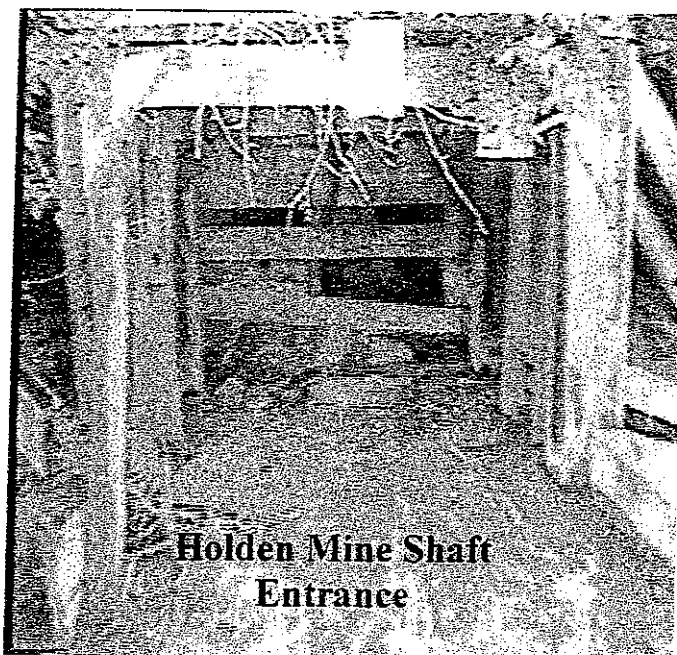


# Identification of Holden Mine Tailing Pile Particulate Matter in Railroad Creek.



By

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## ABSTRACT

Holden mine on Railroad Creek in the Washington Cascades shut down in 1957. As part of Holden Mines legacy to the area several tailing piles were left on the surrounding landscape. The associated environmental impact on Railroad Creek from the acid mine drainage and seepage from the tailing piles can be visually observed in the creek bed. Mine drainage and seeps from the tailing piles are known inputs to the creek of acid wastes and metals. The impact of the tailing pile material on the creek is not as well known.

This study examines the stream sediments to determine if the tailing pile material is still entering the creek. Size and density separation of the sediments followed by microscopical examination of the particulate matter showed the presence of tailing pile particulate matter in the stream sediments. The mine tailing grains are recognizable microscopically by the presence of an orange-yellow "goethite" coating on the mineral grains. Microchemical testing for solubility of the coating in warm hydrochloric acid and heating in a closed capillary help distinguish the coated grains from the naturally yellow colored grains in the sediments. Particulate matter characteristic of the mine tailings was found in all three sediment samples collected below the tailing piles.

### Introduction:

Holden mine is located above Railroad Creek which drains into Lake Chelan. The mine (cover photo) is located on the flank of Copper Mountain. A steady stream of water now issues from the mine shaft entrance and drains into Railroad Creek below. The map, Kilburn et. al., 1994, Figure 1 shows the location of Holden mine. The approximate locations of the sampling stations have been added.

Holden mines began operation in April 1938 and ceased operation in June 1957. During the ensuing 19 years 10 million tons of ore was processed to extract 212 million pounds of copper, 40 million pounds of zinc, 2 million ounces of silver and 600 thousand ounces of gold, (McWilliams, 1958), leaving behind several large tailing piles, Figure 2. The tailing piles dominate the local landscape and extend to the edge of Railroad Creek.

The impact of the mining operation on the creek, almost thirty years after operations ceased, is still visually apparent. Upstream, Station RC-1, the creek has a natural grayish color. Figure 3, whereas adjacent to the mine and downstream of the tailing piles, Station RC-4, the creek -bed becomes a brownish-red color, Figure 4. Near Lucerne, Station RC-3, at the mouth of the creek discoloration is still present in the stream but the stream is starting to regain it's normal color.

Over the years attempts have been made to stabilize the tailing piles by capping them with native soils and planting vegetation along with placing fiber mats on the sides to stabilize the slopes. The unvegatated top of the tailing piles and one of the fiber mats (the darker band on the lower slope of the pile) used to stabilize the pile can be seen in Figure

2. The success of these endeavors has not been determined. Large areas of the tailing piles remain exposed to the elements. Consequently material from the tailing piles is available for both wind and water transport to Railroad Creek and subsequently Lake Chelan.

The purpose of this study is to see if tailing pile material is entering Railroad Creek and is being transported downstream. To accomplish this the tailing pile material and sediments were examined microscopically to determine if tailing pile material is present in the stream sediments.

**Sampling:**

Sampling was done following the Quality Assurance Project Plan (QAPP), Johnson and White, 1996. Three stations were selected. The first one RC-1 was located about one mile upstream from the mine tailings. Station RC-2 was approximately one-half mile downstream from the lower tailing pile. A fourth station was added while in the field. It was labeled RC-4 and was about three miles below the mine tailings. The last station RC-3, was located in the village of Lucerne about a quarter mile from Lake Chelan. The stations and their locations are given in Table 1.

**Table 1. Railroad Creek Sediment Sampling Locations and Dates**

<u>Station ID</u>	<u>Latitude</u>	<u>Longitude</u>	<u>Sampling Date</u>	<u>Laboratory #</u>
RC-1	48° 12.22' N	120° 47.06' W	Sept. 11, 1996	96-378284
RC-2	48° 09.14' N	120° 46.74' W	Sept. 11, 1996	96-378295
RC-4	48° 11.50' N	120° 42.4' W	Sept. 11, 1996	96-378326
RC-3	48° 11.85' N	120° 35.98' W	Sept. 11, 1996	96-378306
TP-1	Tailing pile near Copper Creek		Sept. 11, 1996	96-378321
TP-2	Tailing pile		Sept. 11, 1996	96-378322

Material from the tailing piles was collected at two locations. The samples are composites taken from exposed surface of the tailing piles. Both samples were from the lower tailing pile, Figure 2. Sample 96378321 was collected from the upstream end of the pile near the base and adjacent to Copper Creek. Copper Creek enters Railroad Creek in a gap between the two lower tailing piles. Sample 96378322 was collected from the downstream edge of the pile near Railroad Creek.

Sediment samples, 96378284, 96378295, 96378306 and 96378326 were composite grabs taken from along the stream bed. These were generally taken in backwater areas or behind logs or boulders where fine sediments accumulated.

Historically the stream bed has undergone "cementation" when the fine grain sediments over a period of time take on the characteristics of "concrete". Several pieces of the old

"cemented" stream bed were collected from Railroad Creek near the lower end of the tailing pile, in the same area as tailing pile sample, 96378322

### **Sample Preparation:**

The tailings pile samples were examined under the microscope to determine the upper particle size range. Most of the material observed was under 250 micrometers. A US Standard 60 mesh brass sieve ( 250 micrometers) was used to sieve the tailings pile material. The fraction passing through the sieve was saved for further analysis. Less than 5% of the total material was retained by the sieve. The material greater than 250 micrometers was mostly plant and non-mineral debris,

The sediment samples were prepared for microscopical analysis by removing 10 grams of wet sediment and drying at 105° Centigrade for 24 hours. An aliquot of the sample was dry sieved through a US Standard 60 mesh (250 micrometer) brass sieve. The material passing through the sieve was collected for microscopical examination

### **Analyses:**

#### Density Separations

Density columns were used to help isolate the tailing pile material from the sediments. The density columns were constructed and prepared as described in McCrone and Hudson, 1976. Toluene was substituted for benzene as the light liquid and methylene iodide was used as the heavy liquid. Ethylene glycol was used as the immiscible liquid in the side arm to control the liquid levels in the density column. The columns were mixed using a small bar magnet removed from a micro-stir bar and placed inside the column prior to filling. The density gradient was established by moving the bar magnet up and down the center portion of the column rapidly 20 to 30 times with an external magnet. The column was then left undisturbed for 2-3 hours to allow the density gradient to form.

