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Environmental Characterization of Soils and Sediments*

**A Progress Report, Part II*

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ENVIRONMENTAL CHARACTERIZATION OF SOILS AND SEDIMENTS*

A Progress Report, Part II

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ABSTRACT

Elevated chromium level in the ground water at the SNL/CA site is a major concern. We have developed an analytical methodology that allows us to identify the possible sources of the contamination. This methodology is focused on the integration of various analytical techniques to characterize physical and chemical properties of the soil samples from the contaminated area. The techniques include inductively coupled plasma spectroscopy (ICP), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), X-ray diffraction spectroscopy (XRD), X-ray fluorescence spectroscopy (XRF), and electron microscopy with image analysis (EM/IA).

This report describes the overall experimental approaches and findings for the soil samples from the area where the Cr-level was found to be elevated. Current findings suggest that the potential source of the Cr is the Cr-containing minerals that are present in natural abundance rather than externally introduced. In addition to chromium, other heavy metals, such as manganese, iron, and arsenic were also characterized and reported.

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ENVIRONMENTAL CHARACTERIZATION OF SOILS AND SEDIMENTS

A Progress Report, Part II

(from the Navy Disposal Site at SNL/CA where Chromium level in the ground water was found to be elevated)

Objective

Characterization of soils and sediments is required in order to assess the baseline contaminant level and establish goals consistent with CERCLA for environmental remedial activities taking place throughout the DOE weapons complex. At Sandia National Laboratories/California (SNL/CA), we have developed and streamlined analytical strategies, including inductively coupled plasma (ICP), x-ray diffraction (XRD), x-ray fluorescence (XRF), and scanning electron microscopy/energy dispersive x-ray spectrometry (SEM/EDS) with image analysis for the characterization of environmental soils (Reference 1). Current efforts include applying the above techniques to characterize the physical and chemical properties of a soil sample from the SNL/CA site, which has an elevated level of chromium in ground-water samples. These results would allow us to identify the source(s) and a scientifically sound solution for the Cr-contamination problem. The properties of particular relevance include baseline levels; the type of minerals or compounds present in the soil and the microstructure; and chemical composition and distribution of Cr-, Mn-, and As-containing compounds.

Experimentation

A. Materials

The two soil samples studied were taken from drill sites at SNL/CA (see Figure 1). The first sample, SNLL-003, was taken from a borehole at a depth of approximately 10 feet on the southern tip of SNL/CA. Both the United States Navy and Lawrence Livermore National Laboratory (LLNL) used this area for general disposal until the late 1950s.

Remediation of the site is being directed by the State of California Regional Water Control Board. Ground water samples from this location have shown up to 10 ppm of chromium. The MCL (maximum contaminant level) for chromium in drinking water is 0.05 ppm. The second sample, BG-100 ft, was taken from a diesel fuel spill site, which is located inside the installation boundary of SNL/CA. Since this area was far enough away from the Navy disposal site, it was used as a background check to determine if levels of chromium were above normal at the Navy disposal site.

B. Sample Preparation

Chunks of samples, approximately 5 cm in size or larger (Figure 2a), were placed in a vacuum oven for 12 hours at 200°C to insure that the excess water was driven off. For the various analyses, portions of this dried sample were subjected to an additional preparation as described below.

1. For ICP and XRD analysis:

A portion of the sample was ground to ~100 µm powder using a mortar and pestle (Figure 2b).

2. SEM/EDS Imaging and XRF Analysis:

A small chunk of sample, approximately 2 cm in size, was non-destructively mounted in epoxy and metallographically polished using a 1 µm diamond grit. The resulting epoxy mounted sample was about 3 cm in diameter (Figure 3).

C. Analytical Approaches

Step 1: ICP to Determine Baseline Bulk Chemical Composition

The powder sample was sent to two EPA certified laboratories (BC Analytical and Eureka Laboratories) for analysis. Both laboratories used the same digestion technique outlined by EPA SW 846 Procedure 6010. The results from the two independent laboratories were compared to assess experimental variation from analysis to analysis.

Step 2: XRD to Identify Minerals or Compounds in the Soil

XRD was used to detect the presence of any compound with a definite crystalline structure. A typical XRD spectrum is illustrated in Figure 4, where each peak corresponds to a “d”-spacing (interplanar spacing) that is unique to a specific compound present in the soil. The limitation of this technique is that it often misses crystalline compounds that are only present in trace amounts (i.e., <1 percent of volume fraction). Also, it is unable to provide chemical information, such as heavy metal impurities in the crystalline compound that is detected.

