identified multiple base-metal rich veins at depth within the Deep Sulfide Feeder zone spatially removed from the Good Hope fault.
The major trace elements found at Gold Quarry are cobalt, molybdenum, strontium, tungsten, vanadium, selenium, and cadmium (Rota and Hausen, 1991; and Harlan et al., 2002). These elements are erratically distributed in low concentrations in hydrothermal feeder faults and associated silicic alteration zones.

**Sulfide Oxidation**

Oxidation of sulfidic refractory ores is critical to gold recoveries since the gold is encapsulated in the crystal lattice of the sulfide mineral and is therefore protected from standard cyanide leach or fine milling. The rate at which the oxidation process proceeds is also of great importance to the economic viability of the mining operation. The high temperatures associated with roasting are capable of oxidizing the sulfides in a matter of minutes, however the process is very expensive and only high-grade ores are economical. The oxidation of low-grade refractory ores must therefore proceed using the slower and less expensive biooxidation processes. Alternatively, sulfide concentration through flotation prior to roasting also allows for the processing of certain low-grade sulfidic refractory ores.

Sulfide oxidation generally occurs relatively slowly under natural conditions because the encompassing host rock greatly impedes the infusion of oxidizing solutions. The two primary oxidizing agents are dissolved O$_2$ and ferric iron (Fe$^{3+}$) present in solution. Iron-oxidizing bacteria catalyze the reaction by rapidly oxidizing ferrous iron (Fe$^{2+}$) to ferric iron (Fe$^{3+}$) which is then released into solution.

Nordstrom (1982) described the general reaction for the abiotic oxidation of pyrite by O$_2$ as:

$$\text{FeS}_2 + \frac{7}{2} \text{O}_2 + \text{H}_2\text{O} \rightarrow \text{Fe}^{2+} + 2\text{SO}_4^{2-} + 2\text{H}^+$$  \hspace{1cm} (1)
Singer and Stumm (1970) argue that Fe$^{3+}$ is the dominant pyrite-oxidizing agent under low pH conditions. The presence of O$_2$ is not required for pyrite oxidation by Fe$^{3+}$, as shown in reaction (2) by Bonnissel-Gissinger et al. (1998). The combination of reactions (2) and (3) create a self-propagating reaction series as depicted below. The Fe$^{3+}$ in solution oxidizes the surface of the pyrite and releases Fe$^{2+}$ into solution. The Fe$^{2+}$ in solution is then oxidized by O$_2$ to Fe$^{3+}$, which in turn further oxidizes the pyrite, releasing more Fe$^{2+}$ (Frau, 2000):

\[
\begin{align*}
\text{FeS}_2 + 14\text{Fe}^{3+} + 8\text{H}_2\text{O} &\rightarrow 15\text{Fe}^{2+} + 2\text{SO}_4^{2-} + 16\text{H}^+ \\
14\text{Fe}^{2+} + \frac{7}{2}\text{O}_2 + 14\text{H}^+ &\rightarrow 14\text{Fe}^{3+} + 7\text{H}_2\text{O}
\end{align*}
\]  

(2)

(3)

Many species of acidophilic bacteria and archaea are able to catalyze (lower the activation energy of) these oxidation reactions, accelerating the rates of reaction by up to six orders of magnitude (Singer and Stumm, 1970). These chemoautotrophic microbes are iron and/or sulfur oxidizers, negating the need for O$_2$ in reaction (3). However, dissolved O$_2$ is usually required to maintain the metabolic functions of most of these microorganisms, the exceptions being anaerobic and facultative aerobic strains. They oxidize the Fe$^{2+}$ to Fe$^{3+}$, thereby gaining the energy they require for growth via electron transfer during the oxidation process. The microbes typically attach themselves to the surface of the aggregate by secreting a polymer matrix forming a biofilm, or simply remain suspended in solution. The greatly increased reaction rates also result in an increase in temperature since the reactions are exothermic, producing approximately 1,500 kJ of heat for every 120 g (1 mol) of pyrite consumed (Nordstrom and Alpers,
1999). The temperature of the oxidizing zone may locally approach 90° C as measured by Newmont probes in heap 8 2006.

The following balanced reactions describe the oxidization of arsenopyrite and base metal sulfides present in the Gold Quarry deposit:

arsenopyrite \[ \text{FeAsS} + 13\text{Fe}^{3+} + 8\text{H}_2\text{O} \rightarrow 13\text{H}^+ + \text{H}_3\text{AsO}_4 + \text{SO}_4^{2-} + 14\text{Fe}^{2+} \] (4)
galena \[ \text{PbS} + 8\text{Fe}^{3+} + 4\text{H}_2\text{O} \rightarrow 8\text{H}^+ + \text{Pb}^{2+} + \text{SO}_4^{2-} + 8\text{Fe}^{2+} \] (5)
sphalerite \[ \text{ZnS} + 8\text{Fe}^{3+} + 4\text{H}_2\text{O} \rightarrow 8\text{H}^+ + \text{Zn}^{2+} + \text{SO}_4^{2-} + 8\text{Fe}^{2+} \] (6)
chalcopyrite \[ \text{CuFeS}_2 + 16\text{Fe}^{3+} + 8\text{H}_2\text{O} \rightarrow 16\text{H}^+ + \text{Cu}^{2+} + 2\text{SO}_4^{2-} + 17\text{Fe}^{2+} \] (7)

**Secondary Sulfate Minerals**

The oxidation of sulfides using artificially accelerated natural processes in a heap type environment leads to the precipitation of varying amounts of numerous secondary sulfate minerals. These secondary sulfate minerals literally act as crystalline reservoirs for metals and acidity (Chou et al., 2002). The formation and/or rapid dissolution of these sulfate minerals has many localized effects upon the chemistry, hydrology, aeration, and the microbiology of the heap pad.

Dehydration of the biooxidation fluid following sulfide oxidation may locally result in supersaturation and the commensurate precipitation of various secondary hydrous sulfate minerals (Appendix I). The dehydration process is primarily due to convection driven by the heat generated from highly exothermic sulfide oxidation reactions. The sulfates that form on the surface of the heap are generally efflorescent (powdery), or extremely fine-grain, and form around natural surface vents. These sulfates rapidly dehydrate under surface conditions and are highly soluble in aqueous solution. Due to their extremely hydrous nature, certain sulfate minerals, such as melanterite, only
form in well-protected areas where humidity approaches 100 percent or where very low solution flow rates or ponding conditions occur. Solution metal cation and the complex sulfate anion (SO\(_4^{2-}\)) concentrations become supersaturated due to continued sulfide oxidation and water loss from the convective process. These sulfates tend to form much larger crystals that in laboratory experiments have reached up to 3 cm across. These larger crystals are much more resistant to dissolution in an aqueous solution.

Exposure of the hydrous sulfate minerals to a desiccating environment results in alteration through dehydration to another less hydrous sulfate mineral. For instance, pure melanterite, FeSO\(_4\)•7H\(_2\)O, is only stable at very high humidity and dehydrates at 96% humidity (Apelbat, 1993) to copiapite, Fe\(^{2+}\)Fe\(^{3+}\)(SO\(_4\))\(_6\)(OH)\(_2\)•20H\(_2\)O, by reaction (8) or rozenite, FeSO\(_4\)•4H\(_2\)O, by reaction (9) (Frau, 2000). Return to a more humid environment reverses the reaction. Melanterite is a clear blue crystal when trace copper is present, or a light green crystal when no trace element impurities are present. Melanterite becomes white and efflorescent when dehydrated to rozenite.

\[
5\text{FeSO}_4\cdot7\text{H}_2\text{O} + \text{O}_2 + \text{H}_2\text{SO}_4 \rightleftharpoons \text{Fe}^{2+}\text{Fe}^{3+}\text{(SO}_4\text{)}_6\text{(OH)}_2\cdot20\text{H}_2\text{O} + 15\text{H}_2\text{O} \quad (8)
\]

\[
\text{FeSO}_4\cdot7\text{H}_2\text{O} \rightleftharpoons \text{FeSO}_4\cdot4\text{H}_2\text{O} + 3\text{H}_2\text{O} \quad (9)
\]

In areas where the pH is raised due to a change in fluid rock interactions such as occurs during transition from a silicified rock-rich zone to a carbonate-rich zone, or when solution mixing occurs involving a higher pH fluid, the sulfate mineral that is precipitated may vary. When the saturated oxidizing solution migrates down through the heap-leach pad from an area containing predominately oxidizing sulfide-rich ore, melanterite is
generally the first secondary sulfate mineral formed (Buckby et al., 2003). Melanterite may be found on the surfaces of the oxidizing sulfide or aggregate, in small voids, or precipitating in relatively stagnant pools. The reaction (10) for the direct alteration of pyrite to melanterite is (Jerz and Rimstidt, 2003):

\[
\text{FeS}_2 + \frac{7}{2}\text{O}_2 + 8\text{H}_2\text{O} \rightarrow \text{FeSO}_4\cdot7\text{H}_2\text{O} + \text{H}_2\text{SO}_4
\]  

(10)

When the same oxidizing solution encounters an area with an acid-neutralizing carbonate mineral such as calcite or dolomite, the mineral formed on the aggregate surface may be a mix of schwertmannite, Fe\(^{3+}\)\(_{16}\)O\(_{16}\)(OH)\(_{12}\)(SO\(_4\))\(_2\), jarosite, KFe\(^{3+}\)(SO\(_4\))\(_2\)(OH)\(_6\), and goethite, FeOOH. Schwertmannite tends to retain arsenic within its structure, distorting the structure and destabilizing the mineral. Schwertmannite preferentially precipitates in the range of pH 2.8 to 3.2, readily altering to goethite by reaction (11) as pH increases or at lower pH with elevated arsenic concentrations within its structure (Bingham et al., 1996):

\[
\text{Fe}_8\text{O}_8(\text{OH})_{5.5}(\text{SO}_4)_{1.25} + 2.5\text{H}_2\text{O} \rightarrow 8\text{FeOOH} + 2.5\text{H}^+ + 1.25\text{SO}_4^{2-}
\]  

(11)

Schwertmannite has not been identified in the heaps at Gold Quarry. This is due to an average biopond pH of 2.5 and generally lower pH values within the heap. Schwertmannite may form in the sulfate goethite cap under certain conditions such as a precipitation event or fresh water wash.

The formation of these secondary sulfate minerals tends to cement the aggregate of the heap into relatively impermeable lenses or umbrellas that then channel the slowly percolating biooxidation solution, locally shielding ore from the solution. This effect has been commonly observed in exposed cross-sections of the heaps during unloading by the presence of white to greenish-white “umbrella” type structures (Figure 6). Initial visual
inspection and the literature cited above suggested that the cemented aggregate lenses of the heaps at Gold Quarry, which display a faint bluish green tint when initially exposed and rapidly alter to a white color upon exposure to the dry Nevada air would turn out to be melanterite and rozenite, respectively. Further analysis identified these sulfates as alunogen (Al₂(SO₄)₃ • 17H₂O) and halotrichite, (FeAl₂(SO₄)₄ • 22H₂O).

Figure 6. Precipitate “umbrellas” are bluish-green/gray to white areas that form “zebra” like patterns throughout the upper portion of the heap. Much of the area beneath the umbrellas is only slightly oxidized.

Formation of secondary sulfate minerals removes from solution various metals and effectively stores acidity as sulfate and water. The metals removed from solution may include toxic metals such as arsenic and cadmium. A large precipitation event can lead to the sudden dissolution of sulfates accumulated over a long period of time, perhaps
months, which may be released in a matter of a few minutes to a few of hours. Laboratory experiments conducted by Frau (2000) show that the addition of melanterite to pure water results in a nearly instantaneous decrease in pH of approximately 3 pH units from a starting point of pH 5.7. Regardless of the amount of melanterite added, total dissolution and constant pH were always achieved in less than 2 minutes. The minimum pH achieved was approximately 3, which is well above the 1.5 to 2.5 normally found within the heap. Therefore, a large precipitation event will have little overall effect on the heap except to force the precipitation of iron oxide, probably as goethite, thereby contributing to the formation of the agglomerated cap. And due to the already saturated state of the biooxidation solution, little if any sulfate dissolution occurs due to pad irrigation.

**Microbial Mineral Oxidation Mechanisms**

The biooxidation of minerals represents a close partnership between chemistry and biology. Current thinking considers biooxidation to be primarily a chemical process in which, depending on mineral type, ferric iron and protons play a major role in the oxidation reactions. Rawlings (2007) provides a good example of this thinking, and why it is incomplete:

“The effect of this is that if a mineral (such as chalcopyrite) is recalcitrant to bio-oxidation at 40° C, the solution to the problem lies primarily in the realm of chemistry rather than biology. For example, an increase in temperature of the biooxidation process to 80° C will allow the chemical reactions to take place at a much faster rate. However, although the solution to a slow reaction rate is based on chemical considerations, the biology needs to follow the chemistry and microorganisms that are
capable of iron and sulfur oxidation at 80° C are required to generate or regenerate the lixiviants (leaching chemicals).”

The bottom line is that mineral oxidation works best when both chemical and biological processes occur together. This is because microbes do more than generate the ferric iron and acid needed for the various oxidation reactions. When microbes attach to a mineral, they do so by producing an exopolysaccharide layer, or biofilm, that facilitates breakdown of the mineral, sulfides, etc. that inhibit recovery of the precious metal of interest (Sand et al., 1995, Gehrke et al., 1998). In essence, the microbes act as catalysts--the microbial exopolymer coating provides a reaction space where mineral dissolution reactions take place more efficiently than they would in the bulk solution. A depiction of this can be seen in Figure 7.

Although biofilms are important, they are not the whole story, since much of the biooxidation that occurs is through the action of unattached microbes. Later work by Sand and coworkers (Schippers and Sand, 1999; Rohwerder et al., 2003) led to the proposal that minerals can be divided into two broad categories with regard to the chemical reactions involved in their biooxidation. The first group is the acid-insoluble minerals that are solubilized via oxidation by ferric iron (e.g. FeS₂, MoS₂, and WS₂). In this case, the chemical bonds between the metal and sulfur break only after a series of six one-electron removal steps that result in thiosulfate as the first free sulfur compound. An example of this is outlined by Rawlings (2007) for pyrite oxidation:

\[
0.25S_8 + 3O_2 + 2H_2O \xrightarrow{\text{Iron-oxidizing acidophiles}} 2Fe^{3+} + H_2O
\]
The second group is the acid soluble minerals that are oxidized by the action of both ferric iron and protons (e.g., CuFeS, FeAsS, PbS, ZnS and MnS₂). Here the chemical bonds between the metal and sulfur are broken by protons resulting in the release of hydrogen sulfide after two protons have bound. Simultaneous oxidation of the sulfur moiety likely occurs if ferric iron is present. After several spontaneous rearrangement and oxidation steps with polysulfides as intermediates, the final product is elemental sulfur as outlined by Schippers and Sand (1999) for sphalerite oxidation:

\[
\begin{align*}
\text{ZnS} + \text{Fe}^{3+} + \text{H}^+ & \rightarrow \text{Zn}^{2+} + 0.5\text{H}_2\text{S}_n + \text{Fe}^{2+} \quad (n \geq 2) \quad (13) \\
\text{H}_2\text{S}_n + 2\text{Fe}^{3+} & \rightarrow 0.25\text{S}_8 + 2\text{Fe}^{2+} + 2\text{H}^+ \quad (14)
\end{align*}
\]
Biooxidation of Refractory Gold-Bearing Ores

Over the last three decades, acidophilic (acid-loving) microorganisms capable of oxidizing metal sulfides have been studied and utilized in various metal recovery processes. These microbes can be found at many locales where mineral oxidation occurs naturally, particularly at the low pH, metal-rich, inorganic environments typically present at many mining operations. The microorganisms isolated from these locations are usually grouped according to their preferred growth temperatures, and fall into three categories: mesophiles (25° to 40° C, no growth >45° C), moderate thermophiles (50° ± 10° C), and extreme thermophiles or hyperthermophiles (>60° C). They are also categorized as either autotrophs or heterotrophs based on the sources of energy and carbon used for their
growth. Autotrophs derive energy from light (photoautotrophs) or inorganic sources (chemoautotrophs, also called chemolithotrophs) and obtain carbon from CO₂ in the atmosphere. In contrast, heterotrophs (chemoorganotrophs) obtain both energy and carbon for growth from organic compounds. The most important microbes involved in the biooxidation of minerals are those responsible for producing the ferric iron and sulfuric acid needed for mineral solubilization and are largely autotrophs. Iron- and sulfur-oxidizing *Acidithiobacillus ferrooxidans* (*At. ferrouxidans*), sulfur-oxidizing *Acidithiobacillus thiooxidans* (*At. thiooxidans*), and iron-oxidizing *Leptospirillum* species (*L. ferrooxidans* and *L. ferrphilum*)---all mesophilic, chemoautotrophic bacteria---represent the strains most commonly isolated from inorganic mining sites. The moderate thermophile *Acidithiobacillus caldus* (*At. caldus*), which uses only reduced sulfur compounds, has also been readily isolated from these processes and likely also plays a major role (Rawlings, 1997). The following paragraphs briefly describe the major processes used to date for biooxidizing refractory gold-bearing ores using these and other microbes. A more in-depth coverage of each technology can be found in the references.