After the density gradient had stabilized the zones where the mineral grains had settled were noted and their heights recorded, Figures 5 a-to f. Each fraction was collected as it eluted from the column for microscopical analysis, Figure 6 and Figure 7. A small aliquot of the toluene-methylene iodide density solution was also taken so that each layer was bracketed. The refractive index was then measured using a Milton Roy refractometer and compared to a calibration curve of density versus refractive index of known toluene-methylene iodide mixtures.

#### Particle Analysis

Particle size analysis was contracted by Manchester Environmental Laboratory to Soil Technology Inc using Puget Sound Estuary Program (PSEP) gravimetric and pipette sediment analysis methods. The fractional percentages (histograms) and cumulative

percentages (line graph) for Stations RC-1, RC-2, RC-3 and the tailing pile, TP-2 are shown in Figure 8.

### Microscopical Analysis

Microscopical analysis was done using an Olympus, SZ-11 Stereo microscope and a Leitz Dialux petrographic Polarized Light Microscope (PLM). Video images were captured using a Sony DX151 video camera or a Sharp Viewcam and Targa + 16/32 video capture board. Images were processed using Media Cybernetics Image Pro and Adobe Photoshop 3.0 software.

### Discussion:

The microscope was used to see if there was a characteristic or unusual particle in the tailing pile which could act as a tracer in the sediment. The density separations were used to help isolate the "tailing pile" fraction from the bulk of the stream sediment. Final isolation of the "tailing pile" particulate matter was accomplished by manual sorting under the stereo microscope using a tungsten needle.

The tailings pile consists primarily of quartz grains, some feldspars, hematite and occasionally hornblende. Some dense magnetic particles are also present in small quantities but as with the hornblende significantly less is present than in the creek sediments. The tailing material is depleted in the heavy minerals, relative to the RC-1 stream sample. Most all of the particles are coated or partially coated with an orange to orange-yellow coating, Figure 7 and Figure 9. The coating appears to be "goethite" based on its chemical reactions. Treatment with warm hot nitric acid and 30% peroxide removed the coatings and the orange-yellow coloration, Figure 10. Warm hydrochloric acid was also very effective at removing the coating.

The goethite ( $\alpha\text{-Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$ ) coating the tailing pile minerals is an amorphous form and displays little of the crystallographic characteristics of the pure mineral. Goethite is described in "The Particle Atlas", (McCrone et al., 1979), as being a common weathering product of iron bearing minerals. Goethite is often fine grained and may be fibrous or bladed. Upon dehydration goethite yields hematite. Optically it is an orthorhombic mineral and may be opaque. Impure aggregates may be apparently isotropic, Winchell and Winchell, 1989. Very few crystallographic properties are displayed by the "goethite" coatings on the mine tailings and it is principally its chemical reactions which are used here to identify it.

This orange-yellow coating, Figure 9 appears to be the most distinguishable feature and it was used to help identify tailing pile grains from native grains in the samples. The presence of naturally occurring "yellow colored" minerals complicates the observations but generally these are solid colors extending all the way through the mineral grain not just a surface coating. The coating was easily visible using oblique illumination on the

microscope. The coating could also be distinguished from the naturally colored minerals by a solubility test with hydrochloric acid. Grains showing the orange-yellow coating collected from the density separations are shown in Figure 11, RC-1, Figure 12, RC-2, Figure 13, RC-4 and Figure 14, for RC-3

The density separations on the tailing pile samples showed that both samples were very similar in behavior. The bulk of the material ended in a relatively well defined band with a scattering of particles above and below. The density of the band ranged from 3.07 to 3.13 gm/cc. Heavy minerals (>3.3 gm/cc) were relatively infrequent in the tailings pile material, but were relatively common in the stream sediment samples. This probably reflects how the material was handled and treated to separate the ore minerals before deposition in the tailings pile. The iron minerals and hornblende present in the stream sediments were separated by the density column from the lighter tailing pile material.

The density for the tailing pile material was around 3.1 gm/cc. The majority of the mineral grains in the tailings pile were quartz which has a density range of about 2.65 to 2.9. The fact that the mine tailing grains were higher in density than the mineral substrate would warrant is attributable to the higher specific gravity of goethite, 4.28 (crystals) with a range of 3.3 to 4.3 gm/cc, (Winchell and Winchell, 1989). The goethite coating increases the density of the underlying mineral grain. This also helps explain the spread in densities.

The upstream site RC-1 Figure 5 a, and b, failed to show a orange-yellow band in the density column similar to that produced by the tailing pile samples, Figure 5e. The sample collected at RC-2 gave a definite orange-yellow band at a density of about 3.05 to 3.13. This was also true for sample RC-4 Figures 5c and d. The particle characteristics were similar to those of the tailings pile, Figure 9, showing the orange to yellow coatings on the many of the mineral grains in that density band. The orange-yellow band was nearly indistinguishable in RC-3, Figure 5f, although some grains characteristic of the tailing piles were found under the microscope, Figure 14.

The particle size analysis shows that the mine tailings are concentrated in a narrow size range, from 250 micrometers to 31 micrometers. Figure 8 a, b, and c graphically compares the particle size analysis results for the tailing pile material and stations RC-1, RC-2 and RC-3. The particle size data alone fails to distinguish the differences between the stations. The larger material in the creek overwhelms the tailing pile size fraction.

The tailing pile material (96378321) showed a near normal distribution around 250 to 31 micrometers, Figure 8a. Almost 97% was between 62 and 250 micrometers in size. Only one percent was greater than 250 micrometers, (i.e. 250 to 500) and the other two percent was less than 31 micrometers. The corresponding size distribution for RC-1 (96378284) was 8%, Figure 8b, RC-2 (96378295) was 37%, Figure 8c, RC-4 (96378326) was 67% and for RC-3 (96378306) 6%.

The two stations, RC-2 (37%) and RC-4 (67%) below the tailing pile had a significant increase in the 32 to 250 micrometer size range compared to the reference station RC-1 (8%). Station RC-3 (6%) had less in the tailing pile size range than the upstream sample RC-1 but a significantly greater number of the individual particulate grains in that size fraction were comparable to those found in the mine tailings samples.

Although the tailing pile grains could be distinguished under the microscope by the patchy orange-yellow coating on the mineral grains, there were some yellow to orange colored minerals which had similar appearance. These could be quickly distinguished using microchemical tests.

An aliquot of the density fractions, 3.05 to 3.15, collected from the density column separations were placed on a microscope slide and the particulate grains coated with an orange-yellow material were separated out using a stereo microscope and tungsten needle. Only a few grains were found in sample RC-1 whereas 50 to 100 grains were easily separated out from samples RC-2, RC-3 and RC-4. The sample of orange-yellow grains was split into two groups of ten to fifteen grains each for microchemical testing. Grains from the tailing pile sample TP-2 (96378322), were used as a control. One fraction was treated with cold 5N hydrochloric acid. Initially no reaction was observed. When the 5N hydrochloric acid was warmed slightly, the orange-yellow coating on the mineral grains rapidly dissolved. This is illustrated for sample, RC-4 in Figure 15a, cold 5N hydrochloric acid and Figure 15b, after slight warming of the 5N hydrochloric acid.

Another aliquot, from RC-4 was tested for goethite by placing the coated mineral grains in a 2 mm OD capillary tube sealed on one end. The other end was sealed and the end with the grains was placed in a butane microtorch flame for one second. The orange-yellow coating on the mineral grains was changed to a reddish-brown color as the goethite was converted to amorphous  $\text{Fe}_2\text{O}_3$ , top capillary Figure 16a, before heating and 16b after heating. If sample size is large enough water vapor can be observed condensed on the capillary tube walls. The capillary test is also shown for the tailing pile sample, TP-2 (bottom capillary) in Figure 16a, and 16b.