Step 3: XRF/X-ray Mapping to Determine the Location, Distribution, and Volume Fraction of Heavy Metal-Rich Area

This technique macroscopically maps the intensity of the characteristic X-rays of each element selected throughout the sample cross section, which is approximately one centimeter square. The X-ray intensity variation is proportional to the concentration variation throughout the area. For example, if the X-ray intensity of the Cr-K α X-ray in certain areas is elevated, then the Cr-concentration in those areas is high. This is illustrated in Figure 5, where bright spots, indicative of Cr X-ray, are seen throughout the sample. Volume fraction of the heavy metal area is measured by a image analysis technique called “SEMPER” which is developed by SYNOPTICS Co. and is available in the Electron Optics Laboratory at SNL/CA.

This SEMPER image analysis technique is illustrated in Figure 5. The image that contained the Cr-rich areas (Figures 5a and 5b) is scanned into the computer system. Through a gray level threshold setting, all the Cr-containing particles that are brighter than the threshold level will be counted. These particles appeared bright, as shown in Figure 5d. The total sample area is outlined through another gray level threshold setting as shown in Figure 5e. The volume fraction is measured using the areas of Cr-rich particles shown in Figure 5d, and the areas of total sample area shown in Figure 5e.

Step 4: SEM/EDS/Image Analysis/X-ray Spectroscopy to Characterize the Physical and Chemical Properties of Heavy Metal-Containing Compounds

Once the locations of the heavy metals in the sample cross section are macroscopically identified by XRF, the next step is to microscopically characterize the physical and chemical properties of these areas that are rich in heavy metals. The properties of interest include microstructure, chemical composition (stoichiometry), and elemental distribution. The characterization of these properties can best be achieved by using the SEM/EDS/X-ray related techniques as described below:

- Microstructure is characterized by both secondary electron images (SEI) and backscattered electron image (BEI) in the SEM (Figures 6a-c). The microstructure displayed by the SEI image is based on topographical variations throughout the analysis area. The brighter the area appears, the higher its elevation. The microstructure displayed by the BEI image is based on atomic weight variation throughout the area. If the area contains heavy elements, it should appear bright in the BEI image. The resolution of the current BEI and SEI imaging is about 200Å. The morphology and size of any feature that is larger than 500 Å can be resolved clearly by these images.
- The chemical composition of a feature seen in the SEM image can be measured by the EDS technique non-destructively in a matter of seconds. The results are illustrated in Figure 7, in which each peak represents certain elements that are present in the soil. The composition of a feature can be measured using the relative peak height and a standard sample reference.
- The location of heavy metals is determined by an EDS/X-ray mapping technique. This technique is similar to the XRF-mapping described in step 3, except that it analyzes a much smaller area (<1 mm square area) and with much higher resolution. In addition to the distribution, the physical shape (or morphology) and size of the heavy metal containing areas can clearly be resolved by this X-ray mapping technique. Figure 8 illustrates a typical BEI image of a selected area and

its corresponding multi-element X-ray map of Si, Fe, Ti, K, Na, Al, Ca, Cr, Mg, and O. The distribution of each element in the area is clearly shown. There is a bright (or heavy element-containing) particle at the center. The morphology and size of this particle is clearly defined by both the BEI and the Cr-map. In addition, the X-ray maps show that the particle is also rich in other elements such as Mg, Fe, and O.

Step 5: Conclusion of the Results

By combining the physical and chemical properties learned from the above steps, we are able to address the following issues that are pertinent to the environmental problems on hand:

- Potential source(s) of the heavy metal-containing compound
- The mechanism by which the heavy metals leach into the ground water
- Identify a sound technical solution for cleanup

Experimental Results

A. Bulk Chemical Composition by ICP

The results from both BC Analytical and Eureka Laboratories are shown in Figure 9. For both samples, some variations in results between the two laboratories are seen and the variations are different from element to element. The ICP results from BC Analytical and Eureka indicate that chromium level in the Navy's dumpsite is 29 ppm and 38.6 ppm, respectively. This level is slightly lower than those found in the background site, BG100, where the Cr-level is 38 ppm and 38.9 ppm, respectively.

The above difference in Cr-level between the two sites may not be significant. However, the Cr-level found in the Navy's dumpsite (29 ppm) appear to be noticeably lower than those found previously in the another background site (Site EW-SNL 707-47.3" in Figure 1) where the average Cr-level found to be greater than 50 ppm (Reference 1).