The BIOX™ process, developed by Gencor in the late 1970’s and still in use today at numerous sites, oxidizes gold concentrates in a series of primary and secondary stirred tank bioreactors containing a mixture of bacteria (Dew et al., 1997; van Aswegan et al., 2007). *At. ferrouxidans, At. thiooxidans*, and *L. ferrooxidans* cultures are used to oxidize the gold-containing sulfide mineral matrix, freeing the trapped gold for subsequent recovery by cyanide treatment. Both temperature and pH are known to affect the composition of the microbial population, which is important because *Leptospirillum* bacteria can only oxidize ferrous iron whereas *At. thiooxidans* bacteria can only oxidize
sulfur-containing compounds. Research indicated that low pH and high slurry temperatures enhance *Leptospirillum* growth (Lawson, 1991). Shake flask tests showed that the oxidative activity of *At. ferrooxidans* is inhibited in the pH range 2 to 3, and tests with *At. thiooxidans* showed little growth between pH 0.5 and 1.0 (van Aswegen et al., 2007). Optimal oxidation rates are obtained by controlling process pH and temperature within narrow ranges in order to maintain the right balance of bacterial species in the bioreactors. The typical optimal operating conditions for the BIOX™ process are 40° C and a pH between 1.2 and 1.8. *Leptospirillum* species and *At. caldus* were identified as the most dominant microbes in arsenopyrite BIOX™ tanks operating at 40° C; the dominant *Leptospirillum* strain was found to be *L. ferriphilum* (Coram and Rawlings, 2002).

Newmont was the first mining company to utilize commercial heap bioleaching for refractory gold recovery in the United States. To date, Newmont has successfully biooxidized 12 commercial batches of sulfidic ore using their BIOPRO™ process (Brierley, 1997; Logan et al., 2007). Field pilot testing was initiated in 1990 and during the first 4 years six whole-ore field tests took place using heaps ranging in size from 360 to 23,000 t (Brierley et al., 1995). The results of multiple biooxidation cycles carried out at their 708,000 t heap demonstration facility between 1995 and 1999 indicated that whole-ore heap biooxidation could be inexpensively carried out on a large scale (Shutey-McCann et al., 1997). Commercial scale biooxidation efforts were realized when the commercial heap biooxidation facility was completed and commissioned in December 1999 with ore placement on a series of 3 biooxidation pads. Original operating
criteria for the commercial facility versus the actual “as built” facility, and the challenges faced during early operation are discussed in detail by K. Temple (2003).

Initial biooxidation runs (field tests and demonstration heaps) used crushed ore inoculated with a bacterial consortium consisting of *At. ferrooxidans*, *L. ferrooxidans*, and moderately thermophilic *Sulfobacillus* species (Logan et al., 2007). A rise in heap temperatures (~80°C) suggested that addition of heat-tolerant iron oxidizing microorganisms might improve gold recovery, which was confirmed in subsequent laboratory column bioleach tests. A microbial mixture containing mesophilic, moderately thermophilic, and thermophilic iron-oxidizing species responded well to temperature changes similar to those occurring in the heaps, and resulted in 41% sulfide oxidation (Brierley, 2003). In preparation for the first full-scale commercial biooxidation cycle, the “adapted” biosolution from the demonstration pond was pumped to the current commercial biosolution pond and subsequently used to inoculate the crushed ore; six months after start-up, thermophilic archaea (including *Acidianus* and *Metallosphaera* species) were added to the inoculum. Thus, the following mixture of microbes made up the inoculum at the commercial biooxidation plant at Gold Quarry: mesophilic *Acidithiobacillus ferrooxidans* and *Leptospirillum ferrooxidans*, moderately thermophilic *Sulfobacillus* species, and thermophilic archea *Acidianus* and *Metallosphaera* species (Olson et al., 2003). The first 3 species belong to the phylogenetic domain Bacteria and the last 2 species belong to the domain Archaea, which includes most extreme thermophiles and other extremophiles. The BIOPRO™ biooxidation process at Gold Quarry is accomplished on three ~800,000 short/ton heap pads (Fig. 8). The ore is crushed to a nominal one inch minus and sprayed with biosolution containing the
microbial consortium (listed above) as the haul trucks are loaded at the secondary crusher. Inoculating the crushed ore with biosolution just prior to loading the pad serves to establish an initial bioleach microbial population throughout the heap during construction and prior to initiation of pad irrigation from the biosolution pond (Fig. 9). Heap dimensions are about 60 m by 160 m with an average height of 14 m above 1.5 m of drain rock. Heap loading consists of a haul truck driving up a ramp and dumping over the edge to a pad height of 17 m. The top 1.5 m are then pushed off with a bulldozer and the upper several feet of the heap are ripped in an attempt to minimize loading compaction. The heap is then plumbed with a drip irrigation system across the top.

Figure 8. Newmont biooxidation pads at Gold Quarry. Pads right to left are A, B and C. View to north with cyanide leach pad in the distance. Biooxidation solution pond is to the northeast of pad C. White tank is for the addition of sulfuric acid when necessary. (From Logan et al., 2007)
The biosolution is stored in an aerated, lined pond and is pumped to the drip irrigation system on the heaps at 0.003 - 0.004 gpm/ft$^2$ for periods at a time determined by elevated heap temperature and lower effluent pH and Eh. Biopond solution volume varies from ~4.5 to 11 million gal. When necessary, potassium phosphate (KH$_2$PO$_4$), a microbial nutrient, is added to the biosolution upstream of the irrigation system. Periodic monitoring of the concentrations of 12 elements (Al, As, Ca, Cu, Fe, P, Mg, Mn, Ni, P, Pb, and Zn) in the pad effluent is conducted by ICP-MS (inductively coupled plasma mass spectroscopy) analysis. Effluent solution chemistry is also monitored for changes in pH, and Eh. Air is injected into tubing installed at the base of the pad to ensure adequate dissolved oxygen levels are maintained within the heap to support microbial respiration and sulfide oxidation. The injected air also tends to complement the natural convective
system within the heap, thereby enhancing the negative effects of biosolution supersaturation and sulfate precipitation.

As noted earlier, the biooxidation procedures at Gold Quarry utilize a consortium of microbes that include both bacteria and archaea. These microbes were originally adapted in the lab by growth on an iron-rich media, and they also most likely experienced further adaptation after exposure to the particular constituents found at the mine site. Thus, in the general discussions that follow concerning members of the consortium, one should keep in mind that adapted microbial strains and populations may differ substantially from the original parental cultures. In addition, many of the growth and oxidation studies reported in the literature have been conducted using “type” strains from established microbial culture collections. Microbiologists have reported that, in some cases, these organisms may show varying properties after multiple rounds of preservation/passage over time.

*Acidithiobacillus ferrooxidans* is a rod-shaped bacterium about 0.5 µm wide by 1 to 1.5 µm long that can occur singly or in pairs. *At. ferrooxidans* is capable of using ferrous (Fe²⁺) iron or reduced sulfur compounds as an electron donor. It typically uses oxygen as a terminal electron acceptor, although it can grow in the absence of oxygen by using ferric iron (Fe³⁺) as an electron acceptor if reduced sulfur compounds are present to serve as an electron donor (Rawlings, 1997). The ability to grow in an anaerobic environment may be important for heap biooxidation since regions of little or no oxygen almost certainly exist. It suggests that the metal mobilizing activity carried out by this organism can still occur if ferric iron is made available to it by the activities of bacteria in oxygenated regions of the heap. Under these conditions, acid production may continue
but ferric iron removal would reduce the concentration of this important leaching reagent (Rawlings, 2007). Optimal conditions for growing *At. ferroxidans* vary a bit from one source to another. Rawlings (1997) states that the species grows best within the pH range of 1.8 to 2.5 and at temperatures between 30° and 35°C, whereas Norris and Johnson (1998) claim a growth pH range between 1.3 to 4.5 (optimum pH of 2.5) and a temperature range from <10° to 37°C with optimum growth between 30° to 35°C. Based on rates of ferrous iron oxidation, Gomez et al. (1999) reported that an *At. ferrooxidans* type strain (ATCC 23270) has a pH optimum range between 2.0 to 2.5 and a temperature optimum of about 30° C, with limited activity at pH 1.25 and at slightly elevated temperatures (≥ 37°C).

*Leptospirillum ferrooxidans* is a ferrous iron-oxidizing bacterium that occurs as a vibroid cell when young and as a highly motile spiral form when mature due to a long polar flagellum. Exopolymer production and flocculation often accompany growth, both of which probably facilitate its biooxidation capabilities. Norris and Johnson (1998) report a growth optimum between pH 1.5 to 2.0 and at temperatures between 30° to 37°C, though the growth range reported for this organism is between <10° to 45°C. Some strains can grow on pyrite at 45°C (Norris, 1983). The oxidation potential of *L. ferrooxidans* has been reported by Gomez et al. (1999) to be generally optimized at >35° C, but certain strains, such as CF12, display a preference for lower temperatures of ~25° C. Since CF 12 was isolated from an Idaho cobalt mine, it is most likely not present at Gold Quarry. However, this demonstrates the variability of specific strains isolated from the environment from the type strains preserved in microbial collections. Gomez et al. (1999) report that repeated subculturing of both *At. ferrooxidans* strain 23270 and *L. ferro-
oxidans strain CF 12 lowered the minimum pH tolerance to ~ pH 1.25. While oxidation was slower at the lower pH for both strains, oxidation virtually ceased at pH 1.0.

Representatives of the species Sulfobacillus are typically isolated from geothermal environments, mineral sulfide-containing ores, coal dumps, and similar sites. One of the best studied strains, Sulfobacillus thermosulfidooxidans, is non-motile, spore-forming and displays cell shape variability depending on growth conditions (Norris, 1990). It is a facultatively moderate thermophile with a wide temperature growth range from 20° to 60° C (Rossi, G., 1990). The optimum pH range for the strain is between 1.9 to 2.4, with a viable growth range between 1.9 and 3.0. Unlike the other bacterial species found in the consortium, both of which are iron oxidizers, S. thermosulfidooxidans is capable of oxidizing elemental sulfur and sulfides other than pyrite, including arsenopyrite, chalcopyrite, sphalerite, galena, covellite, and antimonite (Norris and Johnson, 1998?). S. thermosulfidooxidans and related bacteria are not as competent as *At. ferrooxidans* at using CO\textsubscript{2}; Clark and Norris (1996) reported that they require 1% v/v CO\textsubscript{2}-enriched air for rapid autotrophic growth.

Hyperthermophilic archaeca (*Acidianus* and *Metallosphaera* species) were added to the Gold Quarry inoculum as a hedge against the rise in temperature found to occur in the biooxidation heaps there. These groups of archaeca belong to the order Sulfolobales and their strains have a coccoid morphology. They are thermophilic acidophiles with pH tolerances to pH 1.0, lower than the mesophilic and moderately thermophilic bacteria in the inoculum. The genus *Acidianus* consists of 3 species with a combined optimum growth temperature of 88° C, a growth range from 60° to 90° C, and a pH optimum of 2.0 (Madigan et al., 2003). Under aerobic conditions, *Acidianus* uses elemental sulfur (S\textsubscript{0}) as
an electron donor, oxidizing $S^0$ to sulfuric acid ($H_2SO_4$). Anaerobically, it uses $S^0$ as an electron acceptor and hydrogen ($H_2$) as electron donor, forming the reduced product hydrogen sulfide ($H_2S$). Thus, the presence or absence of $O_2$ determines the metabolic fate of $S^0$ in cultures of *Acidianus*. *Acidianus brierley* is a facultative aerobe, iron oxidizing species originally isolated by James Brierley from an acidic hot spring at Yellowstone National Park. It was originally classified in the genus *Sulfolobus*, but was later reclassified as *Acidianus* when its ability to grow anaerobically was recognized. It has an optimum growth temperature of 70° C and a growth range between 45° and 75° C; its pH optimum range is between pH 1.5 and 2.0, with a growth range from pH 1.0 to 6.0 (Norris and Johnson, 1998). *A. brierley* has been shown to oxidize pyrite, chalcopyrite, and sphalerite at an accelerated rate compared to *At. ferrooxidans* (Konishi et al, 1999). The genus *Metallosphaera* contains 2 species, both of which are aerobic and oxidize sulfur and multiple sulfides (Norris and Johnson, 1998). The combined growth temperature optimum is 75° C, with growth ranging between 50° and 80° C; the combined optimum pH for growth is 2.0 (Madigan et al., 2003). The best known strain, *Metallosphaera sedula*, has a temperature optimum of 75° C, a growth range between 50° and 80°C, and a viable pH range between 1.0 and 4.5 (Norris and Johnson, 1998). Norris and Parrott (1986) found that archaea that oxidize ferrous iron as well as sulfur, such as species of *Acidianus* and *Metallosphaera*, can catalyze more rapid and efficient extraction of metals from mineral sulfides than the bacteria that were being used in industrial bioreactors for mineral processing.
Procedures

In-situ Columns

Four in-situ columns were installed in biooxidation pad 8-5C at Gold Quarry on November 8, 2005. Each column consisted of a 3 m long by 30.5 cm diameter thick-wall PVC sewer pipe that was capped at the bottom. Both the pipe and the cap were perforated with approximately 160 7/8 inch diameter holes to facilitate maximum air and fluid transfer between the column and the heap without compromising pipe structural integrity. Each column was installed in an individual hole by track hoe approximately 3 m inboard of the loading ramp in a line perpendicular to the ramp. The columns were loaded by hand with composite ore removed from the installation hole. A drip point on the biosolution drip lines located across the top of the heap was positioned directly over each column to ensure proper fluid flow within the columns. The spacing between the columns was maintained at approximately 4 m in order to prevent disturbance of the neighboring column during excavation and removal. The columns were buried with the top approximately 0.5 m below the surface. The final column to be removed was equipped with four evenly-spaced temperature probes. The probes were connected to a battery-powered data logger with readings taken every 5 min. Measurements were averaged and recorded every hour with minimum and maximum values recorded every 24 hr. Data were downloaded and batteries changed when a column was removed.

The columns were removed for sampling in a progressive order at 41, 80, 118, and 157 days after installation (Fig. 10). A track hoe was used to excavate around the column, which was then removed with a cargo strap. A battery-powered circular saw was used to cut an approximately 30 cm wide strip from the full length of the column.
The exposed ore was photographed and samples were then taken at four equally spaced intervals and stored in plastic sample bags for transport to the University of Nevada, Reno for mineralogical analysis. The samples were then dried at approximately 40º C for 5 days in order to arrest the oxidation process. Samples were also recovered using sterile procedures and preserved by chemical means, or freezing, for possible microbial studies in the future.

