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Technical Support for the Soiltech
Soil Washing Project

T. S. Tomascik

✓ Interim Report

✓ August 1994

Prepared by
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MARTIN MARIETTA ENERGY SYSTEMS, INC.
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EXECUTIVE SUMMARY

The organic removal ability of a surfactant solution was studied for an "as-received" soil sample. A 15% surfactant solution was added to an equal portion of the soil sample, by volume, and blended. The mixture was then stirred with a magnetic stirrer. A black precipitate resulted, which was then periodically skimmed off the top of the solution. This was done at both room temperature and at 150°F.

The soil sample was examined before and after processing with optical microscopy, environmental scanning electron microscopy (ESEM), energy dispersive x-ray microanalysis (EDS), and analytical chemical analysis (total oil and grease and petroleum hydrocarbons).

Optical microscopy revealed that the solution, when used at both room temperature and 150°F, did clean the soil but not entirely. Scanning electron microscopy and energy dispersive x-ray microanalysis detected the presence of organic material in the as-received soil but did not detect the organic material after processing at both temperatures. Analytical chemistry provided the most insight into the solution's ability to remove the organic material from soil. A 33.3% decrease in total oil and grease and petroleum hydrocarbons was found for the sample processed at room temperature; a 16.67% decrease in total oil and grease and a 33.3% decrease in petroleum hydrocarbons was found for the sample processed at 150°F. Since there was an insignificant difference in the solution's ability to remove organic material at both room temperature and 150°F, and, since only a small amount of soil sample was available for analysis, processing at 150°F was not pursued further in this study; however, its potential usefulness in increasing the solution's capability to remove organic material from the soil has not been ruled out.

To more closely approximate the actual parameters used in a larger soil washing system, another study was performed. Soil was processed at room temperature, using a 60:1 solution to soil ratio, by volume. Because of the small amount of soil that remained for this study, optical microscopy was hindered (a thin cross section could not be prepared for analysis), and total oil and grease analysis could not be performed. ESEM/EDS analysis produced similar results to those previously discussed. A 74.4% decrease in petroleum hydrocarbons was, however, found, through analytical chemical analysis, which indicates that the larger ratio of solution to soil was a significant contributor to the effectiveness of the process.

1. INTRODUCTION

A two pound soil sample was received from Soiltech Environmental Systems, Inc. The sample is representative of the contaminated soil of a petroleum refinery.

2. SAMPLE PROCESSING

2.1 OVERALL OBJECTIVE OF SAMPLE PROCESSING

The sample was processed with a surfactant solution, provided by Soiltech, to determine if this solution would be potentially effective for implementation in a larger process, such as soil washing. The surfactant solution's primary goal was removal of organic materials, such as, asphaltine in the soil.

2.2 SELECTION AND SAMPLE PREPARATION

Portions of the sample that were selected for analysis were randomly selected from the overall, representative sample. A portion of the sample was analyzed in the as-received form, dried, molded with epoxy, and polished to achieve a uniform cross sectional area. The dried sample was also fragmented and examined.

2.3 CHARACTERIZATION AND ANALYSIS

The goal of soil characterization was to determine the composition of the soil and the organic material it contained, to better understand the mechanisms by which the soil constituents and organic material are entwined. An assumption that this single sample is representative of the entire site was made. The soil was characterized by optical microscopy, particle size, environmental scanning electron microscopy (ESEM), energy dispersive x-ray microanalysis (EDS), x-ray diffraction (XRD), and U. S. Environmental Protection Agency certified analytical methods (EPA418.1: total petroleum hydrocarbons, and EPA413.2: total oil and grease).

2.4 SURFACTANT PROCESSING

A portion of the as-received sample was blended with a solution of surfactant (10% by volume) and mechanically stirred at both room temperature and at an elevated temperature (150°F). The resulting solids were then analyzed to determine how the properties of the soil sample were altered by the surfactant solution.

3. CHARACTERIZATION AND ANALYSIS BEFORE SAMPLE PROCESSING

3.1 VISUAL INSPECTION

The soil, as received, can be seen in Figure 3.1. The soil ranged in color from dark grey to black and was moist before processing. The soil components were of various sizes and did not appear to be strongly bonded; however, in regard to the finer particles, a great deal of agglomeration was encountered. Many of the particles were spherical in shape. Some of the particles were soft, almost clay-like, and could be crushed by hand, especially the agglomerates. When crushed, some left a black residue that had a gritty, oily consistency. The soil was not magnetic in nature, except for a few fine particles.