For manganese, the results from Eureka Laboratories indicate that Mn-level in the Navy's dumpsite is 309 ppm which is also lower than those found in the background site where the Mn-level is 484 ppm.

Arsenic contents are lower than 5 ppm in both sites. At this low concentration, it is difficult to draw any conclusion regarding the As-level between the two sites.

B. Identification of Minerals or Compounds in the Soil by XRD

XRD results (Figure 4) show that the common minerals present in both soil samples are quartz, plagioclase, K-feldspar and clay. None of the Cr-, Mn- or As-bearing compounds were detected by the current XRD technique.

C. Location and Distribution and Volume Fraction of Heavy Metals in the Non-Destructed Soil Sample by XRF/X-ray Mapping

An X-ray intensity map of the Cr K μ X-rays for the entire sample cross section of sample No. SNLL-003 is shown in Figure 5. Areas with high Cr X-ray intensity spots ($\ll 100\ \mu\text{m}$) are seen throughout the sample area. This indicates that Cr-rich particles (or areas) are present throughout the sample. Mn and As have not been analyzed by XRF. However, location and distribution of these elements were studied in great detail by SEM/EDS techniques as reported below. The volume fraction of the Cr-rich areas measured by the "SEMPER" image analysis technique is about 0.2-0.5 percent.

D. Physical and Chemical Properties and Distribution of Heavy Metal Containing Compound by SEM/Imaging, SEM/EDS/X-ray Mapping

1. Typical microstructure of the soil SNLL-003 is revealed by BEI and SEI images as shown in Figures 6a-6e.

Based on the brightness variation in these BEI images (Figures 6a-6b and 6e), there are about four minerals or compounds in this sample. Most minerals are dense, uniform, and approximately $100\ \mu\text{m}$ or larger in size. The SEI image shows that in between these large mineral particles there are porous clays (arrow B in Figure 6c). In addition, there are minor amounts of small heavy metal containing particles ($\leq 20\ \mu\text{m}$) throughout the sample (arrow A in Figures 6b and 6e). The particles also appeared to be solid and uniform with particle size about $20\ \mu\text{m}$ being typical (Figures 6e-6f). The composition of these particles is studied below.

2. The chemical composition of the overall area was qualitatively measured by the EDS technique.

It should be noted that the current EDS analysis is not as suitable for quantitative analysis of the trace elements as those done by ICP. The overall sample contains mainly O, Al, Mg, Si, Ca, K, Ti, and Fe. The elements Cr, Mn, and As were not detected in the EDS spectrum (Figure 7) due to the high detection limit (one percent or 10,000 ppm) of the EDS analysis.

Since our primary interest is in heavy metals (i.e., Cr, Mn, and As), chemical composition of the minerals or the compounds that did not contain heavy elements were not analyzed or studied by the EDS analysis.

3. The location of heavy metal-rich areas in SNLL-003 were evaluated by the SEM/EDS/X-ray mapping technique.

Cr -rich particles were observed using the Cr X-ray map as shown in Figure 8 and Figure 11. Both Cr-rich particles are surrounded by the porous clay. It is noted that these particles are the same as the bright and dense particles observed in Figures 6a-f.

Mn was present throughout the clay areas where the Fe was also present. This is illustrated by comparing the X-ray map of Mn and Fe shown in Figures 12a and 12c for the area shown in Figure 12b.

4. Chemical composition (or stoichiometry) of heavy metal containing particles and areas by EDS/X-ray microanalysis.

Two types of Cr-rich particles were found in the sample. The first type is found in Figure 8 and Figure 10. They contain Cr, Fe, Al, Mg, and O. The chemical composition of these particles is measured by EDS analysis quantitatively as shown in the table of Figure 13. With five elements in this compound, its chemical formula is unclear. The second type of Cr-containing particle is the one shown in Figure 11. This particle contains Fe, Cr, and O (Figure 14). The chemical composition measured by EDS matches well with FeCr_2O_4 (iron chromite) as shown in the table of Figure 14.

The Mn-impurity in the clay shown in Figure 12 is measured also by EDS analysis (Figure 15). The area contains approximately 15-20 wt % of Mn as shown in the table of Figure 15.

5. As-rich areas were not detected by the SEM/X-ray mapping technique. This means that if it were present, it might be in trace amounts and distributed uniformly throughout. In this case, it would be too low to be detected by the current EDS/mapping analysis .