**Laboratory Columns**

Eight columns were erected and instrumented in the pilot plant of the Applied Research Facility at the University of Nevada, Reno (Fig. 11). The columns were
constructed using the same pipe type and dimensions as the in-situ columns. Each
column had preinstalled equidistant sample ports to facilitate uncontaminated sample
recovery during column tear down. The ore was inoculated with biooxidation solution
from the Gold Quarry biopond using a 2 ft³ plastic barrel concrete mixer that added
approximately 500 ml to each load of ore. The ore was then lowered into the columns
using small buckets to minimize compaction and grading. Biooxidation solution for each
individual column was stored in a dedicated aerated plastic reservoir directly below that
column. The solution was pumped from the reservoir to the top of the column by a
peristaltic pump and dripped onto the ore. Air was injected into the base of the column to
ensure sufficient oxygen within the ore pile. The solution drained from the bottom of the
column back to the solution reservoir. Each column was equipped with a temperature
probe located mid column. Measurements from all temperature probes were recorded on
a data logger with the same parameters as for the in-situ columns. Eh and pH readings
were taken manually at the column drains and the reservoirs using handheld Extech
PH110 and RE300 digital self-calibrating meters.

Four of the columns (L1, 2, 7, and 8) were operated at ambient temperature. The
four remaining columns (L3-6) were individually fitted with thermostatically controlled
heating cables; L4 and 5 were operated at intermediate temperature, and L3 and 6 at high
temperature. The cable was spiral wrapped around each column and controlled from a
remote mounted controller. The columns were then wrapped with R19 fiberglass
insulation blankets to minimize heat loss and minimize temperature fluctuations.

The columns were started on December 20, 2005. Initial flow rate in each of the
columns was 5 ml/min. The heaters on the four heated columns were turned on January
6, 2006 and column temperature was lowered and stabilized at 38º C by the next day. Temperature was maintained at the lower setting until system hardware issues were resolved. Excessive wear and multiple tubing failures forced the replacement of new pump heads and larger capacity tubing on January 12, 2006 allowing for increased solution flow rates at much lower pump speeds. The temperature of the two intermediate temperature columns (L4 and 5) was increased incrementally to 50º C over two days starting January 13, and the two high temperature columns (L3 and 6) were increased to 75º C over a three-day period.

These specific temperatures were selected to study the oxidation potential of moderately thermophilic bacteria (50º C) and extremely thermophilic archaea (75º C) and the effects on specific lithologies and ore mineralogical assemblages. These temperatures were also intended to mimic the intermediate and high core temperatures typically occurring in the biooxidation heap. Microbe survivability under fluctuating temperature, low oxygen to anoxic environments, and extremely low pH conditions were also of great interest. As will be demonstrated later, the actual heap core temperature averaged approximately ten degrees higher than expected. The flow rate for the four heated columns was increased on January 13, 2006 to 15 ml/min to counteract increased evaporation at the higher temperatures and maintain nominal solution through flow.

Reservoir solution levels were maintained initially (until January 28) by adding biosolution from the reserve tank to replenish losses due to evaporation and leaks. All later replenishments used distilled water because pump tubing was clogging repeatedly due to sulfate precipitating from solution. Leaks were generally caused by sulfates clogging the drain holes and rarely by tubing failure.
The columns were run for 150 days. Two of the heated columns, one intermediate temperature (L5) and one high temperature (L6), were cycled to near ambient temperature on two separate occasions (between February 3 and March 8), with incremental transitions over ten and eight days, respectively. These temperature alterations were designed to imitate the pump cycling practiced by Newmont to control heap temperature and solution effluent chemistry. When the heap solution effluent turned green or anoxic as measured by a sharp decrease in both pH and Eh, the solution pumps from the biopond were turned on to lower the heap temperature and increase solution Fe$^{3+}$ concentrations to maintain oxidation rates. All columns were shut down completely, pumps and heaters off, on March 15 and restarted incrementally on April 3. Columns L5 and 6 were again cycled down to low temperature beginning on April 19 and back up on April 30. Final column shut down occurred on May 9. The columns were allowed to drain and cool for 11 to 12 days, simulating pad unloading.

All laboratory columns were dismantled on May 20 and 21. Aggregate samples were recovered from 3 of the ambient temperature columns (L1, 7, and 8) from three equidistant zones (top, middle, and bottom) through sample ports in the side of each column after the column was repositioned horizontally. The heated columns (L3 thru L6) and ambient temperature L2 were sampled by removing a section of the column lengthways with a circular saw. This was necessary due to aggregate cementation by secondary sulfate formation. Aggregate removal from the lower portions of these columns required a pick and sledge hammer. Solution samples for ICP and microbial analysis, and solid sulfate samples for ICP, XRD, and SEM analysis, were collected at specific junctions preceding and following induced temperature changes and when
noticeable changes in solution appearance occurred. Generally, samples were collected
from all columns at the same time for comparison purposes. Sulfate samples were
collected from the crusts that formed on the top of the heated columns, from the sulfate
cemented aggregate at the base of the columns, and from the sulfate channel wall in L2.
Sulfate samples were also collected from the walls of all the reservoirs above the solution
level and from the bottom of the reservoir for L3.

**Analytical**

Thirty milliliters of biosolution was periodically collected from the effluent
discharge of each column, specifically bracketing large changes in temperature or
solution appearance. These samples were then shipped to Newmont analytical labs in
Englewood, Colorado, for viable microbial counts. Early microbial count procedures
conducted by Newmont are described by Brierley (1997):

“The population of acidophilic iron-oxidizing bacteria present on ore from an
experimental biooxidation heap was estimated using a most-probable-number (end point
dilution) procedure with -10 mesh fractions of ore samples (Brierley et al., 1995). The
initial population density, following inoculation, was determined to be about 5.3 X 10^5
bacteria/g ore. The bacterial population increased to about 3.5 X 10^7/g ore by day 30 of
biooxidation pretreatment. Bacterial numbers were maintained at a level of about 1.1-1.3
X 10^7/g over 98 days of pretreatment.”

The population of viable acidophilic biooxidizing bacteria present on ore in the
experimental biooxidation heaps at Gold Quarry was estimated using a most-probable-
number (MPN), or end point, dilution procedure with -10 mesh fractions of ore samples
(Brierley et al., 1995). Ore samples were first shaken overnight in flasks containing low
nutrient medium to dislodge bacteria from the ore. In addition, the total microbial population was estimated using acridine orange staining and epifluorescence microscopy, though this method does not distinguish between live and dead microbial cells. The viable microbial populations in subsequent heaps (demonstration heap and commercial biooxidation heaps) were determined using heap effluent solutions as a way of monitoring the microbiological health component of the biooxidation process.

In the current research, Newmont Metallurgical Labs (Englewood, CO) analyzed solution samples from the laboratory columns run at UNR by the MPN method and included “as sent” biopond solution used for the column experiments and subsequent column effluent solutions (biosolution). Thirty milliliters of biosolution was periodically collected from the effluent discharge of each laboratory biooxidation column, specifically bracketing large changes in temperature or solution appearance, and sent overnight at ambient temperature to the Newmont lab for analysis by their technical staff (Jack Tryon). A MPN dilution series (out to 10), consisting of 1 mL of sample solution diluted with 9 mL of modified Kelley medium (MKM) + iron, was prepared for each growth incubation temperature being used. MKM was prepared as follows: 0.04g/L K2HPO4, 0.40 g/L MgSO4·7H2O, 0.04g/L (NH4)2SO4 (then acidify with H2SO4 before addition of ferrous sulfate), 33.3 g/L FeSO4·7H2O4 (then adjust to pH 1.4 to 1.6 using H2SO4), and sterilize at 10 psi for 10 min or 15 psi for 5 min (MacKintosh, M.E, 1978). In most cases 30°C, 50°C, and 60°C were chosen for incubating the dilution series; 15°C and 75°C incubations were used in a few cases, based on the temperature profiles/history of particular biooxidation columns (Appendix II). In the case of diluted samples incubated at 60°C and above, a small amount of Bacto™ agar plus a small piece of pyrite were
added to the tubes because these additions were found to facilitate the growth of the extremely thermophilic (archaeal) microbes (J. Tryon, personal communication). Tubes from the dilution series were placed in appropriate water baths for growth of the various categories of iron-oxidizing microbes and left for 1 to 2 weeks until colony growth could be detected via the presence of greenish-colored areas in the media (conversion of ferrous to ferric iron). A 30°C temperature bath is optimal for the growth of the mesophilic strains (and probably some of the other microbes with higher growth optima), 50°C is the optimal temperature for growth of the moderate thermophilic strains, and 60°C for the extremely thermophilic microbes. It should be noted that many thermophilic archaea, once thought to be almost exclusively found in regions of high temperature, have now been identified in microbial populations present in locales that are more moderate in temperature. And some moderately thermophilic strains, such as Sulfobacillus thermosulfidooxidans, are known to have a broad temperature range for growth (40°C to 60°C) under certain conditions.

In addition, bacteria were visualized by microscopy using the hanging drop technique (Tortora et al., 1995). This method allowed a semi-quantitative assessment of the microbial populations with regard to the various strains of interest (Acidithiobacillus ferrooxidans, Leptospirillum ferrooxidans, Sulfobacillus thermosulfidooxidans, Acidianus species and Metallosphaera species).

Ore samples were taken from both the in-situ columns embedded in commercial biooxidation heap 8 (section C) at Gold Quarry, and the eight laboratory biooxidation columns at UNR. They were preserved via three techniques for future microbial analyses using molecular techniques, such as fluorescent in-situ hybridization (FISH) and/or
quantitative polymerase chain reaction (Q-PCR), which directly identify the various strains of microbes present and yield quantitative information on their numbers.

Separate biosolution samples were also collected at the same time and sent to ALS Chemex for total iron and iron speciation analysis. Commensurate ICP analysis of biosolution and crystalline sulfates were conducted to determine specific elemental concentrations in the biosolution at the University of Nevada, Reno, Nevada Bureau of Mines and Geology (UNR-NBMG) facilities.

XRD analysis of the sulfates was also conducted at UNR-NBMG facilities. Analysis of the more hydrous sulfate minerals, such as melanterite, is very difficult since it readily alters to rozenite. In initial trials, crushing the sulfate in petroleum jelly or mineral oil inhibited dehydration and provided only minor spectral interference. Analysis of the more stable, less hydrous, sulfates was also conducted by XRD using standard powder methods. Factors complicating mineral identification included multiple intergrown mineral phases and exceedingly minor differences in the individual mineral spectra. Combined SEM imaging and EDS analysis were used to identify individual sulfate mineral habits and associations.

Polished sections of select aggregate samples were used for sulfide sulfosalt mineral identification and paragenesis by reflected light and scanning electron microscope - backscatter electron and energy dispersive spectroscopy (SEM/BSE/EDS). Optical analysis of polished thin sections was used to identify sample alteration style and the corresponding efficiency of sulfide oxidation below the aggregate surface. These procedures and results are addressed in detail following the sulfate discussion (see page 65).
Results

In-situ Columns

The temperature readings from the in-situ columns indicated a temperature gradient that ranged from a high of 61.5º C to a low of 4.5º C over a distance of 3 m between 0.5 and 3.5 m depth. The high temperature recorded just below the surface (0.5 m depth) was 78.7º C; at 3.5 m depth it was 82.6º C. A Newmont probe at ~ 9.5 m depth and approximately 50 m inboard from the columns toward the center of the heap, registered a high of 87.4º C. A second Newmont probe near the center of the heap in section 8b and at a depth of 45 ft was used for the temperature profile shown in figure 12 due to later failure of the probe closest to the in-situ columns.

Large temperature fluctuations occurred in the periphery of the heap due to episodic irrigation (Fig. 12). This becomes necessary when the discharge effluent Eh decreases below approximately 500 mV and the solution begins to turn green, indicating a transition from oxidizing toward unfavorable reducing anoxic conditions within the core of the heap. When this transition was indicated during effluent monitoring, the irrigation pumps were turned on for short durations, generally two to three days, to slightly cool the interior of the heap (Fig. 12) and increase solution ferric iron and dissolved oxygen values in the biosolution. The reducing ferrous iron-rich green solution creates a non-oxidizing environment that rapidly lowers local biosolution pH to levels that may exceed microbial tolerances. Lab columns operated under these conditions produced effluent with pH values as low as 0.36. Heap effluent pH does not drop to such low levels because it is an average from across the entire heap where local zones operating under normal conditions will dilute anomalously low values. When this
solution was allowed to pond and evaporate, negative pH values beyond the measuring capacity of the pH probe of pH 0.0 were realized. Extensive precipitation of crystalline melanterite from solution also occurred. Average crystal size was approximately 5 mm; the largest crystal measured 4 cm across with a rhombohedral habit.

Figure 12. Temperature profile for in-situ column 4 over time. Depth indicates distance of temperature probe below the surface. The black trace is from Newmont’s probe at ~ 9.5 m. The sharp decrease from the Newmont probe is anomalous and probably erroneous. Shaded areas indicate active irrigation and generally correspond to heap cooling trends.

Locally, these low pH anoxic conditions may be generated in the heap, particularly at the base by restriction, and ultimately the total cessation, of induced air flow through the aggregate due to the formation of secondary sulfates around the air injection ports. Sulfates form preferentially around the ports due to the dehydrating
effects of injecting the normally very low humidity Nevada air. Often, especially during the summer months, the humidity levels are in the low teens to single digit range. This hot, dry air promotes nearly instantaneous sulfate precipitation from the already hot supersaturated biosolution. The sulfates may entirely clog the air injection ports that formed sulfates earlier or faster in the heap cycle, while creating sulfate-lined channels or ducts from other ports that slowly propagate toward the surface. This is reasonable, since only one squirrel cage fan provides the air for each pad. Air flow will follow the path of least resistance resulting in reduced air flow and increased probability of complete closure at one port while the resulting increased air flow at another port increases the potential for more extensive channelization. This effect was replicated in the laboratory columns and is discussed later (see page 64). The area around the clogged port will then develop a low pH, anoxic environment that essentially halts further oxidation by most of the microbes.

The surface of the biooxidation heap has randomly distributed steaming fumaroles and fissures due to venting from its high temperature interior (Fig. 13). Some of the vents may be partially linked by sulfate channelization to the underlying air injection system. During visits to the heap over its entire duration, no change in the vent distribution or specific locations near the in-situ columns was noted. Unloading of the pad tends to destroy these sulfate barriers as they would be most defined near the base of the heap. The ejection of steam indicates that the channels do not reach the heap surface probably due to a slow propagation rate relative to the heap operation time. Surface zones around the vents display multiple types of sulfate formation. The specific minerals deposited include halotrichite, goethite, alunogen, copiapite, rhomboclase and rare
gypsum. Sulfates also precipitated at the air exhaust port of the aerated biosolution storage tank for the laboratory columns.

Figure 13. Vent site on top of the heap with escaping steam not visible due to a hot low humidity day. Tan mineral is copiapite, white is halotrichite with minor gypsum, and red is goethite.

Optical and XRD (Appendix II) analyses of mineral precipitates of a hand sample from the upper zone of a previously completed pad indicate that alunogen, \( \text{Al}_2(\text{SO}_4)_3 \cdot 17\text{H}_2\text{O} \) (Fig. 14), the platy white mineral resembling muscovite, is primarily responsible for cementation of the aggregate. The fibrous white mineral is halotrichite, \( \text{FeAl}_2(\text{SO}_4)_4 \cdot 22\text{H}_2\text{O} \). The accompanying yellowish-tan coating on individual aggregate members is copiapite, \( \text{Fe}^{2+}\text{Fe}^{3+}_4(\text{SO}_4)_6(\text{OH})_2 \cdot 20\text{H}_2\text{O} \), with a minor associated clay component, probably dickite and kaolinite. Both dickite and kaolinite have been identified by thin section analysis of unoxidized ore as alteration products that are locally present within the Gold Quarry deposit. Rare jarosite may also be locally present high in the heap. These
same sulfate minerals, as well as local clay-rich zones and compaction during pad loading, result in the formation of mineral and permeability specific horizons within the heap that affect both air and fluid flow.

Figure 14. Hand sample of sulfate-cemented aggregate from a lens or umbrella zone high in the heap. Tan mineral is copiapite with minor associated clay minerals kaolinite and dickite.