3.2 OPTICAL MICROSCOPY

A thin cross section of a polished sample was analyzed by optical microscopy at low power (in most cases, less than 150X) to gain a visual perspective of the soil's components and relation of those components to the organic material.

Figure 3.2a illustrates the typical way in which soil components and organic material are spatially oriented. Organic material encases both individual soil components and agglomerates of components. (The dark brown circle that encompasses particles in the center of the picture is the organic material. The lighter tan color in the background is the epoxy that was used to hold the particles together.) The primary soil components are quartz crystals and feldspars.

In a few instances, soil particles were bound together by organic material, as can be seen in Figure 3.2b. The darker spheres within the particulate mass are organic material as well. Figure 3.2c shows a large amount of organic material surrounding quartz crystals. Agglomerations of the organic material can also be seen in this figure. The lower left portion of the pink crystal in this figure was looked at with a higher magnification (250X) to better discern the boundaries between crystal and organic material. This can be seen in Figure 3.2d. In this figure, the organic material is the lighter tan colored material surrounding the crystal; the epoxy appears as the dark brown substance.

Optical microscopy proved to be a very useful tool in providing a physical relation between the organic material and the soil constituents. The organic material was optically discovered to be present in a solid form throughout the sample as well as serving as a coating for the soil components. Optically, the organic material was not found within any of the several individual particles that were examined (the soil components did not exhibit high levels of

porosity). This is a positive factor toward washing the soil components with surfactant because penetration into the particulate subsurface would be much harder to remove.

3.3 Environmental Scanning Electron Microscope (ESEM) & Energy Dispersive X-ray Microanalysis (EDS)

ESEM and EDS were used jointly to analyze the sample in both as-received form and in epoxied, polished form. The ESEM was used at various magnifications ranging from a low of about 180X to a high in excess of 25,000X, depending on the characteristics of the particular area of the sample that was examined, and with both gaseous and backscatter detectors. The ESEM beam was set at 15.0 KeV for the analysis.

As expected, the main soil component was silicon, however, some other elements were also seen such as iron, calcium, potassium, magnesium, and in some instances, carbon. In its as-received form (see Figure 3.3a), the sample was not optimized for analytical study because its uneven surface hampered scattering and greatly lowered the achievable number of counts on the EDS, reducing reliability of the results; therefore, most of the analysis was done with the polished cross section. The polished cross section was dried and mounted with epoxy but otherwise was no different in composition than the as-received sample, thus, the results for both should be comparable. A macroscopic view of the polished cross section that was used can be seen in Figure 3.3b. Figure 3.3c shows a portion of the polished cross section in the ESEM, at low magnification; Figure 3.3d shows the EDS spectrum for the same "bulk" area shown in Figure 3.3c.

The carbon in the EDS spectrum primarily indicates the presence of organic material in the sample. The carbon that was found at one specific point in the sample melted under the 15 keV ESEM beam because of its organic nature. A picture of this carbon in the preprocessed soil can be seen in Figure 3.3e; Figure 3.3f is the EDS spectrum for this particular area of carbon. The spectrum clearly shows the carbon peak, as well as the other constituents, such as silicon and calcium, that are present.

(Note that low energy x-rays, associated with low Z elements, have low detection efficiencies, therefore, a significant amount of the element (generally, greater than five weight percent) is needed to obtain a detectable signal.)

The ESEM was also used to examine the content of a few small fractured particles from the sample. They were found to be representative of the overall sample. The carbon in the fractured sample appears as small nodules. This can be seen in Figure 3.3g. An EDS spectrum is also presented in Figure 3.3h. (Note the carbon intensity.)

The major role of the ESEM and EDS in this report was that of a comparative tool to see how the carbon appeared in the as-received sample and if it still could be seen after surfactant processing. A lack of carbon in the processed cross section would indicate the success of the surfactant solution in removing the organic material; whereas, presence of carbon would indicate either failure or only partial success. Some additional photos of the as-received, unpolished surface are given in Figures 3.3i through 3.3l.