One of the most striking things about Railroad Creek is the change in the stream bed color, Figure 3 above the mine tailings compared to below the mine, Figure 4. Obviously the mine tailings contribute to the coloration of the stream bed but how the tailing material interacts with the creek is not as obvious.

Analysis for total suspended solids (TSS) and dissolved metals resulted in the production of two sets of filters. The glass filters used in the TSS procedure were saved along with the membrane filters used in the field to filter the creek samples for dissolved metals. The filters were arranged from upstream to downstream and visually compared for color, Figure 17, for TSS and Figure 18 for dissolved metals. Both sets of filters showed a marked change in color from the upstream station to the station below the tailing piles, decreasing further downstream.

Examination of the TSS filters and dissolved solids filters, Figures 17 and 18, did not show a large number of goethite coated mineral grains or many other mineral grains either. The particulate matter consisted primarily of yellow to orange yellow flakes, Figure 19. The material appears isotropic and had a refractive index greater than 1.660. The material exhibited the same behavior as the orange-yellow coating on the mine tailing grains. It dissolved rapidly in warm HCl and when heated in a closed capillary tube exhibited a change in color from orange-yellow to reddish-brown. The orange color on the filters is not directly due to an abundance of mine tailing grains suspended in the creek but appears to be the orange-yellow coating from the grains which may have been dislodged from the grains by turbulence. Due to their "flake like" nature they remain suspended and impart an orange color to the filters when concentrated. The darker orange color of the TSS filters is due to their drying at 105° C for 24 hours which may have started converting the goethite to amorphous Fe<sub>2</sub>O<sub>3</sub>.

Scraping off some of the coating from the "cementation" fragment, Figure 20, resulted in orange-yellow flakes similar to those on the filters, Figure 17 and 18. Characteristics under the microscope, isotropic refractive index greater than 1.660 were also similar to the filter particles, Figure 19.

The tailing pile grains are getting in the creek as demonstrated by their presence in the sediments at RC-2, RC-4 and RC-3. Some of the coating is dislodged by stream action and these "flakes" appear to be the material collected on the filters. It may also be important in the cementation process since it is similar to the material coating the cemented fragment of stream bed. The "cemented" stream bed is very similar to concrete. A small fragment was broken off and the cross-section examined, Figure 21. The external orange coating and darker purplish minerals, Figure 22 dissolve in warm hydrochloric acid reacting similarly to goethite. The solubilized coating gave a positive test for iron using potassium ferrocyanide, Chamot and Mason, 1940.

Scanning Electron Microscopy and X-ray microanalysis of the coating on the tailing pile grains Figure 23, Station RC-2, Figure 24 and the orange coating on the "cementation" sample, Figure 25, showed similar chemistries. Elements detected in all three samples included iron, oxygen, aluminum, silicon, and calcium. The coating on the tailing pile grains and Station RC-2 also showed some sodium, potassium and sulfur.

The loss of sodium, potassium and sulfur in the cementation coating makes sense in the aqueous environment of the stream. The "cementation" sample coating also contained small amounts of carbon. The carbon may be due to biological activity on the cemented grains.

The amounts of tailing pile particulate matter in the sediments was estimated by microscopic examination of the 40 to 250 micrometer fraction from each station. From 800 to 1200 grains were counted at each station. The percentages of "tailing pile" grains identified at each station were, RC-1, 0.26%; RC-2, 14.1% and 13.1%; RC-4, 11.7% and RC-3, 5.0%.



## **Conclusions:**

Examination of Railroad Creek sediments indicates the presence of "mine tailing" material in the creek. The closer to the tailing piles the greater the amount present. Upstream of the tailing piles at RC-1, some grains attributable to the tailing pile can be detected but very few (<1%) and they are most likely wind borne. This is possibly a second mode of entry into the stream next to rain events. Down stream of the tailing pile at RC-2 and RC-4 the numbers are much higher (12 to 14%) but decrease to (5%) further downstream at RC-3. The mine tailing grains are recognizable microscopically by the presence of an orange-yellow coating on the mineral grains. The coating reacts slowly with room temperature hydrochloric acid and dissolves rapidly in warm hydrochloric acid. Heating of the grains in a closed capillary causes the grains to change color from the orange-yellow to a brownish-red color.

The investigation of the colors on the TSS and dissolved metals filters showed that the coloring agent was not organic matter or mine tailing particulate matter suspended in the stream but "flakes" of an iron bearing mineral, possibly goethite, based on its similar microchemical tests. A possible source for these flakes is the goethite coating the tailing pile minerals which has come loose as the grains tumble in the current. Having a higher surface to mass ratio the material remains suspended in the stream longer.

Examination of the filters left over from the TSS and dissolved metals determinations also showed very little suspended tailing pile material. The low amounts of tailing pile grains in the water column may be in part due to the dry weather conditions. During heavy rains the amount of material entering the stream from the tailing piles may be significantly greater.

## **Recommendations:**

1. Collect water grab samples during or just after a rain event and check for tailing pile grains
2. Look downstream of the tailing pile for an area of quiescent water, such as a beaver pond or lake where a delta would form from the sediments. Core the sediments and examine the particulate matter deposited to identify mine tailing material. The number of layers showing mine tailing material would give an indication of the amount and frequency of mine tailing transport incidences.
3. Take a core off the mouth of Railroad Creek in Lake Chelan to look for mine tailing material. Do sediment dating to see the historical pattern of mine tailing input.

4 Investigate the mineralogy of the cemented stream bed using thin-sections to determine mineralogy. Compare this to the mineralogical makeup of the mine tailing pile material.

5. Use Scanning Electron Microscopy / Energy Dispersive X-ray analysis to more fully characterize the goethite coating on the mine tailing mineral grains. Also see what other elements are associated with the goethite and the coatings. The coatings on the cemented creek bottom could also be examined as well as the internal mineralogy of the "cemented" sediments.