The irrigation biosolution that is dripped onto the heap contains extremely high concentrations of many elements, with Fe, Al and Mg taking the lead. The elevated heap temperature, which on the surface may exceed 45°C, and the low atmospheric humidity promote rapid evaporation and therefore the formation of sulfates. These sulfates form a hard aggregate rind or cap on the heap (Fig. 15) restricting fluid percolation and air egress. The application of fresh water from rain or snow causes dissolution of the sulfates and a sudden local increase in pH resulting in increased precipitation of insoluble
goethite and jarosite within the cap and gradually increasing the cap’s thickness. Locally, the reduced permeability of the cap results in the surface accumulation of biosolution from the irrigation system which forms low pH puddles and ultimately sulfate crystal accumulations. Figure 16 shows the formation of melanterite crystals at the surface of the heap where the pH was 0.37.

Cracks or fissures that form in the crust due to local subsidence or adjacent to vehicle wheel ruts may allow for somewhat focused solution and air flow through the cap. During pad unloading, large local zones of sulfate formation were exposed at the base of the heap (Fig. 17). This type of late sulfate formation is probably due primarily to
convective vapor transport and to a lesser extent capillary style wicking. This may be indicative of solution ponding or saturation in the base or the heap. These zones may be caused by local compaction of the base layer during successive unloading cycles and excessive local formation of sulfates clogging drainage pathways. This style of sulfate clogging was found in several of the laboratory columns. Clogged air injection ports or associated channelization may also result in the formation of these zones. Replacement of the upper portion of the base layer and air injection tubing was conducted following every third pad cycle. Replacement of the base and tubing after each cycle was implemented in 2007 to address these issues. The long-term effects of these changes are unknown since the biooxidation program was terminated as of spring 2008.

Figure 16. A small puddle of biosolution with green melanterite crystals forming on the top of the heap. The solution pH was unexpectedly low at 0.37. Numerous such puddles are present on the heap amplifying crust/cap formation. The extremely low pH is also below the growth tolerances of the microbes added originally to the Gold Quarry inoculum.
Figure 17. Local zone of mass sulfate formation that has formed on top of the base layer of the heap following unloading, a process driven primarily by convective evaporation and capillary action. Such zones suggest local ponding or saturation of solution in the base layer of the heap which would also result in local anoxic conditions and reduced oxidation potential.

The four in-situ columns displayed a clear increase in sulfates, primarily copiapite, over time. The first extracted column (day 41) displayed a generally grayish-tan color to the ore with minor to moderate compaction and relatively common open spaces within the aggregate. During the extraction of column 4, the walls of the extraction hole clearly showed that extensive tan-colored copiapite had formed (Fig. 18). Figure 19 illustrates the progression of sulfate formation over time; by the time column 4 was removed from the heap (day 157), the color of the column contents was primarily tan with only rare, small diffuse areas of gray remaining. The void spaces within the aggregate were fewer and much smaller due to infilling by various sulfates with a paste-like consistency. This would serve to reduce permeability and encase individual aggregate clasts, minimizing oxidation potential. Further dehydration over several weeks
following the termination of heap irrigation allowed for the growth of the larger more
differentiated crystalline structures. The formation of these later macro-crystalline forms
ultimately resulted in the local agglomeration or cementation of the aggregate within the
heap exclusive of the channelization effects.

Figure 18. The exposed wall of the hole dug to remove the final in-situ column contained large zones rich in copiapite (tan area). The amount of copiapite in the upper portion of the heap increased markedly over the five-month column test period.
Figure 19. All four in-situ columns following their removal from the heap. A strong trend toward increased copiapite is clearly indicated by the increase in tan color and less open space. The columns also displayed increased compaction due to subsidence. Note distortion of column 4 (D) due to the high temperature within the heap.
Laboratory Columns

The laboratory column experiments were designed to simulate several variations of specific environmental conditions and operationally-induced cycles found within the biooxidation heap. Eight columns were operated with four at ambient temperature and four heated to approximately simulate two specific temperature environments within the interior environment of the heap. The three nominal operating temperatures were selected to preferentially optimize for specific microbes previously added to the biosolution pond at Gold Quarry. The biosolution for each column was contained in a dedicated reservoir. This allowed for some columns to be operated under very low pH and Eh, thereby simulating the extreme conditions expected at various times and locations within the heap. Multiple equipment failures also induced unexpected parameters on individual columns inadvertently producing interesting results or accidentally mimicking specific local heap conditions, such as solution ponding and the creation of anoxic environments. Other specifics such as sulfate formation both high and low within the columns, channelization, subaqueous sulfate precipitation in the reservoirs and sulfate formation from vapor by precipitation through condensation, were all successfully demonstrated.

The temperature profiles of the unheated columns displayed a close correlation with the prevailing weather conditions. A large negative spike occurred during a cold spell and the late general rise in temperatures correlated with the normal spring warming trend (Fig. 20). The microbial counts for these columns were generally good, but there was not enough mass present to provide sufficient insulation or generate enough heat to overcome thermal losses. These columns simulated only the outermost cover on the sides
of the heap as the large convective thermal losses through the top of the heap maintains an elevated surface temperature.

Figure 20. Temperature profile of unheated columns over time. Patterns mimicked prevailing weather conditions. Large negative spikes correlate with cold fronts. Late average increase occurred during spring warm up. Shaded areas indicate solution pumps running except for C7 off at dashed line due to equipment failure.

The heated columns more closely simulated the environmental and operating conditions found within the heap (Fig. 21). The intermediate temperature columns (L4 and L5) were operated at about 50° C and the high temperature columns (L3 and L6) at approximately 75° C. The actual maximum temperatures attained within the core of the heap approached 90° C which would have compromised the structural integrity of the laboratory columns, as demonstrated by the distortion of in-situ column number 4 (Fig 19D). A shutdown period mandated by an overseas fieldtrip is indicated in Figure 21.
The early narrow temperature peak immediately followed by a dip was due to initial equipment failures at startup and a partial shutdown to a lower maintenance temperature for necessary retooling. The cycled columns (L5 and L6) were each subjected to three gradational cycles that were implemented in 10° to 25° C increments. Minor differences in the relative temperature of the two high-temperature columns as well as the two medium-temperature columns at the same points in time may be due to variances in column insulation and effective convective heat loss. All of the heated columns also suffered random temporary irrigation stoppages due to sulfate formation in the tubing. These stoppages generally lasted less than 24 hr.

![Temperature profile of heated columns over time. Columns 5 and 6 were varied intentionally to mimic actual heap operating conditions. Shaded area indicates complete shutdown of all columns heaters, pumps and air. Dashed red lines indicate when heaters were turned on.](image)

Figure 21. Temperature profile of heated columns over time. Columns 5 and 6 were varied intentionally to mimic actual heap operating conditions. Shaded area indicates complete shutdown of all columns heaters, pumps and air. Dashed red lines indicate when heaters were turned on.
The large ~ 21°C temperature drop from day 24 through 27 for column 3 was due to extremely efficient convective loss prior to drain clogging and the commensurate cessation of induced air flow. This efficient air flow and convective loss was only achieved in the early stage of column operation. Sulfate formation and aggregate compaction due to ore slump are primary contributors to decreased air flows and resulting higher column temperatures. The more subtle alternating temperature transitions correlated with the periodic resumption of column drainage and air flow and recurrent clogging of the drain by sulfates. Minor temperature fluctuations may also have been influenced by relatively large temperature variances in the local weather. The transport of fines through the columns was negligible primarily due to the very low biosolution irrigation rate and was not a factor associated with the drain blockage issues.

The pH trends over time generally aligned with temperature transitions. All columns showed a slight increase in pH following an initial sharp decrease (Fig. 22) after initiation of irrigation. The initial rapid decrease in pH was probably due to the nearly instantaneous oxidation of extremely fine-grain sulfide partials liberated during crushing. This is analogous to the instantaneous oxidation of sulfides (black water) when cutting with a water-cooled diamond saw. The unheated columns displayed a generally steady decrease in pH over time with only minor increases when the reservoirs were recharged. Biosolution was added to the reservoirs for the first 45 days of column operation to maintain reservoir levels during initial ore saturation and to replenish losses due to evaporation or convection and several random leaks from equipment failure or clogging. After that time, distilled water was added rather than biosolution in order to maintain reservoir solution levels and increase solution pH without modifying the resident
microbial populations (or lack thereof) by adding microbes contained in the initial biosolution used to irrigate the columns. The addition of distilled water did not force the precipitation of sulfates or oxides since the solution pH was never increased above 1.5. The unheated columns suffered only minor evaporative losses from their reservoirs and no convective loss and therefore required only minimal biosolution or distilled water addition. The unheated columns also did not suffer any drain clogs or realize significant sulfate formation other than from condensation on the reservoir walls with the exception of column 2, which formed sulfate channels (Fig. 23).

![pH of Lab Columns vs. Time](image)

**Figure 22.** Column effluent pH over time. The sharp increases after day 30 are due to periodic dilution with distilled water for all column reservoirs and partial solution removal from heated column reservoirs due to excessively low pH. Shaded area indicates biosolution pumps off. Dashed red lines indicate heaters on. The pH values of the heated columns displayed an immediate divergence at the beginning of the experiment compared to the unheated columns. The heated columns displayed a rapid pH decrease with temperature increase.
At approximately 60 days, the average rate of pH decrease for both heated and unheated columns became nearly identical as illustrated in Figure 22. The heated columns displayed three sharp pH increases due to partial solution removal and replacement with an equal volume of distilled water. The dilution was necessary in order to compensate for excessively low solution pH levels. The excess solution was retained in buckets and allowed to evaporate, resulting in the formation of various sulfate minerals discussed below. The reservoir for L3 also contained a large quantity of melanterite crystals that had precipitated from solution (fig. 24).

Figure 23. (A) Light gray area (arrow) in the sulfate cemented ore is a dry air channel ~ 1.75 m from the bottom of L2. The air channel is separated from the still damp portion of the ore by a thin band of sulfate. (B) Two sulfate bands in another ore segment 0.5 m lower in the column. The longer band probably formed after the first channel became clogged with sulfate.
The pH of L3 reached a minimum of 0.35 and dipped below 0.4 on four occasions prior to initial dilution. Following a clogged drain, the solution went green in color which is representative of anoxic conditions. The drainage clog was preceded by the formation of a melanterite stalactite at the bottom of the drain tube (Fig. 25). Figure 26 illustrates melanterite forming on the drain tube of L4 due to vapor transport. The anoxic green solution was accompanied by a sharp decrease in both pH and Eh. Convective loss was also greatly reduced due to the cessation of air flow through the column. Significant air throughput was not reestablished due to sulfate aggregate agglomeration (Fig. 27) and commensurate air redirection out of the drain tube.

Column 5 also experienced a clogged drain and went anoxic but recovered and returned to an oxidizing (red) solution when additional biosolution was added and the drain cleared. Solution removed from the L3 reservoir and left to evaporate in a bucket produced pH values that went below the measuring capability of the equipment to register at 0.0. The pH of column 3 only briefly surpassed a value of 0.45. This suggests

Figure 24. Crystals from L3 solution. (A) Very large 4 cm melanterite crystal with a rhombohedral habit. (B) Smaller melanterite crystal with a platy layered appearance.
that negative pH values may be realized locally within the heap. It was also necessary to remove approximately two gallons of green melanterite crystals from the reservoir for L3

Figure 26. Melanterite forming on the drain of column 4 due to vapor transport. Notice sulfate formation also on the underside of the column support table.

Figure 25. Melanterite stalactite forming from the drain on column 3.

Figure 27. Sulfate-cemented aggregate from the bottom of L3 where air flow was completely stopped by sulfate clogging. The hard, dry microchanneled appearance evident in Figure 28B has not formed here due to high local humidity.

in order to allow for an increase in solution pH. Distilled water was then added to increase pH and attempt to dissolve the remaining melanterite crystals. The melanterite crystals slowly decreased in size but many did not completely dissolve as the solution remained near saturation. This suggests that once crystalline rather than efflorescent melanterite forms within the base of the heap, it is resistant to rapid dissolution by fresh
water wash and the bulk of the crystals will remain in place until physically removed. This may have a negative effect on gold recoveries due to the deleterious nature of ferrous iron on the mill circuit. Oxidized sulfates, where only ferric iron is present, do not negatively affect the mill circuit.

The heated columns experienced much larger decreases in Eh (oxidation potential) than the non-heated columns (Fig. 28). Unlike pH, the effects of dilution on Eh were less pronounced in most columns. The large late-term increase in Eh associated with L7 is anomalous as its reservoir was still aerated, but irrigation was terminated ~ 25 days prior to the rest of the columns due to equipment failure. Continued oxygen input into the reservoir without the addition of drain effluent allowed for the total oxidation of all residual ferrous iron, thereby increasing both pH and Eh. L3 maintained the lowest average Eh and reached the lowest absolute pH over the course of the experiment. This column also produced the largest quantity of sulfate minerals, primarily precipitated in solution as melanterite. During the first month of the experiment, L3 possessed the highest rate of convective air flow and was also at the highest temperature.

One or more columns were temporarily shut down on three separate occasions. All columns were completely shut down for 19 days from day 83 to 102. Columns 3 through 6 lost solution irrigation for 2 days due to a pump drive failure. Unexpected increases in pH occurred immediately following the restart of columns on day 102, in all columns except L1 and L7, and at day 125 for L6. Dilution with distilled water accounts for the pH increase associated with L2 and L5 - L8 in every instance. Column 1 exhibited the expected decrease in pH while columns 3 and 4 show an unexpected increase in pH. Columns 3, 4 and 6 also display minor increases in pH while column 5 was relatively
unchanged following pump failure. Column 7 was permanently shut down early due to a tubing failure on day 120 and also displayed a minor follow-on increase in pH. Minor variations may be explained by the limitations of measuring equipment. Larger variations remain unexplained.

Figure 28. Column effluent Eh over time. Increased column temperature generally corresponds to decreased Eh. Shaded area indicates columns off. Dashed red lines indicate heaters on. Dashed blue line indicates heaters off. Data prior to day 42 not taken, except for starting solution due to lack of equipment. Late increases for L5 and L6 are due to increased dilution.
The unheated columns produced far fewer sulfate minerals than the heated columns both in type and volume. The unheated columns only produced a tan-colored mineral on the walls of the reservoirs that was identified by XRD analysis as copiapite (Fig. 29A). The heated columns all produced melanterite on reservoir walls and suspended tubing (Fig. 29B, 30A). Melanterite rapidly dehydrates to rozenite. Multiple XRD spectra captured over time depict the formation of melanterite and its subsequent dehydration to rozenite (Appendix II). The other sulfates on the reservoir wall include copiapite (tan), halotrichite (white) and rhomboclase (purple). Rare gypsum crystals are intergrown with the copiapite. Alunogen was not observed to have formed from vapor transport.
Column 3 produced ~ 10 pounds of melanterite that ICP indicates does not contain any appreciable contaminants. Many of the crystals were large and euheral with a rhombohedral habit (Fig. 24). The other heated columns only produced small amounts of melanterite following significant solution evaporation.

When the columns were dismantled, three of the unheated columns displayed minor local copiapite within the aggregate and were easily sampled via the sample ports.

Figure 29. The tan sulfate mineral on the wall of an unheated column reservoir is copiapite (A). Multiple, preferentially located, sulfate minerals on the wall of a heated column reservoir (B). Green mineral is melanterite and white mineral physically associated with the green is rozenite. Purple color on tan is rhomboclase and the massive white mineralization is halotrichite. All reservoirs displayed similar sulfate formation through vapor transport.

Figure 30. Melanterite (green) and rozenite (white) on solution tubing of column 3 (A). Melanterite, rozenite, and other sulfates on reservoir wall. Acicular melanterite crystals on glass slide (B).
Unheated column 2 was agglomerated by sulfate and had to be cut open with a circular saw. Approximately midway down the column a channel of dry ore became apparent (Fig. 23A). Another 0.5 m down the column, a second possibly earlier channel that had probably become internally clogged with sulfate became visible (Fig. 23B). Both of the channels tended to neck down with height. Column 2 also realized the lowest average pH and Eh of the unheated columns. This, along with a larger volume of distilled water required to maintain reservoir level, indicates that L2 maintained a higher rate of air flow resulting in more low-temperature evaporative loss prior to the restriction of the air flow to the sulfate lined channel. Initial sulfide oxidation within column 2 may also have been more rapid as more sulfate was precipitated within the column and on the reservoir walls relative to the other unheated columns. The sulfate-mediated channelization of air within L2 ultimately reduced the total ore volume exposed to biosolution and efficient oxidation as the experiment progressed.