3.4 X-RAY DIFFRACTION

The soil sample was also analyzed by x-ray diffraction. The resulting diffractogram (Figure 3.4) showed the presence of quartz, calcite, feldspar, and magnetite. Due to the complexity of the diffractogram, several lines were not identified, but are believed to be typical soil components, such as, amphibole, layered silicates (chlorite and micas), and other minor minerals usually found in soil. As expected, no evidence of organic material was found in the diffractogram.

3.5 PARTICLE SIZE

The particle size for a representative portion of the soil sample was analyzed. The results are summarized in Figure 3.5.

3.6 ANALYTICAL CHEMISTRY ANALYSIS

The analytical chemistry results are presented in Table 3.6 for the as-received sample. Total Oil and Grease in the soil was found to be 6000 ppm and total petroleum hydrocarbons was found to be 3900 ppm. These numbers are believed to be variable due to the heterogeneity of the soil sample. Previous results for total oil and grease have yielded concentrations as high as 36000 ppm.

4. CHARACTERIZATION AND ANALYSIS AFTER SAMPLE PROCESSING

4.1 VISUAL INSPECTION

The soil, after surfactant processing, can be seen in Figure 4.1. After processing, the material was similar in appearance to the as-received soil, with the exception that the surfactant processing breaks down the smaller pieces by mechanical blending, so most of the larger agglomerates were destroyed.

4.2 OPTICAL MICROSCOPY

The soil components of the post-processed sample were finer than the as-received sample. This change can be attributed to the physical and chemical agitation of the process. Two thin cross sections were prepared. The first cross section was for a sample that had been processed at room temperature; the second, was for a sample that had been processed at 150°F.

Figure 4.2a shows quartz crystals and other soil components from the sample that was processed at room temperature. Less organic material can be seen around the crystals than before processing (compare with Figure 3.2c); however, organic material is still visible (dark brown and black material). Figure 4.2b shows a larger area of the sample. Soil components are no longer bound together by organic material, but organic material is still visible as distinguishable agglomerates.

Figure 4.2c shows soil components (including quartz and feldspars) from the sample processed at 150°F. Organic material is still present in agglomerated form. Figure 4.2d shows soil components of various sizes after processing at 150°F that are in close proximity to organic agglomerates. Particles and agglomerates, as in the case of the sample processed at room temperature, are no longer encased by organic material. The least amount of organic material can be seen in the sample that was processed with the heated surfactant solution. The thin cross sections of the processed samples, when analyzed with microscopy, indicate that surfactant processing is more effective when the solution is heated than when used at room temperature; however, neither method is completely effective in the removal of organic material.

4.3 ENVIRONMENTAL SCANNING ELECTRON MICROSCOPE (ESEM) & ENERGY DISPERSIVE X-RAY MICROANALYSIS (EDS)

After the sample was processed with surfactant, a black precipitate resulted. This precipitate was analyzed with the ESEM and EDS. A photo of the precipitate from the ESEM is given in Figure 4.3a; its respective EDS spectrum is presented in Figure 4.3b. The precipitate was very high in carbon content, which indicated that the surfactant succeeded in removing a portion of the organic material from the sample.

A picture of the sample that was used for ESEM/EDS analysis is given in Figure 4.3c. Various areas of the sample were checked for residual organic material. In every area examined, the carbon peak was not detected by EDS, indicating that the surfactant processing was effective in removing the organic material to levels below ESEM detection limits. The elemental composition of the soil agreed with that of the as-received sample. Figure 4.3d shows surface and subsurface details from the processed sample as imaged by the

ESEM's backscatter detectors. Most of the larger agglomerates are gone and the sample surface consists primarily of finer particles. Figure 4.3e shows the EDS spectrum for the area of the sample illustrated in Figure 4.3d. Notice the absence of the carbon peak, indicating that the organic material has been removed.

Two sets of photos and spectra from other locations on the sample have also been included in this report. (Figures 4.3f through 4.3i) Figure 4.3f shows an area of sample that has few agglomerates; carbon was not detected by EDS in this area (Figure 4.3g). Figure 4.3h shows an area where some agglomeration at the microscopic level is still present; however, the EDS spectrum from this area (Figure 4.3i) shows that, even in agglomerated areas, no carbon is present.

The ESEM/EDS analysis after processing with the surfactant solution indicates that a major portion of the organic material has been removed from the soil sample.