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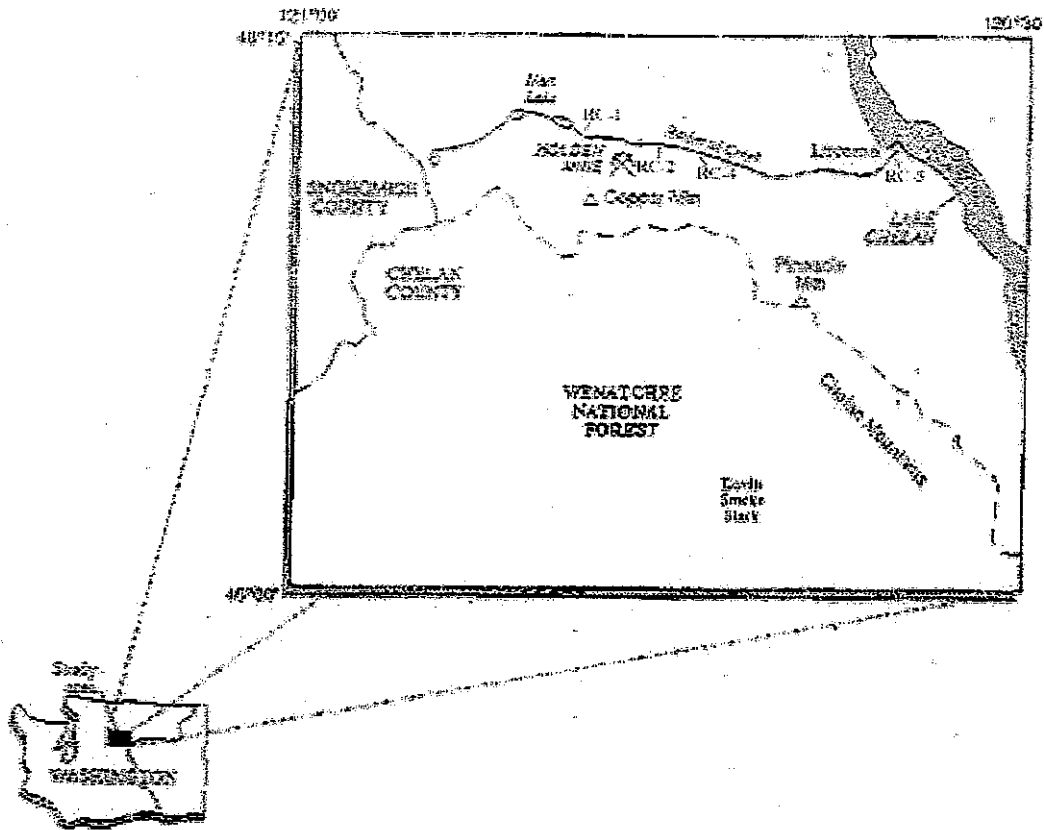
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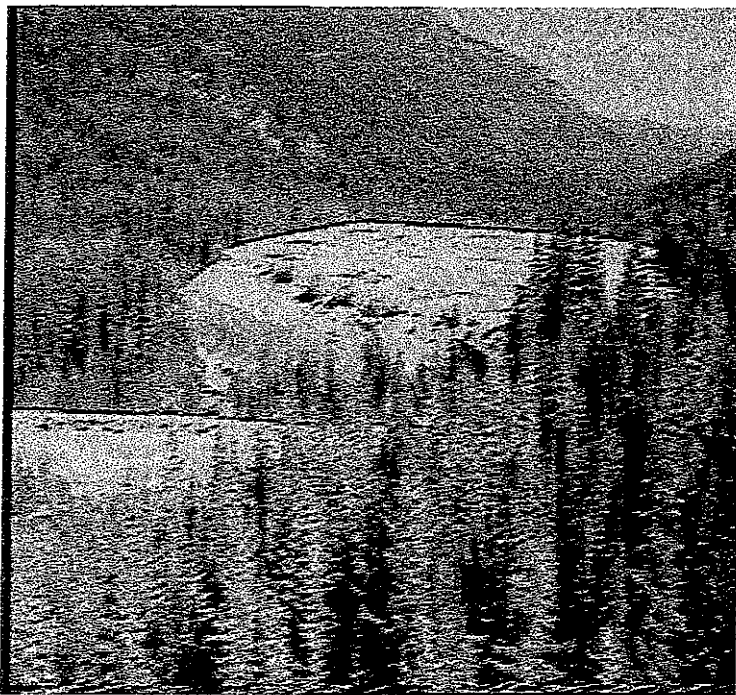
**Acknowledgment:**

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## Figures



**Figure 1. Location of Holden mine and sampling stations.**  
(Kilburn et al., 1994)



**Figure 2. View of lower tailing piles from upper tailing pile near the mine entrance.**

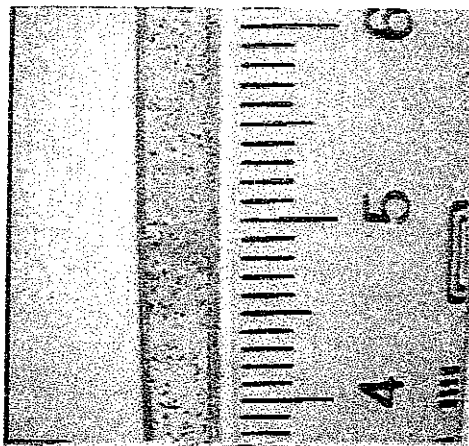


**Figure 3. Railroad Creek at Station RC-1.**

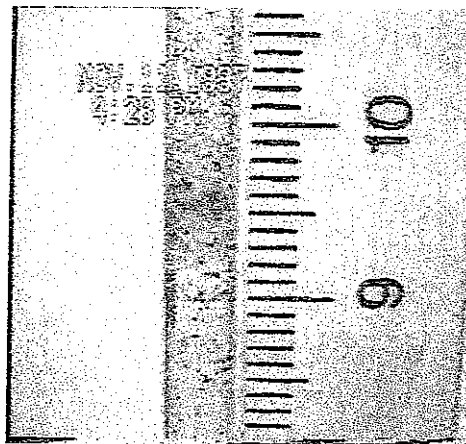


**Figure 4. Railroad Creek at Station RC-4**

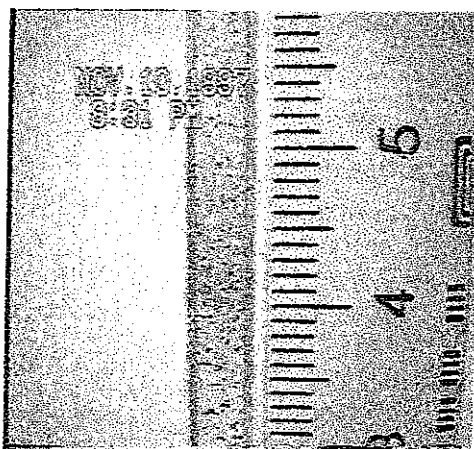
Figure 5 a-f. Density Column separations for Railroad Creek and Tailings Pile. Density range 3.05 to 3.13.