The sulfate band or ribbon in L2 was thin in its upper extent, and thickened dramatically with depth. The width of the actual band varied from approximately 2 to 8 mm (Fig. 31A). There was a clear delineation between the darker damp ore outside of the channel where biosolution flow was concentrated and the light-colored dry ore inside the channel where air flow was constrained. The sulfate forming the band was comprised primarily of a mix of very fine-grain copiapite and halotrichite. Microchannelization and sulfate encapsulation of individual aggregate fragments intensified dramatically at the bottom of the column (Fig. 31B). In columns where the air flow was interrupted, the sulfate still formed (Fig. 27) but without the characteristic sculpted microchannels evident in Figure 31B, as illustrated below.
Figure 31. (A) Column 2 sulfate channel boundary varied from 2 to 8 mm. The band was continuous and relatively straight. Notice clear difference in moisture content where dark = damp and light = dry. (B) At the base of the column, narrow individual channels abound. Extent of connectivity is unknown. Much of the aggregate is completely encapsulated by sulfate.

**Sulfate analysis**

Sulfate identification was accomplished by both XRD (Appendix II) and SEM/EDS (Appendix III) methods. Many of the fine-grain phases were intricately intergrown resulting in complex XRD spectra. Through SEM imaging and EDS analysis, these phases were identified along with specific crystal habits and trace element contamination. Surprisingly, no sulfates with detectable arsenic or arsenates were identified.

The melanterite generally contained no trace contaminants. Copiapite was commonly found with trace to moderate levels of zinc and locally with magnesium. Copiapite exhibited a fine-grain platy habit with random orientations. Where magnesiocopiapite was encountered, it displayed a pronounced radial pattern (Fig. 32). Halotrichite was found to exhibit two distinct habits. Where halotrichite occurred exclusively, it tended to exhibit a wispy sheet-like texture (Fig. 33). Halotrichite is more commonly found intergrown with copiapite, forming broad-based needles that appear to
grow directly from the platy copiapite substrate (Fig. 34). The halotrichite needles are composed of bundles of spaghetti-like strands. These needles generally grow in hemispherical groups but may also be found in randomly oriented clusters. Alunogen forms plates that locally may form books reminiscent of muscovite. The books may grow to several millimeters in all dimensions and are easily identifiable in hand sample. Alunogen also may form carbonate-like rhombs and is commonly found with intergrown halotrichite (Fig. 35).

Figure 32. SEM photomicrograph of Magnesiocopiapite displaying a radial platy habit. Needles are halotrichite.

Figure 33. SEM photomicrograph of halotrichite displaying wispy texture. Platy substrate is copiapite.
Figure 34. SEM photomicrographs of halotrichite needles. Platy substrate is copiapite. Note spaghetti-like fibers comprising the needle.

Figure 35. SEM photomicrograph of alunogen displaying platy and locally rhombic habit. Needles are halotrichite.
Rhomboclase was less common and appeared to only form late from vapor condensation on earlier halotrichite. Small dark spheres that also formed late are an unknown Fe-Mg-Al sulfate that is also locally vanadium rich. The spheres are generally approximately 1 mm in diameter (Fig. 36A) and have an opaline luster. They possess an outer rim around a vanadium poor core and all examples contain trace to moderate zinc concentrations. All vanadium-rich zones displayed a very distinct crystal habit of randomly shaped interlocking plates with a strong preference for sides with relative angles of $135^\circ$ and $90^\circ$ (Fig. 36B). Bounding sulfate that is similar in chemistry but lacking vanadium has a fibrous rather than platy habit. More in-depth analysis is required to establish the stoichiometry and crystalline structure of these sulfates.

Figure 36. (A) SEM photomicrograph of a spherical vanadium-rich sulfate. (B) Only the ring partially depicted in the enlargement contains vanadium. Note unusual crystal habit.
Solution chemistry

Biosolution samples were periodically collected from the column drains and reservoirs. ICP analysis of 21 elements was conducted on all samples (Appendix IV). Resulting elemental concentrations from the drain and reservoir of each column were averaged to compare the oxidation efficiency of all the columns (Appendix V). Iron concentration and speciation were determined by titration (Appendix VI).

The Excel graphs of the ICP analyses tended to correlate with column operating parameters such as air and fluid flow. When column operation was initiated, column 3 possessed the most efficient convective air flow. This was clearly represented in the ICP analysis by substantially higher solution concentrations for most elements. Arsenic is a notable exception to this trend, for as yet unknown reasons, but the anomalies are restricted to the heated columns. Column 3 suddenly clogged due to melanterite formation at its base, as evidenced by a melanterite stalactite emanating from the drain tube. Column 3 never regained convective air flow but produced a substantially larger volume of sulfate both within the column and the reservoir. Column 3 produced more sulfate on the reservoir walls, was the only column to directly precipitate sulfate (melanterite) from solution in the reservoir, and routinely suffered irrigation tubing blockages due to melanterite crystal formation.

Column 2 was the most efficient of the unheated columns in terms of air flow and sulfide oxidation as indicated by the ICP analyses. This was ultimately represented by the formation of sulfate channeling, which is indicative of a larger air volume throughput than the other unheated columns. Bar graphs of average individual elemental concentrations appear to give accurate column efficiency representations. While the
arsenic anomalies remain unexplained, antimony also does not correlate with apparent column efficiency and, like arsenic, is restricted to the heated columns.

The ferrous iron to ferric iron ratios ($\text{Fe}^{2+} : \text{Fe}^{3+}$) track column efficiency as expected. Higher values accompany periods of clogged drains and lost air flow. Those columns that were able to reestablish drainage realized a partial recovery. Substantial air flow was not reestablished in any column other than column 2, which then generated the only sulfate channel.

Columns 2 and 3 each exhibited important parameters necessary for efficient biooxidation, although under different environmental conditions. Both columns experienced substantial air flow. Column 2 maintained air flow over a greater period of time but was unheated and only realized limited oxidation compared to the heated columns. The prolonged air flow also resulted in the formation of the sulfate channel which limited late stage oxidation. Column 3 ($75^\circ C$) initially operated at an extremely accelerated rate of oxidation due to optimal temperature and air flow conditions, but these conditions also led to the premature loss of drainage and air flow due to excessive sulfate formation. These were the two most efficient columns, but due to their own success they also suffered the most catastrophic failures; yet they still realized the highest oxidation levels relative to the other columns within their respective groups.

**Microbial populations**

Microbial viability analysis was conducted at Newmont labs in Denver, Colorado. Samples were collected four times (January 5, March 2, March 15, April 26) during the column experiments. The first sample collection was only from the drains for all columns. The second set of samples was from the drains for the unheated columns and
from both the drains and reservoirs for the heated columns. The final two sample sets were collected from the drains and reservoirs for all columns. The biosolution samples were shipped to Newmont where they were stained and visually counted using a fluorescence microscope. Results are reported in appendix VII. The primary focus was on determining the early microbial populations and later, the resulting microbial population demographics near the end of the experiments. This information was used to establish survivability criteria for specific microbial species under a given set of environmental conditions.

Column 2, which attained a low pH of approximately 0.63, had a bacteria count totaling $10^6$ cells/ml near the end of the column test. Column 2 was unheated meaning only those bacteria populations favored in ambient low temperature environments are represented in numbers above the biosolution reservoir population levels. *Leptospirillum ferrooxidans* was identified in the first sampling and *Acidithiobacillus ferrooxidans* was not. The second sampling registered a decrease in *Leptospirillum ferrooxidans* counts and the appearance of *Acidithiobacillus ferrooxidans. Acidithiobacillus ferrooxidans* numbers increased steadily over the full term of the experiment from not observed to $10^6$ cells/ml. *Leptospirillum ferrooxidans* was not recorded following the second sampling date. Columns 1, 7 and 8 generally mimicked column 2 with only minor variations in the second sampling counts for *Leptospirillum ferrooxidans*.

The heated columns displayed changes in population speciation but not density at the second sampling. The intermediate-temperature columns added a thermophilic microbe population to the preexisting mesophilic population and the high-temperature columns suffered the elimination of the mesophilic population, which was replaced by
thermophilic bacteria and archaea populations as determined by microbial viability counts.

By the third sampling, microbial populations in the heated columns showed only a slight increase in density of one order of magnitude from $10^1$ - $10^2$ to $10^2$ - $10^3$ cells/ml with the exception of column 5 (14 - 50°C), which realized an increase in the mesophilic microbial population density of four orders of magnitude to $10^5$ cells/ml and an increase in the thermophilic population density of two orders of magnitude to $10^3$ cells/ml. Column 5 was cycled through heating and cooling periods which allowed mesophilic populations to recover. This column also generally maintained a higher convective air flow rate than column 4 (50°C), verifying that sufficient air flow facilitates for increased microbial activity. Column 3 (75°C), continued to suffer severely restricted air flow and realized only a single order of magnitude increase in thermophilic microbial density from $10^2$ to $10^3$ cells/ml whereas, column 6 (15 - 75°C), which also underwent temperature cycling, realized an increase of two orders of magnitude from a lower starting point of $10^1$ to $10^3$ cells/ml. This suggests that a constant air supply is more critical to maintaining or increasing microbial density than any possible deleterious effects of temperature cycling within the ranges studied over the life of the heap.

The microbial population density for the intermediate-temperature columns remained stable from the third sampling date through the remainder of the experiment. The high-temperature columns realized a marked increase in both thermophilic bacteria and archaea population densities ultimately overtaking the intermediate columns in both categories. Column 3 realized an increase of three orders of magnitude from $10^3$ to $10^6$ cells/ml for the thermophilic bacterial population and an increase of one order of
magnitude for the archaea population from $10^2$ to $10^3$ cells/ml. Column 6 realized an increase of three orders of magnitude from $10^3$ to $10^6$ cells/ml for the thermophilic bacteria population and an increase two orders of magnitude for the archaea population from $10^2$ to $10^4$ cells/ml. Column 6 was sampled twice in quick succession only 4 days apart (April 26 and 30) as it was registering an internal temperature 5.5° C higher than the heater setting of 21° C. Viable microbial analyses indicated a ten-fold increase in archaea population density from $10^3$ to $10^4$ cells/ml, however these numbers are still very low. While this occurred during a temperature lowering phase for this column, it was indicative of a self-sustaining biooxidation reaction as no the column temperature never exceeded the heater temperature setting for any of the other columns. This may not represent a real increase as the overall trend for archaea population growth appeared to severely lag bacterial population growth throughout the experiment. Archaea replication may be slower than bacterial replication, or optimum replication parameters were not achieved and may only have been approached near the terminus of the experiment.

In summary, microbial viability analyses to date suggest that the minimum survivable pH threshold for microbes in the biosolution may be lower than previously reported. Appendix VII lists the microbial counts for a specific species or thermal preference group (i.e. mesophilic, thermophilic or archaea). The strains of archaea present in the biosolution appear to be more sensitive to very low pH environments as the only archaea population count to exceed $10^3$ cells/ml occurred in the column with the highest pH at 1.16 to 1.09 (column 6). But, other factors or conditions not yet recognized by this study could also be at work within the various column environments to either decrease or enhance the number of viable archaea present in the samples. In addition,
factors involved with shipment to Denver for analysis may also affect the reported results. Further work beyond the scope of this study is necessary to refine the pH ranges specific to individual microbial species and relative thermal environments.

**Aggregate Sample Acquisition**

**Laboratory Columns**

Freshly crushed non-oxidized and non-inoculated ore was sampled directly from the secondary crusher discharge conveyor at the haul pack loading station and loaded into plastic-lined 55-gallon steel barrels. The ore was then shipped from Gold Quarry to the University of Nevada, Reno along with a 250-gallon tank of biosolution from the biooxidation solution storage pond (biopond). Thirty four samples were collected from the non-oxidized ore for petrographic and microbeam analysis.

Following the completion of the biooxidation experiments, samples were collected from three zones (bottom, middle, and top) within each of the eight laboratory columns via sample ports prior to unloading the columns. Four to five aggregate samples were collected from each port for a total of one hundred samples. Multiple samples were collected from each sampling point in an attempt to represent all lithology and alteration styles. Samples were immediately washed and air dried thereby minimizing any further oxidation. Three criteria for sample selection were utilized.

1. Sample size- large enough to cut at least one billet with sufficient sample remaining for further analysis in the future if necessary.

2. Color- light tan or yellow is most highly oxidized, grey indicates siltstone or limestone, dark to black is generally highly silicified and is commonly brecciated.
3. Surface texture and morphology- irregular shape and a rough surface texture are generally indicative of intense silica flooding or an intensely silicified breccia. Tabular shapes or planar surfaces generally indicate a laminated weakly silicified or more intensely argillized siltstone or limestone. Note: These textures are due in part to crushing and are not necessarily applicable to in-situ or blast muck ore samples.

**In-situ Columns**

Sample bags were loaded with composite ore obtained from each of the four holes excavated during installation of the in-situ columns. The columns were installed contemporaneous with initiation of heap irrigation. The ore samples were air dried at 40°C for four to five days. Four to five samples were collected from each of the four bags of ore following drying for a total of 15 samples. Four to five samples were also collected from four equidistant zones within each column following their removal from the heap for a total of 62 samples. Sample selection criteria were the same as for the laboratory columns listed earlier. The samples were then washed and dried. During pad unloading, approximately four months after cessation of heap irrigation and the commensurate removal of the final column, 12 additional samples were collected from the heap near the original installation location of the in-situ columns. Four samples were also prepared from aggregate collected during the unloading of an earlier heap for comparison purposes. Depth of origin for these final 16 samples is unknown as they were collected from an active dig-face.
Sample preparation

The maximum dimensions of each sample were recorded and the sample was then cut into a billet oriented at approximately 90° to bedding or foliation where discernable or possible. Two hundred thirty eight one-inch diameter polished rounds were made utilizing UNR facilities to reduce cost and time. The vast majority of the rounds were drilled from cut slabs using a one-inch diamond core bit to maximize the available surface area of the completed section. Those too brittle for drilling were cut to size or fashioned from fragments to attain maximum surface area. The samples were then cast in one inch molds using thin-set epoxy to maximize atmospheric impregnation (without vacuum) and minimize bubbles. The samples were then polished on a Buehler echo met polisher using a six-section holder. Polishing grits comprised 120 and 180 grit diamond disks, 240, 380, 480 and 600 grit paper and 9, 6, 3 and 1 µm diamond solution on cloth.

Polished section distribution was as follows:

- Previous heap from 2005- 4 sections
- Composite samples from in-situ column installation- 15 sections
- In-situ columns- 65 samples
- Post in-situ column heap dig-face- 12 sections
- Unoxidized laboratory column ore- 35 sections
- Laboratory columns- 105 sections

Fifty eight of the 238 total samples were selected for optical analysis in order to categorize lithology and alteration styles. Where possible, samples representative of each lithology and alteration style were selected from all specific oxidation environments.
Polished thin sections were prepared by a commercial contractor. Polished section distribution was as follows:

- Previous heap from 2005 - 1 section
- Composite samples from in-situ column installation - 3 sections
- In-situ columns - 18 samples
- Post in-situ column heap dig-face - 8 sections
- Unoxidized laboratory column ore - 14 sections
- Laboratory columns - 14 sections

**Petrography**

**Transmitted light**

Transmitted light petrography was conducted primarily to further resolve specific lithology and alteration styles and associate them with certain hand sample characteristics such as color, shape and surface textures that relate directly to varying degrees of permeability and silicification. These specific characteristics may then be used to visually estimate a given ore’s amenability to heap style biooxidation.

Sericite veins commonly cut and offset quartz veins as illustrated in Figure 37. Figure 38 shows a quartz vein cutting a stylolite. Stylolites were found in many samples including rare examples with cross-cutting stylolites at approximately 90°. The stylolite in Figure 38 is the only one found to date that was infilled with sericite. Other stylolites in the same section did not display this unique mineralogy.

Vug infill was common with varying types of minerals filling the vugs. Open fill vugs are commonly lined with euhedral quartz crystals. Replacement style infill lacks a
quartz lining. Locally, the primary vug fill minerals were comprised of sericite, barite, calcite, dolomite, alunite and dickite, with rare local gypsum.