4.4 ANALYTICAL CHEMISTRY ANALYSIS

Analytical chemistry analyses were performed for both the sample that was processed at room temperature and the sample that was processed at 150°F. The results are presented in Tables 4.4a and b. Petroleum hydrocarbons and total oil and grease are both lower than as-received by 33.3%. Results are not significantly different (petroleum hydrocarbons is identical, total oil and grease differ by only 20%) for the sample processed at room temperature and the sample processed at 150°F, indicating that the heated processing does not provide appreciably better results than the room temperature processing.

5. PROCESSING WITH A LARGER SURFACTANT SOLUTION TO SOIL RATIO

5.1 DESCRIPTION AND RATIONALE

The as-received soil was processed with a higher surfactant solution to soil ratio (60:1, by volume) to better approximate the soil washing process that are currently in use, that use a solution to soil ratio of approximately 200:1. The ratio of 60:1 was chosen because of blender volume limitations. Thirty grams of soil were placed in a blender with 750 ml of 15% surfactant solution and were blended, at room temperature, for 10 minutes. The solution was allowed to sit for 5 minutes and was then blotted with an oil absorbing pad to remove the oily precipitate that developed. This process was carried out six times over a 1.5 hour period. A clean oil absorbing pad was used each time. Figure 5.1 shows the oil absorbing pads that were used.

5.2 VISUAL INSPECTION/OPTICAL MICROSCOPY

The soil sample appeared to be the same color and have the same texture as it did when it was processed previously (Figure 5.2).

The sample was examined under a low power (40X) optical microscope. Organic material was not detected optically; however, the ability to examine the sample more closely, as before, was lost because only a small portion of the as-received soil remained after the initial processing; this portion was too small to make into a thin cross section for further examination.

5.3 ESEM AND EDS ANALYSIS

The processed sample was examined again with the environmental scanning electron microscope and analyzed with energy dispersive x-ray microanalysis. The analysis was impeded by the same limitations as before, in that, detection of organic material is, generally, only possible if it exists in a sample in greater than approximately 5 wt%. The sample also provides more accurate ESEM/EDS results when it is fashioned into a smooth cross section; because of the small amount of sample that remained for this particular analysis, a smooth cross section was not made. Figures 5.3a and b show the processed sample at a magnification of 180X, as seen with gaseous scatter and backscatter detectors, respectively. Figure 5.3c shows the EDS spectrum for the processed sample. The major soil components are silicon, calcium, aluminum, potassium and iron. Carbon (i.e. organic material) is not detected. Figures 5.3d and e show a closer view (1000X) of the surface and subsurface of the processed sample as seen with gaseous scatter and backscatter detectors. The EDS spectrum for this smaller region of the sample was identical to that of Figure 5.3c, and was, therefore, not included.

5.4 ANALYTICAL CHEMISTRY ANALYSIS

The results of the analytical chemistry analysis for total oil and grease and petroleum hydrocarbons for the processed sample are given in Table 5.4. The benefit of using a large surfactant solution to soil ratio can be seen. Petroleum hydrocarbons were reduced by 74.4% (1000 ppm as opposed to 3900 ppm, as-received). Analysis of total oil and grease was not performed on the processed sample because there was an insufficient amount of soil for an accurate analysis.

6. SUMMARY

Organic material was removed from the soil sample most effectively by the 15% surfactant solution in a large (60:1 in this study) ratio, by volume, with the soil at room temperature. No significant benefit was found in heating the solution above room temperature; however, this course of action has not been ruled out as a subject for further consideration. The most insight into the effectiveness of the solution's ability to remove organic material from the soil was provided by the analytical chemistry analyses, the results of which are summarized below.

TABLE 6.1. SUMMARY OF ANALYTICAL CHEMISTRY ANALYSES RESULTS

	As - Received	Processed at Room Temperature	Processed at 150°F	Processed at 60:1 Solution to Soil Ratio
Oil & Grease	0.6	0.4	0.5	N/A
Petroleum Hydrocarbons	0.39	0.26	0.26	0.10

Note: Units are weight percent.