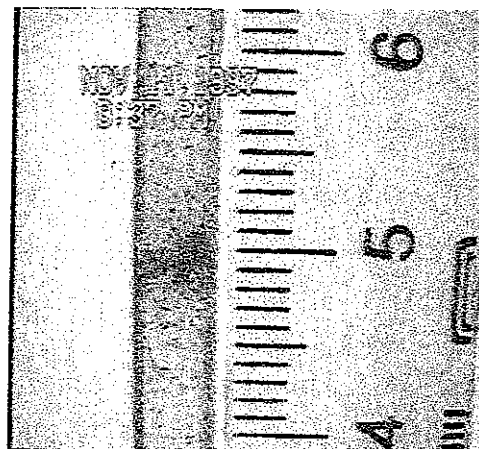
a: Station RC-1 2.4X



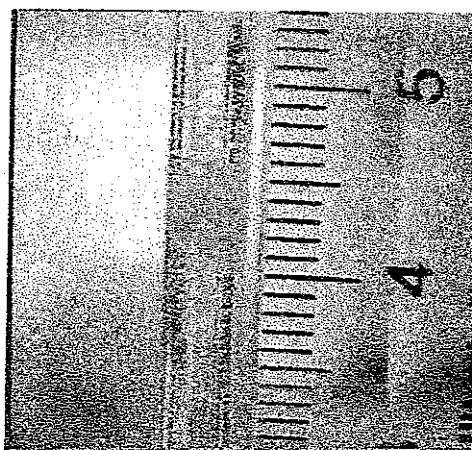
b: Station RC-1 2.3X



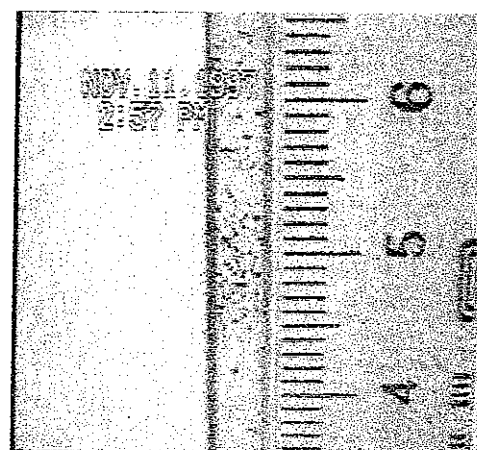
c: Station RC-2 2.1X



d: Station RC-4 2.5X



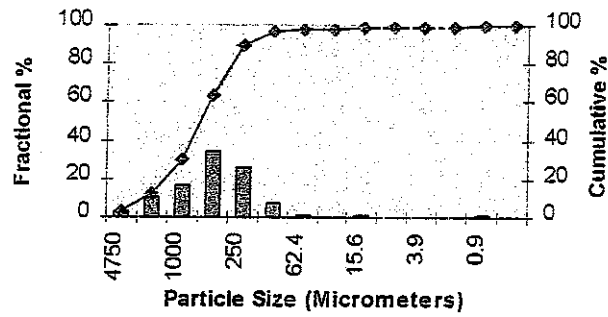
e: Tailing Pile TP-2 2.5X



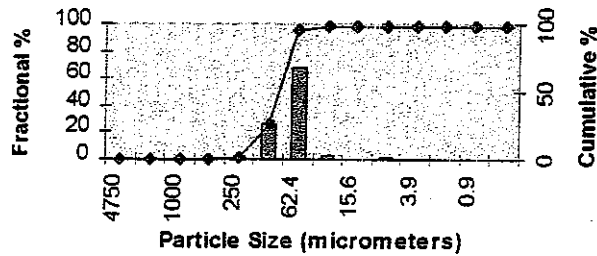
f: Station RC-3 2.0X

Figure 8. Particle Size Distribution for RC-1, Tailing Pile, RC-2 and RC-3

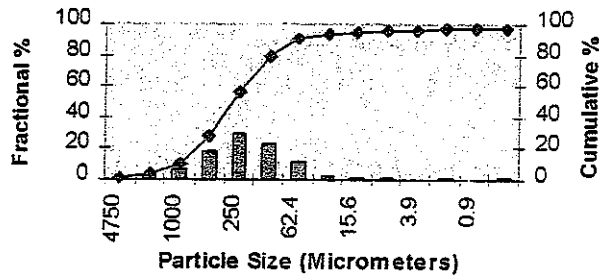
RC-1 Particle Size Distribution



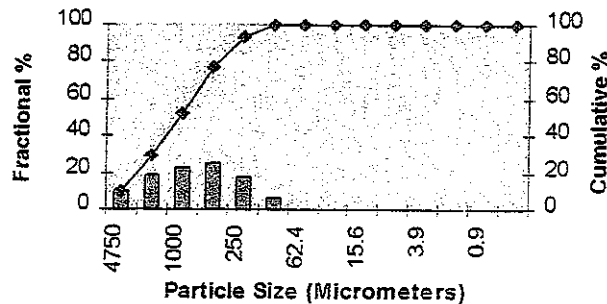
Holden Mines Tailing Pile Particle Size Distribution



RC-2 Particle Size Distribution



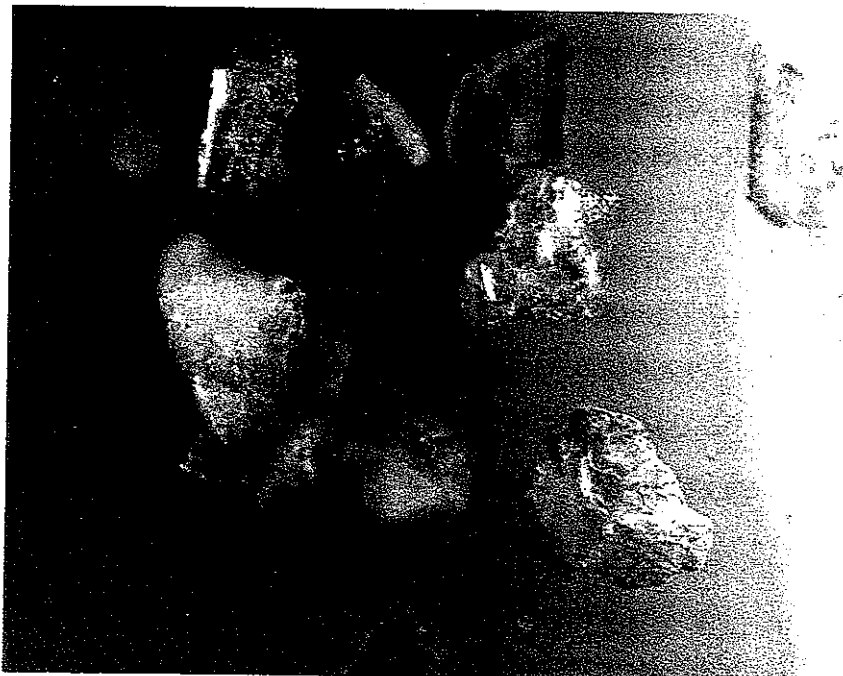
RC-3 Particle Size Distribution



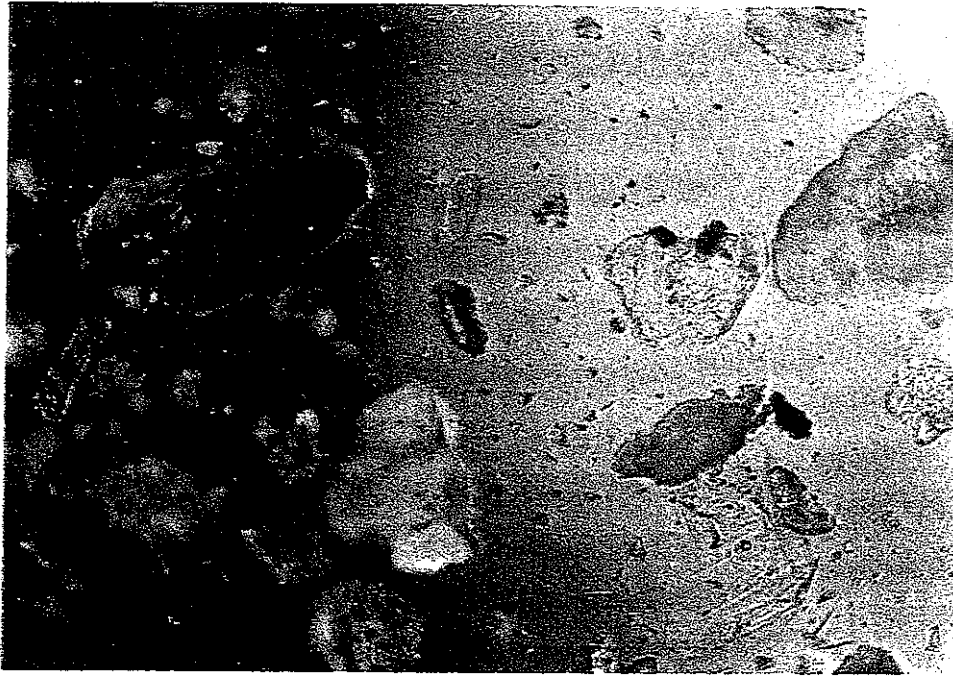




**Figure 9. Tailing pile TP-2, Sample 96378322, 100X oblique light. Note the orange-yellow coating on the clear mineral grains.**



**Figure 10. Tailing pile TP-2, Sample 96378322 after removal of orange-yellow coating with nitric acid. 100X oblique light**