Multiple veining events are common throughout the Gold Quarry deposit. Quartz veins predominate, however sericite, barite and calcite veins are locally plentiful. Only one alunite vein was identified in the 58 samples examined for this study. Locally, reactivated quartz veins may display a late sericite or alunite event. Barite commonly fills open space in euhedral quartz lined veins.

Six of the polished thin sections displayed some degree of crystalline jarosite. Sample C443 displays crystalline jarosite throughout and total sulfide destruction except where protected by silica encapsulation. This is probably due to in-situ supergene oxidation rather than heap biooxidation. C431 and 444 displayed only minor crystalline jarosite that was restricted to open space vein and fractures that are open to the aggregate surface. This can probably be attributed to jarosite precipitation from the biooxidation solution as both samples are from at or near the bottom of the final in-situ column. Sections P82 and P84 showed minor near-surface and fracture or open space vein-fill
crystalline jarosite. Section P86 displayed more extensive near-surface oxidation to approximately 4 mm depth perpendicular to bedding and 8 m along bedding with accompanying near total sulfide destruction. The differences in the depth of oxidation are due to decreased permeability across bedding relative to along bedding resulting in decreased diffusion. Locally, the oxidation intensity gradually decreased inward over the final 1 to 2 mm. The oxidized rind covers the entire surface of the sample which along with the lack of crystalline jarosite elsewhere in the sample suggests the oxidation front was due to biooxidation in the heap rather than in-situ. P86 also displayed a more coarsely crystalline silicification texture along with ~ 80% sericite destruction or loss in the oxidized zone.

**Reflected light**

**Iron sulfides**

Each polished section was first analyzed under reflected light using an Olympus BX51 microscope for sulfide mineralogy and related textures. Points of interest were photographed and located on a map created on a scanned image of each section. Specific sulfide grains and assemblages were selected for further examination by SEM/BSE/EDS and microprobe analysis.

**Arsenian pyrite**

Arsenian pyrite was the topic of primary interest in this study as it sequesters the majority of the gold in these rocks. Arsenian pyrite rims occurred locally on most pyrite crystal habits (Figs. 39, 40) with the exception of frambooidal pyrite. Arsenian pyrite also occurred as local very fine-grain disseminated individual grains within the matrix or as local agglomerations or clouds (Fig. 41). The clouds were commonly strata or fracture
controlled or locally displayed a discontinuous vein-like appearance when present in a breccia matrix. Most samples that contained abundant pyrite only locally displayed arsenian rimmed pyrite. The majority of the pyrite occurred either without rims, had rims that were less than 1 \( \mu \text{m} \) wide and were consequently difficult to verify due to SEM/BSE edge effect or had arsenic concentrations below the detection limits of the SEM. Due to the extremely low gold values associated with each of these scenarios, they are of minimal importance. The arsenian rims identified to date during this study locally vary in thickness from approximately 12 - 15 \( \mu \text{m} \) to less than the minimum SEM/BSE resolution of approximately 0.1 \( \mu \text{m} \). Arehart et al. (1993a) reported arsenian rims up to 25 \( \mu \text{m} \) thick, however no specific location within the Carlin trend was given and no arsenian rims approaching this size were recognized at Gold Quarry during this study. The rim thickness also may vary substantially on an individual pyrite grain although, in many cases, this may be apparent thickness rather than true thickness due to the change in aspect of the various crystal faces relative to the plane of the polished surface.

The majority of the marcasite present in some samples appeared to be paragenetically late and is not arsenian nor is it rimmed by arsenian pyrite. The late non-rimmed marcasite may be found locally as vein material that also encompasses arsenian rimmed pre-ore pyrite (Fig. 42). Vein-style marcasite was more commonly associated with arsenian pyrite than the more widely distributed anhedral marcasite crystals. Locally, vein-style marcasite may be found overgrown by and or overgrowing arsenian pyrite.
Figure 39. Euhedral pyrite with arsenian rim. Matrix is quartz. Rim thickness varied from approximately 3 to 12 µm. Field of view is 0.43 mm.

Figure 40. Anhedral pyrite with arsenian rim. Matrix is quartz. Rim thickness varied from approximately 3 to < 1 µm. Field of view is 0.43 mm.

Figure 41. Subhedral pyrite with arsenian rim in halo of disseminated arsenian pyrite. Field of view is 0.43 mm.

Figure 42. Vein marcasite displaying two generations. Arsenian rim on adjacent pyrite crystal is not visible on vein under reflected light but is by SEM/EDS and on non-eroded faces of the pyrite crystal. Field of view is 0.43 mm.

Many obvious rims detected under reflected light are not arsenian. Figure 40 shows a pyrite crystal with a marcasite rim. The arsenopyrite in Figure 41 is itself overgrown by arsenopyrite. Euhedral arsenopyrite was also commonly overgrown by
pyrite (Fig. 45) and only rarely arsenian pyrite (Fig. 46). The early stage pre-ore arsenopyrite was predominately euhedral and commonly extremely elongate.

Figure 43. Anhedral pyrite with a marcasite overgrowth. Field of view is 0.43 mm.

Figure 44. Anhedral arsenopyrite with arsenopyrite overgrowth. Field of view is 0.43 mm.

Figure 45. Euhedral arsenopyrite with pyrite rim. Field of view is 0.43 mm.

Figure 46. Euhedral arsenopyrite with arsenian rim. Note grey rutile. Field of view is 0.43 mm.

**Base metal sulfides and sulfosalts**

Many other sulfides were identified in these samples. Base metals were relatively rare overall with only a few samples containing significant concentrations. Sphalerite generally occurred as small inclusions in pyrite and marcasite veins and locally totally
replaced pre-ore euhedral pyrite (Fig. 47). Sphalerite was also found overgrowing stannite (Fig. 48) and in association with chalcopyrite. Rarely, sphalerite displayed chalcopyrite disease. Chalcopyrite also appeared to replace early pyrite and was locally associated with tennantite and tetrahedrite as well as rare bornite and covellite. Galena was only found as rare very small inclusions in pyrite only detectable by microbeam analysis. Other sulfide minerals generally occurred as inclusions or in intergrown fine-grain replacement masses and could only be identified by SEM. Further discussion concerning these minerals is included in the microbeam section below. Continuing work beyond the scope of this study has identified several other sulfide and sulfosalt minerals and vein assemblages that will be discussed in a future dissertation.

**Textures**

Multiple interesting and locally unique sulfide textures were identified in the current study. These include radial platy pyrite, probably after marcasite, with marcasite infilling between the plates (Figs. 49, 50). They occurred in a generally linear trend and
probably formed along a small open structure in samples of silicified breccia. Framboids have been reported as common in many areas of the Carlin trend (Arehart et al., 1993a; Theodore et al., 2003). Framboids have only been identified in three samples to date (Fig. 51). Early stage euhedral arsenopyrite was very often and generally displayed an extremely elongate habit with a length to width ratio that may locally exceed 100:1 (Fig. 52).

Figure 49. Radial platy pyrite after marcasite. Infill between plates is marcasite. Texture associated with small vein-like structures. Field of view is 0.85 mm.

Figure 50. Radial platy pyrite after marcasite. Infill between plates is marcasite. Slightly crossed polars texture associated with small vein-like structures. Field of view is 0.43 mm.
Due to the fact that all samples were collected from previously crushed and blended ore, their points of origin from within the pit are unknown. Consequently, sulfide paragenesis can only be determined on an individual sample basis. The primary focus of this study has been on gold-bearing sulfides and the representative levels of oxidation as related to arsenian pyrite phases in specific lithologies and alteration zones. Ongoing work beyond the scope of this study is attempting to associate these various minerals, textures, lithologies and alteration styles with spatial elemental and structural features and generating tighter paragenetic constraints.

**Microbeam analysis**

**Iron sulfides**

All samples were examined by SEM/BSE/EDS procedures on a JEOL JSM-840A scanning electron microscope. Samples displaying visible rims, overgrowths or multiple sulfide assemblages under reflected light were selected for more detailed analysis. Samples with only fine-grained disseminated pyrite or marcasite were only analyzed for
arsenic content. Samples with small inclusions or assemblages of intermediate sulfides were further analyzed on a CAMECA MBX550 microprobe. Further analysis examining the possible existence of silicification of arsenian pyrite rims was also conducted using the microprobe.

Arsenian rims

Many rims and overgrowths were visible under reflected light, but their relative arsenic content and therefore possible gold association may not be postulated as the optical properties of arsenian pyrite are very similar to barren pyrite. Locally, texture or color variations, explained in detail below, may be used to differentiate between arsenian and non-arsenian pyrite. Other issues that may serve to complicate arsenian pyrite identification include extremely narrow rim width (Fig. 53), very small host grain size (Fig. 54) or relatively low arsenic values. Commonly, arsenian pyrite rims associated with overgrowths, pyrite or marcasite veins, anhedral host crystals or colloform pyrite cannot be optically differentiated and require SEM/BSE analysis for rim recognition (Fig. 55). Due to low gold values, the auriferous nature of the arsenian pyrite in this study could not be determined by SEM/EDS or microprobe analysis. Ongoing work beyond the scope of this study is utilizing laser ablation inductively coupled plasma mass spectroscopy (LA-ICPMS) and secondary ion mass spectroscopy (SIMS) analysis in order to associate the gold with specific arsenian events. Pyrite rims on pyrite or arsenopyrite may also appear to be arsenian rims under cursory reflected light analysis when they are extremely narrow, requiring SEM/EDS analysis for definitive identification (Fig. 56).
Some rims displayed an oscillatory or multiple generation sequence of two to five arsenian mineralization events (Fig. 57). These multiple rims may locally neck down to fewer or possibly narrower, and therefore individually indistinguishable, oscillations (Fig. 58) or one generation may appear to suddenly fade out. Single generation or very narrow rims generally possessed relatively constant arsenic values across their full width. More commonly, many oscillatory rims displayed gradational arsenic values that generally decrease outward. Rare instances of arsenic values increasing outward have been noted occurring in conjunction with the more common outward decreasing rims.
Figure 55. Pyrite with arsenian core (a), weaker arsenian outer core (b), non arsenian pyrite zone (c), inner rim of arsenopyrite and outer rim of arsenian pyrite. Bright inclusions are sphalerite.

Figure 56. Arsenopyrite overgrowth on arsenopyrite. Reflected light image in Figure 10. Note epitaxial texture of overgrowth.

Locally, rims may be covered or obscured by large post arsenian overgrowths of pyrite and typically displayed very sharp non-diffuse boundaries (Fig. 59). Rims so covered were not visible under reflected light (Fig. 60), but were readily revealed by SEM/BSE imaging (Fig. 58). Rims with thick overgrowths provided an additional impediment to biooxidation due to the large amount of material requiring oxidation prior to encountering the underlying auriferous arsenian pyrite.

Locally, both arsenian and marcasite overgrowths were directly fed by intersecting microveinlets (Fig. 61). Multigenerational rims with a relatively long duration of deposition generally displayed thick outer rim zones that are largely depleted in arsenic (Fig. 62). These outer rim growths may be easily mistaken for post-ore pyrite
overgrowths but are actually a later low-grade arsenian event. Gold is closely associated with arsenic values (Arehart et al, 1993a), and is therefore primarily constrained to the innermost zone of the rim. Consequently, the outer rim growth serves to shield the auriferous zone from oxidation.
Figure 59. Pre-ore euhedral pyrite with two generation arsenian rim overgrown locally by relatively large post-ore pyrite.

Figure 60. Reflected light image of same pyrite sample as seen in the SEM/BSE image shown in Figure 55. Notice the arsenian rim is not visible. This pyrite would normally be classified as barren without microprobe analysis. Field of view is 0.43 mm.

Figure 61. Pre-ore pyrite with arsenian rim overgrown by vein-supplied marcasite.

Figure 62. Euhedral pyrite with gradational arsenian pyrite overgrowth. Outer rim appears to be pyrite overgrowth, but was actually a low arsenic termination of the arsenian event.
Anhedral more intensely corroded pre-ore pyrite commonly displayed rimming and infilling of interior voids by arsenian pyrite (Fig. 63). This type of mineralization is much more resistant to relatively short-term biooxidation methods. Due to the lack of a true three-dimensional perspective, the true depth of mineralization below the exterior rim is speculative. Many pre-ore recrystallized pyrite crystals, generally euhedral to subhedral, were moderately to highly fractured and corroded. Where arsenian mineralization was present, these fractures tended to be lined throughout the crystal (Figs. 64, 65, 66). Due to the fractured and corroded nature of the crystal and the fact that the majority of the arsenian mineralization was contained within the interior of the crystal, this type of mineralized zone is difficult to recognize without SEM/BSE analysis and is also much more resistant to biooxidation. Interior fractures are also more likely to be sealed during the waning stage of mineralization or late-stage silicification.

Many pre-ore euhedral pyrite crystals with arsenian rims displayed preferential corrosion patterns. In some cases, both the arsenian rim and the preore core were locally corroded (Fig. 67). Generally, the core sustained more extensive corrosion than the rim with the rim undermined by the resulting void. Two observations argue for post arsenian corrosion of the pre-ore pyrite. First, the inner perimeter of the voids did not display any arsenian rimming; secondly, the rims must have formed on a substrate which has since been removed. Figure 68 clearly shows preferential corrosion of specific growth zone within the preore pyrite including the zone directly beneath the arsenian rim. Note the near complete removal of the underlying pyrite zone as a series of interconnected spherical voids below the virtually untouched arsenian rim.
Figure 63. Arsenian rim and infill in a moderately eroded pre-ore pyrite. Small bright inclusions are galena, stannite, and monazite-(Ce). Infill type rims require more intense oxidation.

Figure 64. A very large 1.3 mm eroded and fractured pre-ore pyrite with multiple generation arsenian pyrite fracture fill. Fracture fill is more resistant to solution style biooxidation.

Figure 65. Closer view of center top of Figure 64 clearly depicting both erosional, random fracture, and dilational arsenian mineralized structures. Banded wedge at lower left corner is marcasite that is clearly pre-ore.

Figure 66. Closer view of figure 65 showing greater detail of arsenian rims, local zones of late-stage weaker arsenian mineralization where rims appear overgrown, and choke or pinch out points. Bright spot is late stage sphalerite.
Euhedral pre-ore pyrite crystals tended to exhibit a higher susceptibility to post-ore corrosion. The euhedral pre-ore pyrite in Figure 67 is extensively corroded as are specific sections of the arsenian rim. In several locations, the rim remained relatively intact while the underlying pre-ore pyrite had been completely corroded. In both Figures 67 and 68, the outermost arsenic depleted zone of the gradational rim appeared more resistant to corrosion than the pre-ore core. Growth zoning in some pre-ore euhedral pyrite crystals was also outlined by preferential corrosion of specific zones (Fig. 68) indicating probable compositional or structural (mineralogical) variations between individual growth zones. Corrosion was also clearly post-ore as the arsenian rim had to form on an intact substrate.

Arsenic values in the rim are probably not the reason for the decreased susceptibility of the arsenian rims to corrosion. The outer portion of the gradational rim
was also locally more resistant to corrosion despite its depleted arsenic values. The upper part of the image in Figure 68 shows an oblique view of the outer surface of the arsenian rim. The morphology down the outer surface of the rim matches its cross-sectional relief indicating that the irregularities in the outer boundary of the cross-section view are depositional in nature rather than corrosive. SEM/EDS analysis of over 100 rims identified silicon in conjunction with approximately 60% of the rims studied. Quartz values varied among rims and appeared to increase with proximity to the outer boundary of the rim. If the electron beam is allowed to drift outward across the rim, the quartz signature in the EDS spectra increased without any corresponding decrease in the iron, arsenic, or sulfur peaks (Fig. 69). As the beam transitions off of the rim to the matrix, the elemental signature changed very rapidly to that of SiO₂. This suggests that during the waning phase of the mineralizing event, the precipitation of quartz gradually increased resulting in partial silicification of the outer portion of the rim. It is not yet entirely clear what form this silicification takes. Co-precipitation of quartz crystals in defects of voids in the arsenian lattice, silicified micro-layers of quartz, or encapsulation of individual arsenian crystals are some of the possibilities. More than one type of silicification may be represented. Silicification of the outer portion of the rim would serve to inhibit surface oxidation. A fracture or defect in the rim would allow fluid ingress beneath the rim facilitating the corrosion of the non-silicified interior regions of the crystal (Figs. 67, 68). Preliminary microprobe linear analysis (Figs. 70, 71) has suggested two distinct styles of arsenian pyrite silicification in the data collected across multiple arsenian pyrite rims. In the first style (Fig. 70), the silica concentration clearly increased without any
corresponding decrease in iron, arsenic or sulfur prior to transitioning into the quartz matrix.