Summary and Discussion

A. Chromium

1. The Cr-content in the soil of the Navy dumpsite was found to be slightly less than that of the background area. This finding is consistent with that determined from the soil taken at the Lawrence Livermore National Laboratory (LLNL) site where the level of Cr in the ground water was also elevated.
2. Approximately 0.2-0.5 percent volume fraction of Cr-rich compounds (i.e., FeCr_2O_4 and Cr-Fe-Mg-Al-O compounds) was found at the Navy Site, No. SNL-003. The microstructure and morphology of the particles were dense and uniform and very similar to those of the natural minerals in the soil.

Because of the above characteristics of the Cr-rich particles, we suspect that they are naturally occurring minerals rather than an externally introduced contaminant. These natural Cr-rich minerals most likely are the source of the elevated Cr in the ground water.

3. The remaining question is, "What are the mechanisms and kinetics through which the Cr is leached into the water?" To address this question, a group in LLNL is conducting a study to determine why the levels of micro nutrients in the Cr-contaminated and background areas were found to be different. The group is also interested in determining how bacteria affect the decomposition of the Cr-containing compounds.

B. Manganese

Mn found in the soil, taken from the Navy disposal site, is primarily located in the same area where Fe is located. This observation is

consistent with the fact that Fe and Mn have a strong tendency to substitute for one another. It is conceivable that the Mn is a natural impurity in Fe-rich natural clay.

C. Arsenic

Arsenic was only detected by ICP and its level is much less than 5 ppm in both soils. Since the X-ray map of arsenic did not show any As-rich areas, and if arsenic were present in the soils, it is an impurity that is distributed uniformly in trace amounts throughout. It is certainly not present as distinctive particles as those observed for Cr and Mn.

D. The Unique Capability of the X-ray Mapping Technique

Although the presence of the trace elements (i.e., Cr, Mn, and Mn) are not detectable by the EDS and XRD analyses because of the localized high X-ray intensity, their location and distribution can easily be revealed and characterized.

Compounds that are small in size ($\ll 5 \mu\text{m}$) and present only in trace amounts often could not be characterized fully and/or identified positively by the current SEM/EDS techniques. These shortcomings result from the limitation of the spot size ($0.2 \mu\text{m}$) of the electron beam and X-ray fluorescence volume ($1\text{-}5 \mu\text{m}$) of the sample in the SEM. For this type of compound, the X-ray absorption fine structure technique (XAFS) is a promising procedure that can positively identify mineral phases and their stoichiometry (Reference 3).

Future Work

Since some of the Chromite particles and Mn-rich particles in the current soils are small ($\ll 5 \mu\text{m}$) and highly porous, we are planning to use XAFS to confirm the type of chromate and Mn-rich compound that are identified by the EDS technique. This experiment can be conducted at the Stanford Cyclotron facility. We are currently in contact with Stanford authorities for arranging the XAFS experiment in FY 1994.

The Mn- and Fe-rich component(s) in the soil are highly porous. Their composition could not be measured quantitatively by the current EDS analysis. We plan to apply XAFS to identify these compounds.

Since both LLNL and SNL/CA have a similar Cr-related environmental problem at hand, both laboratories have agreed to combine their technical efforts in the future to address this problem.

The current analytical strategies will be applied for the soil characterization in conjunction with the other bio-remediation activities for the gasoline spill problem at SNL/CA.

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1. "Environmental Characterization of Soils and Sediments," a progress report, N. Y. C. Yang, SNL/CA; D. K. Smith, LLNL, June 1992.
2. "Image Analysis Code SEMPER," developed by Synoptics Co.
3. "Speciation of Trace Elements in Coal From XAFS Spectroscopy," F. E. Huggins, J. Zhao, N. Shah, and G. P. Huffman, Proceedings of International Conference of Coal Science, Sept. 12-17, 1993, Baniff, Alberta, Canada.

Figure 1. Map of the locations for soil sample BG100 and SNLL-003

Figure 2. Physical form of the soil sample.

(a) As-received

(b) Powder samples for XRD and ICP

Figure 3. Typical soil sample that is metallographically mounted and polished for SEM/EDS/X-ray mapping analysis

Figure 4. Typical XRD spectrum for the soil sample SNLL-003.

Figure 5. Typical optical image and its corresponding X-ray maps done by XRF/X-ray mapping technique.

- (a) Fe K α
- (b) Cr K α
- (c) Optical image at magnification of 4X

Figure 5. (Continued) The images, generated by the SEMPER technique, for the volume fraction measurement.