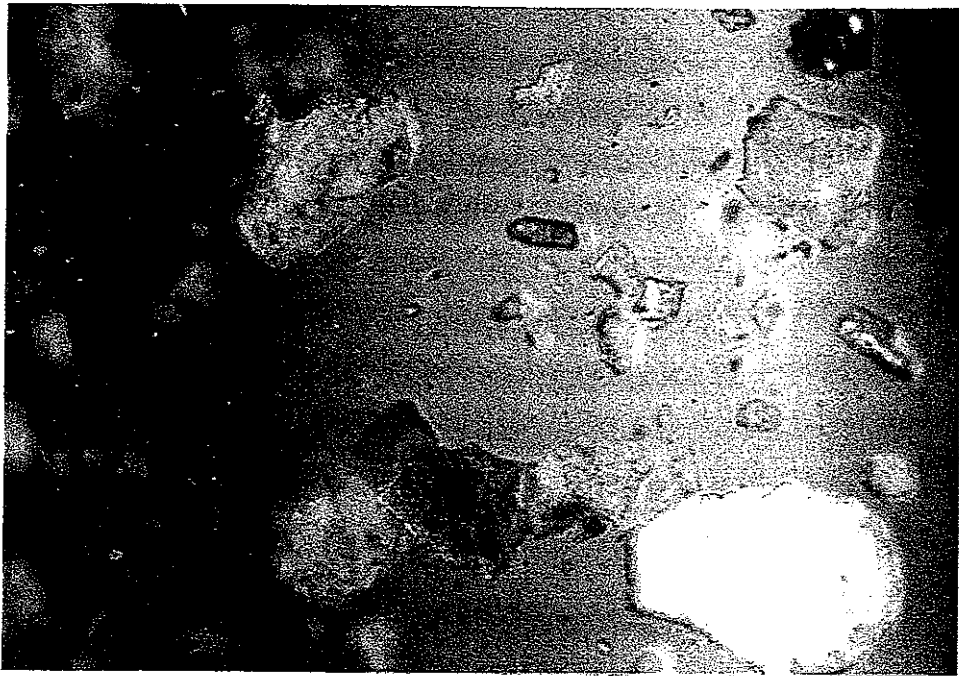
**Figure 11. Sample RC-1, Station 96378284. 100X oblique illumination  
Note solid orange-yellow mineral grains and absence of coated grains.**



**Figure 12. Station RC-2, Sample 96378295, 100X oblique illumination.  
Note orange-yellow coating in transparent mineral grains.**



**Figure 13. Station RC-4, Sample 96378326, 100X Oblique illumination**



**Figure 14. Station RC-3, Sample 96378306 100X oblique illumination**

Figure 15 a, b. Hydrochloric acid test for goethite- Sample RC4 (96378326).  
Dissolution of orange-yellow coating in warm hydrochloric acid. Stereo 50X

a



Before Warming in 5N Hydrochloric Acid

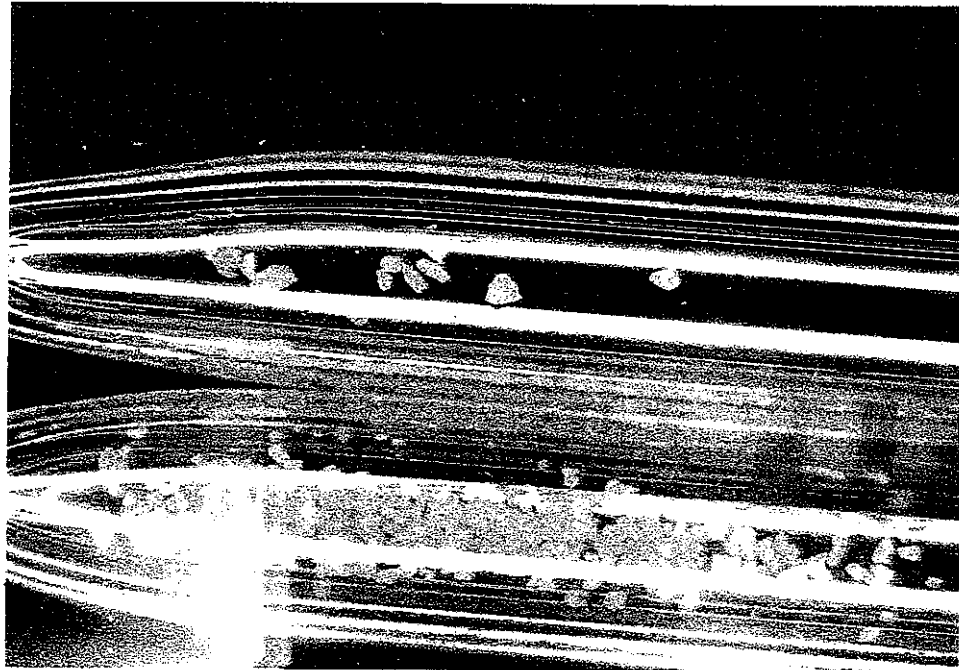
b



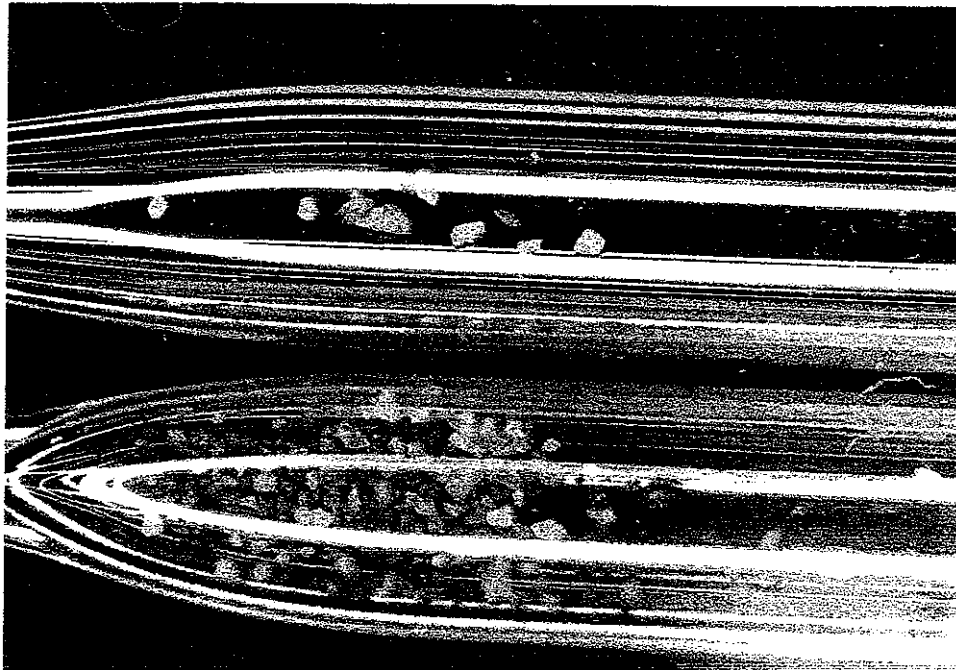
After warming in 5N Hydrochloric Acid. Orange coating dissolved.