Figure 69. SEM/EDS elemental spectrum for arsenian rim on pre-ore pyrite. Spectrum is representative of typical silicified arsenian rim. Silicification may serve to inhibit oxidation of the exterior surface of the rim.

This may be due to the initiation of late stage coprecipitation of silica or post arsenian silica infusion into pores in the outer portion of the rim. Figure 71 clearly depicts a second silicification style, with coprecipitation of silica across the entire width of the arsenian rim. Several arsenian rims with the second style have been analyzed to date and appear to display variable degrees of silicification. Silicification of the arsenian pyrite may have implications for metallurgy that relate to both biooxidation and flotation. Locally, certain polygons from Gold Quarry have realized abnormally low flotation recoveries (personal communications with Newmont geology and metallurgy personnel, 2007) that may be related to the silicification of the arsenian pyrite. The coprecipitation of silica with arsenian pyrite may also have major implications, leading to a better understanding of the ore solution geochemistry and environmental conditions during the main stage ore-forming event.
The surface texture of the arsenian rims was commonly very irregular. It may have a linear folded or etched appearance (Fig. 68), a fine-grain crackly surface (Fig. 72), or a splattered texture (Fig. 73). These multiple textures may be due to a variety of reasons including, but not limited to, excessive quartz precipitation concurrent with arsenian pyrite deposition, very rapid arsenian pyrite precipitation, or corrosion prior to or during quartz precipitation. Certain textures may be indicative of a specific silicification style or lack thereof. Ongoing microprobe work beyond the scope of this project is being conducted in an attempt to verify these possible associations.
Figure 71. 100 spot microprobe linear track starting in the arsenian rim and transitioning to the quartz matrix. Notice the coincidence of silicon and arsenic in the rim. This may represent the coprecipitation of silica with the arsenian pyrite.
Base metal sulfides and sulfosalts

The Carlin trend is host to numerous minerals (Appendix VIII). Gold Quarry is also well known for hosting an extensive list of minerals (Appendix IX). Many of these minerals, both abundant and rare, have been recognized during this study. In addition, the present work has documented the presence of some minerals not previously identified at Gold Quarry and one new, previously unknown, Fe-Ni-As sulfide. Many of these minerals are only found as inclusions. Many other sulfide and sulfosalts minerals not included here have since been recognized along with specific structural associations but are beyond the scope of this project and will be discussed in detail in the follow-on Ph.D. dissertation.
Stannite is common as small inclusions. Rare larger masses possibly replacing pyrite with sphalerite overgrowth and galena inclusions (Fig. 74) may be found locally. Tetrahedrite and tennantite are found as inclusions and more commonly as replacement of anhedral pyrite or local zones of interstitial fill bounding pyrite. Figure 75 depicts the complete tetrahedrite-tennantite solid solution series including the argentotennantite mid-member replacing pyrite with minor inclusions of an unnamed Fe-Cu-Zn-Ni sulfide. Locally, tetrahedrite is argentiferous and may be associated with other silver minerals such as an unidentified Cu-Ag-sulfide and rare acanthite where increasingly elevated silver values are encountered.

Figure 74. Stannite core with sphalerite overgrowth. Bright inclusions are galena. Reflected light image figure 45.  

Figure 75. Tetrahedrite-tennantite solid solution series. Tetrahedrite (a), argentotennantite (b), tennantite (c), pyrite (d), unknown Fe-Cu-Zn-Ni sulfide (dark spot) (e). Pyrite is not visible due to contrast settings necessary to establish solid solution boundaries.  

Chalcopyrite is rare in general but occurred most commonly as a pyrite replacement or in conjunction with the tetrahedrite-tennantite series. Both the
chalcopyrite and the above mentioned Cu-Ag sulfide minerals generally display a paragenetically late relationship with tetrahedrite-tennantite. Sphalerite is common as small inclusions commonly associated with anhedral pyrite, marcasite, and sulfide veins. Rare sphalerite containing numerous extremely fine-grain disseminated arsenopyrite crystals is found partially replacing pre-ore pyrite and overgrown by later stage low-iron sphalerite (Fig. 76). Sphalerite iron concentrations range from extremely high (marmatite) values to only trace levels where it is optically nearly clear.

Figure 76. Arsenian sphalerite replacing preore pyrite. Overgrowth at lower left and outlying bright areas are non-arsenian sphalerite.

Locally, rare tiemannite (mercury selenide) occurred as intermittent rims (Fig. 77) or apparent inclusions in pre-ore pyrite. Further microprobe analysis of the rims will be required to understand their morphology, but the inclusions appear to be thin surface coatings in pre-established voids and therefore late stage. Ongoing work beyond the scope of this study has identified other mercury minerals within the Gold Quarry deposit.

Figure 77. Preore pyrite with rare tiemannite rim. Tiemannite also occurs as thin surface coatings in eroded vugs in pyrite. Dark area contains quartz saturated pyrite.
Samples from two different lithologies contained multiple examples of a previously unrecognized Fe-Ni-As sulfide (Fig. 78) as partial rims as well as inclusions in euhedral and anhedral pyrite, arsenian rims, and vein marcasite. Small euhedral crystals have also been located (Fig. 79). SEM/EDS spectra (Fig. 80) and microprobe-derived stoichiometry are consistent among multiple samples from both lithologies. Larger quantities of the mineral have been found in core during follow-on study and will be used to establish the crystal structure and unit cell parameters by X-ray diffraction (XRD). Current speculation is that it may possibly be an altogether new mineral or that it is a new member of the gersdorffite family. Several other sulfides, including a previously unnamed sulfide, may also have been recognized but require future microprobe analysis for identification.

Figure 78. Anhedral pyrite with a bright partial rim and inclusions of an unknown possibly new, Fe-Ni-As sulfide mineral. Intermediate colored blotches are arsenian pyrite.

Figure 79. Close-up view of euhedral unknown sulfide from upper left of Figure 78.
Several sulfides and other minerals, both rare and common, occur as micro-inclusions in various sulfide hosts, primarily pyrite, and may prove to be valuable marker minerals or mineral assemblages for ore control and exploration. Common mineral inclusions include galena, sphalerite, stannite, monazite-(Ce), tetrahedrite, and tennantite. Rare minerals include argentotennantite, tiemannite, bornite, covellite, greenockite, and probable brannerite and ekanite. Orpiment, realgar, and stibnite are noticeably rare. By far, the most abundant inclusion mineral is anatase. Anatase occurs as small, generally less than 10 - 15 µm long, anhedral inclusions in preore pyrite and marcasite, in association with arsenian pyrite, in local semi-agglomerated masses containing fragmental pyrite, in polymetallic sulfide veins, and disseminated widely throughout most of the silicate matrix.

Other local textures of interest include rare epitaxial overgrowths of arsenopyrite on pre-ore pyrite (Fig. 71) and small local groupings of anastomosing pyrite microveinlets (Fig. 82) which rarely exceeded 5 mm in total length. Low-contrast
SEM/BSE images of the arsenopyrite overgrowths clearly depict compositional zoning suggesting a possible decrease in arsenic values within the outer portion of the overgrowth (Figs. 83A, B). The arsenopyrite overgrowths also locally display a close paragenetic relationship with arsenian pyrite which may indicate the possibility for locally elevated arsenopyrite-hosted gold values. Each texture occurs rarely in specific lithologies and may serve as marker textures.

Figure 81. Pre-ore pyrite center with pyrite overgrowth and later epitaxial arsenopyrite overgrowths. Arsenian pyrite rims both pyrite and arsenopyrite.

Figure 82. Anastomosing pyrite microveinlets. Total local extent of the microveinlets rarely exceeded 5 mm.
Conclusions

Many complex factors affect the efficiency and overall effectiveness of the heap biooxidation process. It is clear, however that sulfate formation deep in the heap plays a very significant negative role in the process. High temperatures combined with biosolution in direct contact with a high-volume air flow initially resulted in an optimal oxidation environment. These conditions, however, also resulted in supersaturation of the local biosolution over time and the commensurate precipitation of excessive quantities of sulfate. This led to sulfate clogging, as illustrated by column 3, or to channelization, as occurred in column 2. Both columns exhibited elevated oxidation efficiency relative to the other columns operated under the same relative parameters, but were also constrained by their relatively small physical parameters. In the larger context of an 800,000 ton heap, the narrow physical constraints generated in the columns, specifically columns 2 and 3, will translate into a far larger volume of the surrounding ore that will be shielded.
from efficient oxidation due to sulfate clogging or channelization associated with a specific air injection port or group of ports. These effects will tend to self propagate through the heap from the origin. Humidification of the induced air will help mitigate this problem to some extent by minimizing fluid and air flow channelization, and thereby the formation of locally anoxic conditions.

The temperature in the core area of the heap periodically exceeded 87° C. This is well above the maximum temperature survivable by most bacteria in solution and may also be above the upper limits for some of the archaea strains present. Uniquely adapted strains from other locations such as the Iron Mountain mine or other historic mines with large unremediated dumps with high sulfide content, may have more extreme temperature and pH tolerances. These microbes, if introduced to the biooxidation process at Gold Quarry, may survive in the more extreme regions of the heap thereby mitigating the locally negative effects of periodic anoxic trending conditions.

Increased temperature tolerances would also allow for more flexible heap temperature management through solution pumping resulting in a more stable internal environment over the life of the heap. This would also serve to minimize the large peripheral temperature fluctuations and therefore increase biooxidation efficiency by promoting more stable microbial populations. A probable negative effect of increased average heap temperature over time is a higher convection rate that would result in an increase in the deposition of sulfate and commensurate thickening of the cement-like cap covering the heap. A higher convection rate would also serve to increase the supersaturation of the biosolution deeper within the heap, leading to increased sulfate formation and clogging. Improved biosolution management through metal cation and
sulfate removal prior to irrigation would help serve to mitigate this issue. Sulfate precipitation through either dehydration, the method that occurs naturally within the heap, or by increasing the solution pH to approximately pH 4 through dilution with fresh water would accomplish this. Increasing pH would also require the later addition of sulfuric acid to restore the biooxidation pH to approximately pH 2. Dehydration would only require the addition of deoxygenated fresh water to increase the solution pH to 2. The original pilot plant configuration utilized increasing the pH resulting in the precipitation of iron oxides. This method did result in increased heap oxidation efficiency, but did not address the elevated aluminum issue. Iron oxides precipitate preferentially to aluminum oxides as pH is increased resulting in final solution iron concentrations below optimum levels for biooxidation from this process.

The formation of excessive sulfate results in the formation of preferred fluid and air channels within the heap. This serves to form numerous local zones of ore that is not exposed to solution flow resulting in dry unoxidized ore or zone of anoxic solution which also precludes ore oxidation. The formation of the sulfate cap over the heap increases in thickness with time and also tends to localize both fluid and air flow to surface fractures. The sulfate coating of aggregate within the heap greatly reduces fluid flow to the aggregate surface resulting in reduced diffusion of oxygen to the microbes and iron cations away from the aggregate surface slowing oxidation. Improved biosolution iron and aluminum concentration management is necessary to minimize these deleterious sulfate effects.

Lithology, specifically as related to permeability and degree of silicification, is of great importance in determining the amount of overall sulfide oxidation of an individual
aggregate sample. Decreased permeability or increased silicification resulted in decreased depth of oxidation below the aggregate surface. Given certain lithologic parameters, oxidizing fluids may only penetrate those sulfides directly exposed at the surface of the aggregate. The average sulfide size rarely exceeded 1 mm, which may result in a very narrow oxidation halo. Petrographic examination of thin sections from biooxidized samples commonly display little or no oxidation below the aggregate surface. The most porous and least silicified samples collected rarely displayed oxidation deeper than 2 to 3 mm below the aggregate surface. Diffusion below the aggregate surface is constrained by aggregate permeability and fracture or pore size. Intense silicification associated with much of Gold Quarry ore extremely fine-grain resulting in minimal diffusion potential. Finer tertiary crushing of highly silicified ore and mixing with coarser, less silicified ore would greatly increase the surface area of the silicified ore and result in increased sulfide oxidation. Locally, unsilicified finely-laminated aggregate exposed on the surface of the heap showed intense delamination due to sulfate wedging, which was not observed within the heap. The unscreened crushing process employed at Gold Quarry dictates a nominal size parameter of one inch minus. Depending upon lithology, this process also realized a common bypass fraction of an estimated one to five percent (visually estimated during lab column loading in 2006) that meets or rarely exceeds three inches in length, allowing for significant subsurface unoxidized potential.

Certain aspects of ore mineralogy and petrology may also play an important role in the biooxidation equation. A through understanding of sulfide mineral paragenesis and corresponding gold mineralizing events is important in determining the optimum duration of the biooxidation cycle for a specific lithologic host and its associated ore mineral
assemblage. Multiple arsenian mineralization events may not all coincide with the mineralizing event and therefore may require more or less complete oxidation than is currently perceived. The coprecipitation of silica with arsenian pyrite may also adversely affect biooxidation efficiency as well as flotation recoveries. Increasing silicification in conjunction with waning ore mineralization may also cause a certain degree of late-stage silica encapsulation thereby locally inhibiting biooxidation.

The biooxidation project was originally initiated to economically process low-grade ore and provide supplementary ore feed to the mill CIL circuit. Biooxidized ore was also used to optimize the ore blend fed to the roaster. The roaster at Gold Quarry is a single stage system that was designed to process typical ore specifically from the Gold Quarry deposit. As Newmont has developed other deposits further north on the Carlin trend that are processed at the Gold Quarry roaster, the ore blending requirements for optimal roaster operation have become more complicated. These deposits such as Leeville or Pete contain very different ore mineralogy’s from Gold Quarry. The biooxidation ore was also used to blend ore from other deposits for the roaster. The termination of the biooxidation process has resulted in further metallurgical complications related to roaster operation.

Future Work

Microbial RNA/DNA analysis: The microbial consortium utilized at Gold Quarry was originally established approximately ten years ago. Adaptation of the population to the specific environments of the heaps has probably occurred during that time. RNA/DNA sequencing will verify the presence of the original microbial species as well
as the presence of any other previously unrecognized microbial populations. This work will be conducted as part of the ongoing study.

One objective is development of a complete and accurate sulfide and sulfosalt mineralogical model of the Gold Quarry deposit with specific emphasis on mineralogical, structural, lithological and alteration associations. The inclusion of trace element and isotope data and, where appropriate, unique crystal textures, will help to further refine these data. This model may then be used to paragenetically constrain relative mineralization episodes related to specific structures or fluid pathways. Mineralogical or elemental variations with distance from source may also be useful in deriving exploration vectors and defining the evolution of the structural plumbing of the system. This type of information will have direct relevance for exploration as well as the more accurate defining of the system genesis.

Certain aspects of arsenian pyrite mineralogy/petrology may also play an important role in determining biooxidation or flotation recoveries. Multiple arsenian mineralization events have been documented (Arehart et al., 1993a; Sha, 1993; Newmont proprietary reports; this study) that may not all coincide with an auriferous event and therefore may not contain significant gold values. Locally, silicification of the arsenian pyrite rims (this study) may have a negative effect on biooxidation efficiency and may also sufficiently alter specific surface properties as to preclude effective flotation. Flotation recoveries vary widely with some polygons reported at below forty percent (Becker, personal communication). This may be due, at least in part, to silicification of the arsenian pyrite or to the majority of the gold being contained in an excessively fine-grain disseminated arsenian pyrite. The silicification, or lack thereof, of the fine-grain
disseminated arsenian pyrite has not yet been tested. The fine-grain arsenian pyrite was also more susceptible to silica encapsulation.