APPENDIX A
FIGURES

Figure 3.1: As-Received Soil

Figure 3.2a: Soil Components Encased by Organic Material (40X)

Figure 3.2b: Organic Agglomerates and Soil Components Bound Together by Organic Material (100X)

Figure 3.2c: Quartz Crystals Surrounded by Organic Material (100X)

Figure 3.2d: Closeup of Crystal/Organic Material Boundary (250X)

Figure 3.3a: Vacuum Dried Soil Sample

Figure 3.3b: Polished Cross Section of Dried Sample

Figure 3.3c: Polished Cross Section in ESEM (135X)

31-May-1998 12:15:43

Execution time = 2 second

soil-polished .

Vert= 1000 counts Disp= 1

Preset= 2 Kint

Elapsed= 384 secs

Figure 3.3d: EDS Spectrum of Polished Cross Section

Figure 3.3e: Organic Material Found with ESEM (220X)

Figure 3.3g: Fractured Soil Particles (Carbon Nodules) (845X)

Figure 3.3f: EDS Spectrum for an area of Sample in Which Organic Carbon was Encountered

Figure 3.3h: EDS Spectrum of Fractured Particles

Figure 3.3i: As-Received Soil Surface (ESEM, in GSD Mode) (260X)

Figure 3.3j: As-Received Soil Surface & Subsurface (ESEM, in BSD Mode) (260X)

Figure 3.3k: As-Received Soil Surface (ESEM) (285X)

Figure 3.3l: Closeup of As-Received Soil Surface (ESEM) (2050X)

Figure 3.4: X-Ray Diffractogram for As-Received Soil

Soil component size follows standard mineralogical definitions.

Figure 3.5 Particle Size Analysis for Petroleum Refinery Soil Sample

Figure 4.1: Processed Soil

Figure 4.2a : Quartz Crystals in Epoxy After Processing at Room Temperature (100X)

Figure 4.2b: Closeup of Quartz Crystal After Processing at Room Temperature (100X)

Figure 4.2c: Feldspar & Quartz After Processing at 150F (100X)

Figure 4.2d: Soil Components & Organic Material After Processing at 150 F (250X)

Figure 4.3a: Process Precipitate (ESEM) (395X)

30-Jun-1994 11:59:08

Execution time = 4 second

SOIL TECH SMEAR

Vert= 500 counts Disp= 1

Preset= 2 Kint

Elapsed= 396 secs

Quantex>

Figure 4.3b: EDS Spectrum of Process Precipitate (Smear)

Figure 4.3c: Processed Soil, Mounted for ESEM/EDS Analysis

Figure 4.3d: Processed Soil Sample (ESEM) (300X)

Figure 4.3e: EDS Spectrum of Processed Soil Sample

Figure 4.3f: Processed Sample (Fine Particles) (ESEM) (195X)

Figure 4.3h: Processed Sample (Agglomerates) (ESEM) (195X)

Figure 4.3g: EDS Spectrum of Processed Soil
(Area Devoid of Visible Agglomeration)

Figure 4.3i: EDS Spectrum of Processed Soil
(Some Visible Agglomeration Remaining)

Figure 5.1: Oil Absorber Pads Used in Processing at Room Temperature
With a 60:1 Surfactant Solution to Soil Ratio

Figure 5.2: Soil Processed at Room Temperature with a 60:1 Surfactant
Solution to Soil Ratio by Volume (40X)

Figure 5.3a: Soil Processed at 60:1 Surfactant Solution to Soil Ratio by Volume (ESEM, 180X, GSD Mode)

Figure 5.3b: Soil Processed at 60:1 Surfactant Solution to Soil Ratio by Volume (ESEM, 180X, BSD Mode)

23-Jun-1994 12:41:47

SOIL TECH #17

Vert= 2685 counts Disp= 1

Preset=

2 Kint

Elapsed=

205 secs

Figure 5.3c: EDS Spectrum for Sample Processed with 60:1 Surfactant Solution to Soil Ratio by Volume

Figure 5.3d: Soil Processed at 60:1 Surfactant Solution to Soil Ratio by Volume (ESEM, 1000X, GSD Mode)

Figure 5.3e: Soil Processed at 60:1 Surfactant Solution to Soil Ratio by Volume (ESEM, 1000X, BSD Mode)