- (d) Cr -rich particles
- (e) total sample area

Figure 6.

Typical microstructure of the soil SNLL-003 revealed by backscattered electron imaging (BEI) and secondary electron imaging (SEI).

- (a) BEI at 40X
- (b) BEI at 220X, Bright areas contain heavy metal-Arrow A
- (c) SEI at 220X, Porous areas are the clay-Arrow B

Figure 6. (continued)

(d)-(e) SEI and BEI image pair—different area from Figures 5b & 5c. The area also contained heavy metal containing particles.

(f) SEI image at 540X show clearly the morphology and particle size of the bright particle in the Fig. 6e.

Overall Chemical Composition of SNLL-003

Fe (wt%)	Cr (wt%)	Ti (wt%)	Ca (wt%)	K (wt%)	Si (wt%)	Al (wt%)	Mg (wt%)	Na (wt%)	O (wt%)
3.3	0	0.2	0.8	2.0	43.0	8.7	1.0	2.0	39

Figure 7. Typical EDS spectrum (top) and composition (bottom) of the overall area shown in Figure 6a for soil SNLL-003.

Cr, Mn, and As are not detected in this spectrum. The majority of the carbon seen in this spectrum is an artifact due to epoxy as sample mounting material.

Figure 8. Typical result of SEM/EDS/X-ray mapping analysis. It contains the video image of the area of interest and its corresponding X-ray maps of those elements detected by the EDS spectrum illustrated in Fig. 7.

Top row from Left to right: Si, Fe, Ti, K
Middle row from left to right: Na, Al, Ca, Cr
Bottom row from left to right: O, Mg, Video image.

BC Analytical Laboratories			Eureka Laboratories	
	SNLL-003	BG-100 ft	SNLL-003	BG-100 ft
Al	8800	14000	15300	15700
As (ppm)	0.47	0.82	2.4	4.9
Cd (ppm)	<.4	<1	<1	<1
Ca (ppm)	1500	3800	1910	4080
Cu (ppm)	13	26	15.3	25.6
Cr (ppm)	29	38	38.6	38.9
Fe (ppm)	15000	27000	18200	29300
Pb (ppm)	7.7	11	15.9	17.7
Mg (ppm)	3400	6500	3980	6660
Mn (ppm)	*	*	309	484
Ni (ppm)	33	45	39.9	45.3
K (ppm)	1200	2600	1690	2450
Si (ppm)	280	490	*	*
Ag (ppm)	<2	<3	0.5	0.5
Na (ppm)	390	350	5970	505
Ti (ppm)	73	130	*	*
Zn (ppm)	29	60	39.2	63

* Not Available

Figure 9. ICP results from both BC Analytical and Eureka Laboratories.

Generally speaking, the compositions of heavy elements detected in the SNLL-003 are lower than those found in the sample BG100.

Some variations in the results were seen between the two laboratories. In general, for the same element, the results from BC Analytical are lower than those from the Eureka Laboratory.

Figure 10. Another Cr-containing particle and its corresponding X-ray maps for the particle found in the SNLL-003 soil (type I).

Figure 11. Cr-rich particle and its corresponding Cr and Fe Maps (type II).

Figure 12. Porous clay area and its corresponding Fe and Mn maps.

Composition of Cr Containing Particle

Fe (wt %)	Cr (wt %)	Al (wt %)	Mg (wt %)	O (wt %)
7.7	19.3	26.1	38.9	7.9

Figure 13. Typical EDS spectrum of the Cr-rich particle (type I) and its composition.
In addition to Cr, Fe, Al, Mg, and O were detected in the particle.

Composition of Cr-Rich Particle

	Fe (wt %)	Cr (wt %)	O (wt %)
Cr-rich particle (measured)	28.0	47.6	remaining
Fe ₂ Cr O ₄ (calculated)	25.0	46.4	28.6

Figure 14. Typical EDS spectrum of the Cr-rich particle (type II) and its composition.

The particle is FeCr₂O₄.

Composition of Mn-Rich Particle

Mn (wt. %)	Fe (wt. %)	Si (wt. %)	Ca (wt. %)	K (wt. %)	O (wt. %)	Ti (wt. %)	Al (wt. %)	Mg (wt. %)
17.87	12.56	29.73	1.36	1.57	19.69	1.86	13.45	1.92

Figure 15. Typical EDS spectrum of the Mn-rich clay and its composition.

The area contains Fe, Mn, Si, Al, Ti, K, Ca, Mg, and O.

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