Multiple arsenic events have been identified in rims and by the presence of other arsenic-rich minerals. While gold is generally associated with arsenic in Carlin-style systems, not all of the arsenic mineralizing events were auriferous. Gold values in the arsenian phases at Gold Quarry are below the detection limits for SEM/EDS and microprobe analysis. Laser ablation ICP-MS (LA-ICPMS) and secondary ion mass spectroscopy (SIMS) will be utilized to verify the gold/arsenic correlation, as well as the barren arsenian phases. This information will be important in determining the degree of oxidation required to liberate the gold from specific ore mineralogy, lithology and alteration styles, and for establishing gold mineralization paragenesis.

Several minerals such as lead-arsenic, lead-antimony, lead-arsenic-antimony and bismuth-antimony sulfides and sulfosalts have yet to be identified. Further microprobe work will be employed for that purpose. More in-depth microprobe analysis of the arsenian pyrite silicification will also be carried out. This is of importance to metallurgy as well as the improved understanding of the system geochemistry.
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List of Appendixes

Appendix I  Possible sulfate minerals................................................................................136
Appendix II  XRD spectrum.................................................................................................138
Appendix III SEM/EDS spectrum..........................................................................................143
Appendix IV Column drain and reservoir 21 element ICP graphs.........................................147
Appendix V  Comparative single element histograms.........................................................156
Appendix VI  Ferrous/ferric iron ratio graphs...........................................................................161
Appendix VII Microbial viability data..................................................................................164
Appendix VIII Documented Carlin trend minerals..............................................................167
Appendix IX  Documented Gold Quarry minerals.................................................................172
Appendix I

List of possible secondary sulfate minerals and the ideal chemical formulas. 
(From Hammarstrom et al., 2005)

Not all minerals listed are present in the Gold Quarry biooxidation heaps. Many sulfate minerals may be present in such small quantity or intricately intergrown with other sulfates that they may not be readily detectable. Several of the sulfate minerals have been shown to precipitate only under very specific environmental conditions. Other unique environments undoubtedly exist within the heap and the column sets that were not recognized or sampled.
<table>
<thead>
<tr>
<th>Sulfate mineral name</th>
<th>Chemical formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminite</td>
<td>Al$_2$(SO$_4$)(OH)$_4$ • 7H$_2$O</td>
</tr>
<tr>
<td>Alunogen (s)</td>
<td>Al$_2$(SO$_4$)$_3$ • 17H$_2$O</td>
</tr>
<tr>
<td>Angleite</td>
<td>PbSO$_4$</td>
</tr>
<tr>
<td>Antlerite (i)</td>
<td>Cu$_3$SO$_4$(OH)$_4$</td>
</tr>
<tr>
<td>Apjohnite</td>
<td>(Mn,Mg)Al$_2$(SO$_4$)$_4$• 22H$_2$O</td>
</tr>
<tr>
<td>Basaluminite</td>
<td>Al$_4$(SO$<em>4$)(OH)$</em>{10}$ • 4H$_2$O</td>
</tr>
<tr>
<td>Bianchite (s)</td>
<td>(Zn,Fe$^{2+}$)(SO$_4$)• 6H$_2$O</td>
</tr>
<tr>
<td>Botryogen</td>
<td>Mg,Fe$^{3+}$(SO$_4$)$_2$(OH) • 7H$_2$O</td>
</tr>
<tr>
<td>Brochantite (i)</td>
<td>Cu$_4$(SO$_4$)(OH)$_6$</td>
</tr>
<tr>
<td>Chalcantinite (s)</td>
<td>CuSO$_4$• 5H$_2$O</td>
</tr>
<tr>
<td>Copiapite (s)</td>
<td>Fe$^{2+}$Fe$^{3+}$-4(SO$_4$)$_6$(OH)$_2$ • 20H$_2$O</td>
</tr>
<tr>
<td>Coskrenite</td>
<td>(Se,Nd,La)$_2$(SO$_4$)$_2$(C$_2$O$_4$) • 8H$_2$O</td>
</tr>
<tr>
<td>Destinezite</td>
<td>Fe$_2$(PO$_4$)(SO$_4$)(OH) • 6H$_2$O</td>
</tr>
<tr>
<td>Dietrichite (s)</td>
<td>(Zn,Fe$^{2+}$·Mn)Al$_2$(SO$_4$)$_4$ • 22H$_2$O</td>
</tr>
<tr>
<td>Epsomite (s)</td>
<td>MgSO$_4$• 7H$_2$O</td>
</tr>
<tr>
<td>Ferricopiapite (s)</td>
<td>Fe$^{2+}$Fe$^{3+}$-2/3Fe$^{3+}$SO$_4$• 6H$_2$O</td>
</tr>
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<td>Ferrohexahydrite (s)</td>
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</tr>
<tr>
<td>Fibroferrite</td>
<td>Fe$^{3+}$(SO$_4$)(OH) • 5H$_2$O</td>
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<tr>
<td>Goslarite (s)</td>
<td>ZnSO$_4$• 7H$_2$O</td>
</tr>
<tr>
<td>Gunningite (s)</td>
<td>ZnSO$_4$• H$_2$O</td>
</tr>
<tr>
<td>Gypsum (i)</td>
<td>CaSO$_4$• 2H$_2$O</td>
</tr>
<tr>
<td>Hexahydrite (s)</td>
<td>MgSO$_4$• 6H$_2$O</td>
</tr>
<tr>
<td>Halotrichite (s)</td>
<td>FeAl$_2$(SO$_4$)$_4$ • 22H$_2$O</td>
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<td>Jarosite (i)</td>
<td>KFe$^{3+}$_3(SO$_4$)(OH)$_6$</td>
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<td>Jurbanite (s)</td>
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<td>Linarite (i)</td>
<td>PbCu$^{2+}$(SO$_4$)(OH)$_2$</td>
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<td>Levinsonite</td>
<td>(Y,Nd,Ce)Al(SO$_4$)$_2$(C$_2$O$_4$) • 12H$_2$O</td>
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<td>Magnesiocopiapite (s)</td>
<td>MgFe$^{3+}$-4(SO$_4$)$_6$(OH)$_2$ • 20H$_2$O</td>
</tr>
<tr>
<td>Melanterite (s)</td>
<td>FeSO$_4$• 7H$_2$O</td>
</tr>
<tr>
<td>Paracoquimbite (s)</td>
<td>Fe$^{3+}$_3(SO$_4$) • 9H$_2$O</td>
</tr>
<tr>
<td>Pentahydrate (s)</td>
<td>MgSO$_4$• 5H$_2$O</td>
</tr>
<tr>
<td>Pickeringite (s)</td>
<td>MgAl$_2$(SO$_4$)$_4$ • 22H$_2$O</td>
</tr>
<tr>
<td>Rhomboclase</td>
<td>(H$_2$O)Fe$^{3+}$(SO$_4$)$_2$ • 3H$_2$O</td>
</tr>
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<td>Rozenite (s)</td>
<td>FeSO$_4$• 4H$_2$O</td>
</tr>
<tr>
<td>Schwertmannite (i)</td>
<td>Fe$^{3+}$-16O$<em>{10}$(OH)$</em>{12}$(SO$_4$)$_2$</td>
</tr>
<tr>
<td>Serpierite (i)</td>
<td>Ca(Cu,Zn)$_4$(SO$_4$)$_2$(OH)$_6$ • 3H$_2$O</td>
</tr>
<tr>
<td>Siserotil (s)</td>
<td>Fe$^{2+}$SO$_4$ • 5H$_2$O</td>
</tr>
<tr>
<td>Slavikite</td>
<td>NaMg$_2$Fe$^{3+}$_5(SO$_4$)$_7$(OH)$_6$ • 33H$_2$O</td>
</tr>
<tr>
<td>Starkeyite</td>
<td>MgSO$_4$• 4H$_2$O</td>
</tr>
<tr>
<td>Szomolnokite (s)</td>
<td>Fe$^{2+}$SO$_4$ • H$_2$O</td>
</tr>
<tr>
<td>Tschermigite</td>
<td>(NH)$_4$Al(SO$_4$)$_2$ • 12H$_2$O</td>
</tr>
<tr>
<td>Zugshunstite</td>
<td>(Ce,Nd,La)Al(SO$_4$)$_2$(C$_2$O$_4$) • 12H$_2$O</td>
</tr>
</tbody>
</table>
Appendix II

XRD spectrum for sulfate minerals

The XRD spectrums are representative of the primary sulfate minerals identified. These minerals along with the jarosite-goethite cap, are the primary minerals responsible for the local permeability and channelization issues found within the heap. Note most of the spectrums are of multiple intergrown minerals. A spectrum representative of melanterite is not presented due to the rapid dehydration to rozenite during the time necessary for the analysis. A constant humidity XRD chamber is required for accurate melanterite analysis.
Appendix III

SEM/EDS spectrum for various sulfide, sulfosalt and sulfate minerals

These SEM/EDS spectrum represent some or the more important elemental variations and mineralogical relationships recognized during this study. Of particular interest is the apparent locally occurring coprecipitation of silica with arsenian pyrite. Also of interest is the local representation of the tennantite-tetrahedrite solid solution series. While the arsenic sink in the sulfate accumulations was not found, a vanadium sink was discovered but not identified.
SEM/EDS spectrum depicting silicified arsenian pyrite (top) and non-silicified arsenianpyrite (bottom). Note silicon peak to the left of the sulfur peak.
SEM/EDS spectrum of the tennantite-tetrahedrite solid solution series. Tetrahedrite (top), argentotennantite (center) and tennantite (bottom).
SEM/EDS spectrum of magnesiohalotrichite (top) and the same basic spectrum but with minor vanadium. The magnesiohalotrichite displays a fibrous habit while the vanadium variety displays an interlocking platy habit.
Appendix IV

Excel graphs of 21 element ICP analyses for the laboratory columns

The graphs are specific to both column drain effluent and reservoir solution. Four graphs were constructed for each sample source based on relative element concentrations. These were divided into major elements with concentrations of tens of thousands and thousands of mg/L, major trace element concentrations of hundreds of mg/L and minor trace element concentrations of tens of mg/L.
Appendix V

Histograms of elemental concentrations

Histograms of the average elemental concentrations for each of the 21 elements analyzed. The Drain and reservoir ICP results were averaged separately and then averaged together for each column. The graphs provide a quick visual reference of overall relative column biooxidation efficiency. This does not reflect relative column oxidation efficiency at any specific point in time. Column three generally showed the highest oxidation efficiency however, these histograms do not reflect that the vast majority of column three’s oxidation was realized in the first month of the five month experiment. Arsenic and antimony also show unexplained anomalies.
Appendix VI

Excel graphs of ferrous (Fe^{2+}) / ferric (Fe^{3+}) iron ratios

A specific graph was constructed for each column. Effluent was sourced from the column drains. Comparative graphs were constructed for all of the columns, the unheated columns and the heated columns. Unheated column 7 was anomalous and was combined with the heated columns for scale purposes.
Appendix VII

Microbial viability data provided by Newmont labs in Denver Colorado. Temperature indicates the temperature at which the sample was incubated. Multiple temperatures occur where the sample was split and incubated at various temperatures in order to isolate low and medium temperature bacteria and high temperature archaea.

Legend:
TS – Original biosolution supply tank
D – Column drain sample
T – Tank (reservoir) sample
<table>
<thead>
<tr>
<th>DATE</th>
<th>ID #</th>
<th>COUNT 30°C (cells/mL)</th>
<th>COMMENTS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jan. - 06</td>
<td>TS1</td>
<td>10⁴</td>
<td>Sample has both Acidithiobacillus ferrooxidans and Leptospirillum bacteria</td>
</tr>
<tr>
<td></td>
<td>L 1S1</td>
<td>10¹</td>
<td>Acidithiobacillus</td>
</tr>
<tr>
<td></td>
<td>L 2S1</td>
<td>10¹</td>
<td>Sample was observed to have very few Leptospirillum and no Acidithiobacillus</td>
</tr>
<tr>
<td></td>
<td>L 3S1</td>
<td>10¹</td>
<td>Sample was observed to have very few Leptospirillum and no Acidithiobacillus</td>
</tr>
<tr>
<td></td>
<td>L 4S1</td>
<td>10¹</td>
<td>Sample was observed to have very few Leptospirillum and no Acidithiobacillus</td>
</tr>
<tr>
<td></td>
<td>L 5S1</td>
<td>10¹</td>
<td>Sample was observed to have very few Leptospirillum and no Acidithiobacillus</td>
</tr>
<tr>
<td></td>
<td>L 6S1</td>
<td>10¹</td>
<td>Sample was observed to have very few Leptospirillum and no Acidithiobacillus</td>
</tr>
<tr>
<td></td>
<td>L 7S1</td>
<td>10¹</td>
<td>Sample was observed to have very few Leptospirillum and no Acidithiobacillus</td>
</tr>
<tr>
<td></td>
<td>L 8S1</td>
<td>10¹</td>
<td>Acidithiobacillus</td>
</tr>
</tbody>
</table>

| Jan 24-06 | L 1S2 | 10³                   | Sample had few Leptospirillum and some Acidithiobacillus |
|           | L 2S2 | 10³                   | Sample had both Leptospirillum and Acidithiobacillus |
|           | L 7S2 | 10³                   | Sample had both Leptospirillum and Acidithiobacillus |
|           | L 8S2 | 10⁴                   | Sample had few Leptospirillum and some Acidithiobacillus |

| 30⁰/60⁰ C | L 3S2 | 10² / 10¹              | Sample was observed to have very few thermophilic bacteria and very few Archea |
|           | L 3S2 | 10² / 10¹              | Sample was observed to have very few thermophilic bacteria and very few Archea |
|           | L 6S2 | 10² / 10¹              | Sample was observed to have very few thermophilic bacteria and very few Archea |
|           | L 6S2 | 10² / 10¹              | Sample was observed to have very few thermophilic bacteria and very few Archea |

| 30⁰/50⁰ C | L 4S2 | 10² / 10¹              | Sample was observed to have very few mesophilic bacteria and possibly a few thermophilic |
|           | L 4S2 | 10² / 10¹              | Sample was observed to have very few mesophilic bacteria and possibly a few thermophilic |
|           | L 5S2 | 10² / 10¹              | Sample was observed to have very few mesophilic bacteria and possibly a few thermophilic |
|           | L 5S2 | 10² / 10¹              | Sample was observed to have very few mesophilic bacteria and possibly a few thermophilic |

| Mar 15-06 | L1D74 | 10⁴                   | Sample contained many Acidithiobacillus bacteria |
|           | L1R74 | 10⁴                   | Sample contained many Acidithiobacillus bacteria |
|           | L2D74 | 10⁴                   | Sample contained many Acidithiobacillus bacteria |
|           | C2R74 | 10⁴                   | Sample contained many Acidithiobacillus bacteria |
|           | L7D74 | 10⁴                   | Sample contained many Acidithiobacillus bacteria |
|           | L7R74 | 10⁴                   | Sample contained many Acidithiobacillus bacteria |
|           | L8D74 | 10⁴                   | Sample contained many Acidithiobacillus bacteria |
|           | L8R74 | 10⁴                   | Sample contained many Acidithiobacillus bacteria |

| 30⁰/65⁰ C | L3D74 | 10³ / 10²              | Sample was observed to have a few thermophilic bacteria and very few Archea |
|           | L3R74 | 10³ / 10²              | Sample was observed to have a few thermophilic bacteria and very few Archea |
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*Note: The numerical values represent bacterial counts.*
Appendix VIII

List of Carlin trend documented minerals
(From Ferdock, G. C., 2004)

This list comprises all documented minerals for the Carlin trend. Not all of the minerals listed have been documented at Gold Quarry to date. The continuation of this study has identified other minerals from this list at Gold Quarry as well as several minerals not previously documented anywhere within the Carlin trend.
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Appendix IX

List of minerals previously documented at Gold Quarry

This list comprises all documented minerals at Gold Quarry. The continuation of this study has identified other minerals not noted on this list at Gold Quarry as well as several minerals not previously documented anywhere within the Carlin trend.
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<td>Augelite</td>
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<td>Vésigniéite</td>
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<tr>
<td>Burangaite</td>
<td>Cacoxenite</td>
<td>Volborthite</td>
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