APPENDIX B

TABLES

Table 3.6: Total Oil & Grease and Total Hydrocarbons for As - Received Soil

Date Printed:
24-JUN-1994 16:22

Oak Ridge K-25 Site
Analytical Chemistry Department

Results of Analyses

ANALIS ID: 940622-034	Project: K008 002	Customer Sample ID: MS940091
Customer: HENSON		Requisition Number:
Date Sampled: 20-JUN-1994		Date Sample Received: 22-JUN-1994
Sampled By: BH		Date Sample Completed: 24-JUN-1994
Material Description: SOIL		Date Sample Approved: 24-JUN-1994
Program Manager: MH BARTLING (# 29893)		[] : Result has been Corrected for Spike

Procedure No.	Analysis	Result	Q Qual	Units	Analyst	QA File Number	Date Completed
***** Wet Chemistry Laboratory *****							
EPA-9071	Oil and Grease	0.6		%	KM HOWERTON	94-2A	24-JUN-1994
ACD-184203.R1	Petroleum Hydrocarbons	0.39		%	AS DELCARMEN	94-7	24-JUN-1994

Replicate Results of Analysis

Analysis	Results	Replicate Results	RPD
Petroleum Hydrocarbons	0.39	0.36	8.0

Table 4.4a: Total Oil & Grease and Total Hydrocarbons for Sample Processed at Room Temperature.

Oak Ridge K-25 Site			Date Printed:
Analytical Chemistry Department			24-JUN-1994 16:23
Results of Analyses			
AnalIS ID: 940622-035	Project: K008 002	Customer Sample ID: MS940092	
Customer: HENSON		Requisition Number:	
Date Sampled: 20-JUN-1994		Date Sample Received: 22-JUN-1994	
Sampled By: BH		Date Sample Completed: 24-JUN-1994	
Material Description: SOIL		Date Sample Approved: 24-JUN-1994	
Program Manager: MH BARTLING (# 29893)		[] : Result has been Corrected for Spike	

Procedure No.	Analysis	Result	Q Qual	Units	Analyst	QA File Number	Date Completed

***** Wet Chemistry Laboratory *****							
EPA-9071	Oil and Grease	0.4		%	KM HOWERTON	94-2A	24-JUN-1994
ICD-184203.R1	Petroleum Hydrocarbons	0.26		%	AS DELCARMEN	94-7	24-JUN-1994

Table 4.4b: Total Oil & Grease and Total Hydrocarbons for Sample Processed at 150°F.

Oak Ridge K-25 Site			Date Printed:
Analytical Chemistry Department			24-JUN-1994 16:23
Results of Analyses			
AnalIS ID: 940622-036	Project: K008 002	Customer Sample ID: MS940093	
Customer: HENSON		Requisition Number:	
Date Sampled: 20-JUN-1994		Date Sample Received: 22-JUN-1994	
Sampled By: BH		Date Sample Completed: 24-JUN-1994	
Material Description: SOIL		Date Sample Approved: 24-JUN-1994	
Program Manager: MH BARTLING (# 29893)		[] : Result has been Corrected for Spike	

Procedure No.	Analysis	Result	Q Qual	Units	Analyst	QA File Number	Date Completed

***** Wet Chemistry Laboratory *****							
EPA-9071	Oil and Grease	0.5		%	KM HOWERTON	94-2A	24-JUN-1994
ACD-184203.R1	Petroleum Hydrocarbons	0.26		%	AS DELCARMEN	94-7	24-JUN-1994

Table 5.4: Total Oil & Grease and Total Hydrocarbons for Sample Processed at Room Temperature with a Surfactant Solution to Soil Ratio of 60:1 by Volume.

Date Printed:
24-JUN-1994 16:23

Oak Ridge K-25 Site
Analytical Chemistry Department
Results of Analyses

AnalIS ID: 940622-037	Project: K008 002	Customer Sample ID: MS940094
Customer: HEWSON		Requisition Number:
Date Sampled: 20-JUN-1994		Date Sample Received: 22-JUN-1994
Sampled By: BH		Date Sample Completed: 24-JUN-1994
Material Description: SOIL		Date Sample Approved: 24-JUN-1994
Program Manager: MH BARTLING (# 29893)		[] : Result has been Corrected for Spike

39

Procedure No.	Analysis	Result	Q Qual	Units	Analyst	QA File Number	Date Completed

***** Wet Chemistry Laboratory *****							
ACD-184203.R1	Petroleum Hydrocarbons	0.10		%	AS DELCARMEN	94-7	24-JUN-1994

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