Figure 16 a, b. Capillary test for goethite. Station RC-2 (96378395) top. Lower tube is Tailing Pile (96378322). Color change from orange-yellow to reddish-brown (hematite). Stereo 17X.

a



b





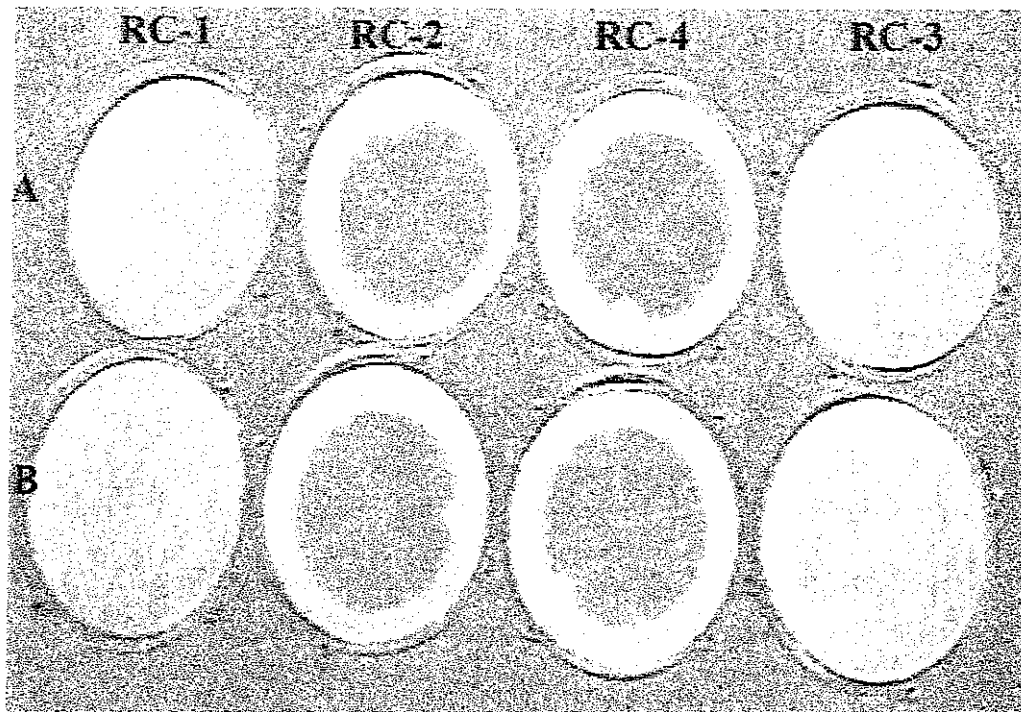


Figure 17. Glass fiber filters from Total Suspended Solids test. Upstream RC-1, RC-2 just below tailing pile, RC-4 3 miles downstream and RC-4 near mouth of Railroad Creek. Magnification 0.8X

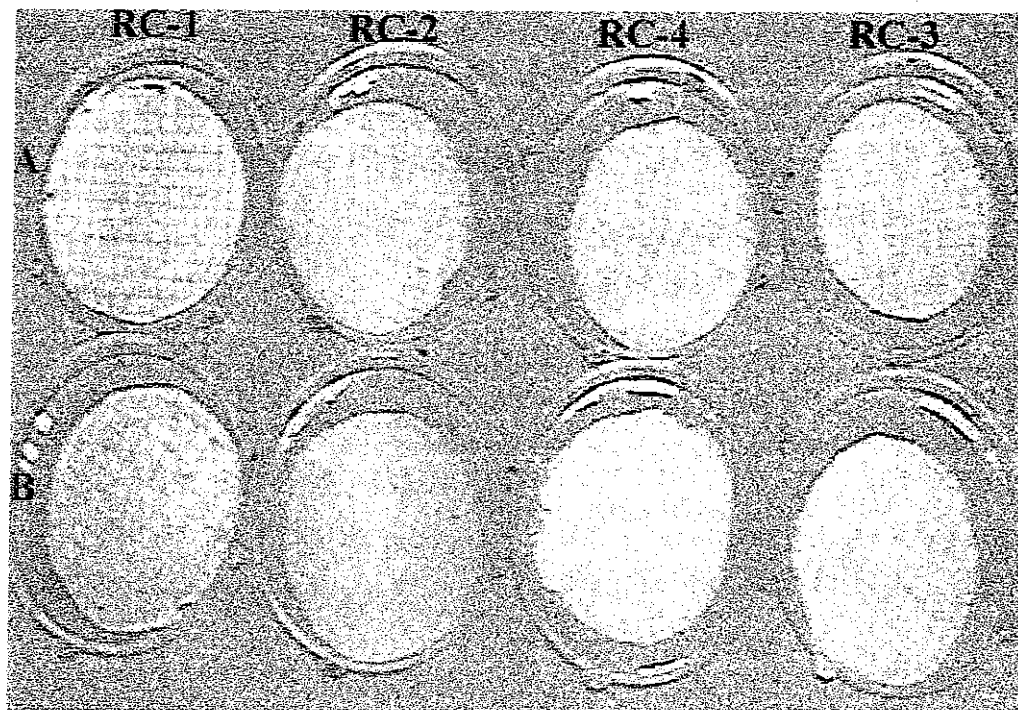
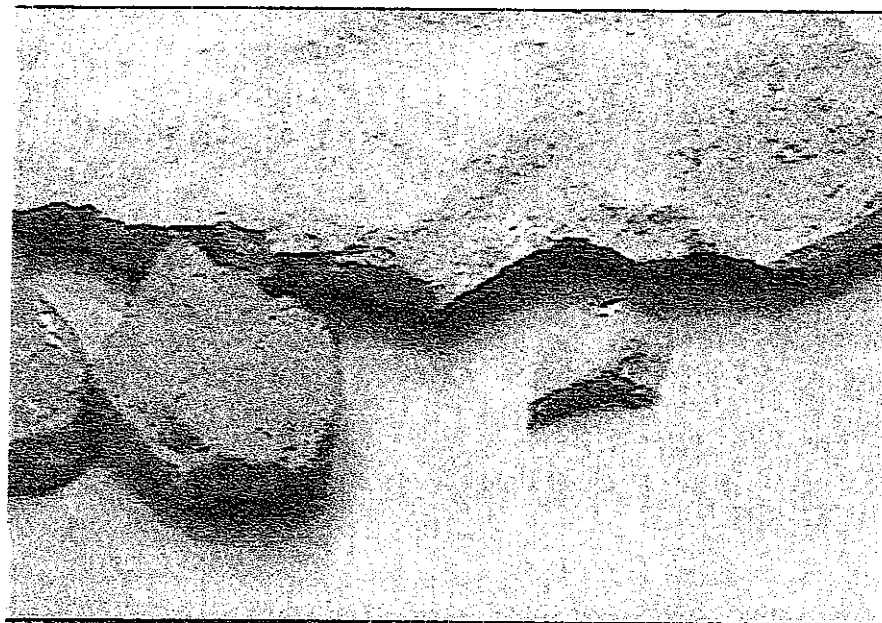


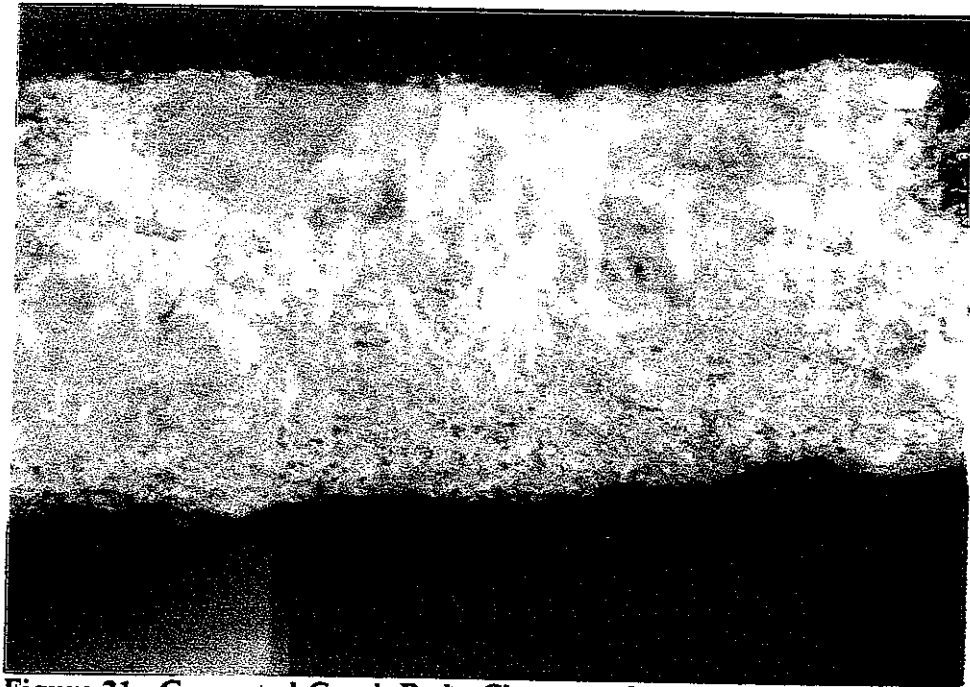
Figure 18. Membrane filters from dissolved metals samples. Upstream RC-1, RC-2 just below tailing pile, RC-4 3 miles downstream and RC-4 near mouth of Railroad Creek. Magnification 0.8X



**Figure 19. Orange-yellow flakes “goethite” and glass fibers from the Total Suspended Solids filter RC-2 (96378292). Transmitted light, 500X.**



**Figure 20. Fragments of cemented creek bed collected from Railroad Creek just below lower tailing pile. Fragment broken off to observe cross-section and sampling. Magnification 1.1X.**



**Figure 21. Cemented Creek Bed - Close up of cross-section from Figure 15. Stereo 35X**



**Figure 22. Cemented Creek Bed - Close up of edge showing mineralization and orange "goethite" coating. Stereo 35X**



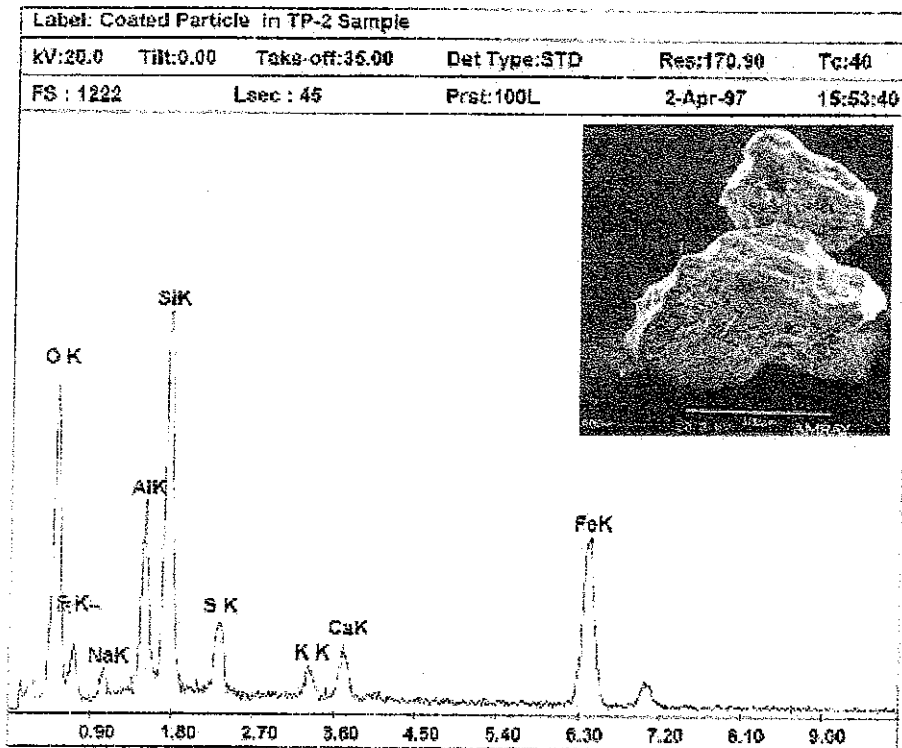


Figure 23. Tailing Pile, TP-2 SEM-X-ray microanalysis of orange coating. White size bar under photo is 100 micrometers long.

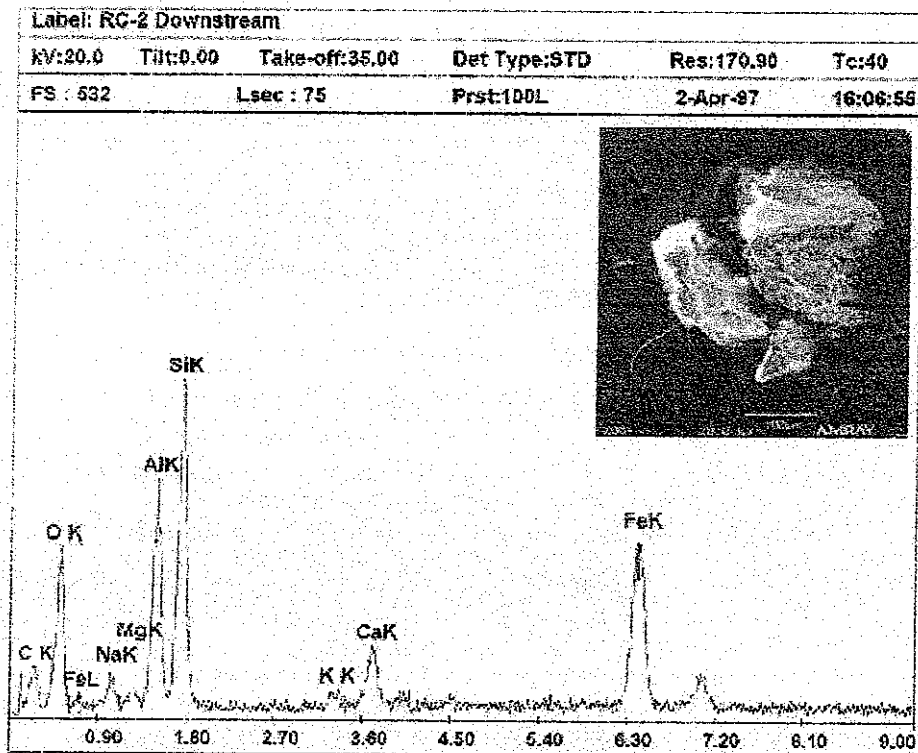
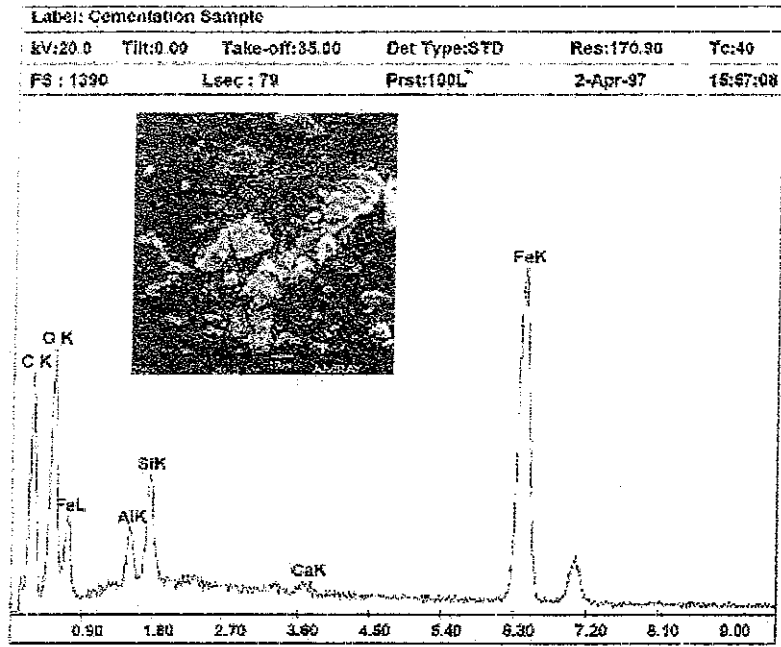


Figure 24. Railroad Creek, RC-2, SEM-X-ray microanalysis of orange coating. White size bar under photo is 100 micrometers long.



**Figure 25. SEM/X-ray microanalysis of Cementation fragment orange coating. White size bar under grains is 10 micrometers**