1R - 237

GENERAL CORRESPONDENCE

YEAR(S):

1995

Shell Oil Products Company

Two Shell Plaza
P. O. Box 2099
Houston, TX 77252-2099

December 12, 1995

RECEIVED
DEC 13 1995
Environmental Bureau
Oil Conservation Division

William Olson
State of New Mexico Oil Conservation Division
Environmental Bureau
2040 S. Pacheco St.
Santa Fe, New Mexico 87504

SUBJECT: CLOSURE REPORT, SHELL PIPE LINE CORPORATION'S HOBBS PIT, LEA COUNTY NEW MEXICO.

Dear Mr. Olson,

Enclosed is Environmental Spill Control's assessment report for the pit located at Shell Pipe Line Corporation's Hobbs Station. This assessment was conducted to identify the potential horizontal and vertical migration, if any, of hydrocarbon. No hydrocarbon contamination was detected, horizontally, outside of the pit. Vertically, total petroleum hydrocarbon (TPH) concentrations decrease rapidly 5-6 feet below pit bottom. Volatile component concentrations were all below Oil Commission Division action levels and decreased rapidly with depth. Leaching analyses of the higher TPH and volatile samples demonstrates that the remaining hydrocarbon is not leachable but immobile.

In summary, contamination is limited to the area immediately beneath the pit, volatile concentrations are negligible, leaching analyses shows the remaining hydrocarbon to be immobile, and the source of the contamination has been removed. The former pit is located on Shell Pipe Line's crude oil pumping station in an industrial/rural setting. I feel that the former pit is not a threat to either public health or the environment. I am requesting approval to close the pit in-place by dozing the side walls into the excavation. The final elevation will be such that it ties to the existing topography and will be graded to allow drainage off of the fill.

I appreciate your consideration of this request. If you have any questions please call me at 713-241-2961.

Sincerely,

Neal Stidham

Neal Stidham
Staff Engineer
Shell Oil Products Company
Representing Shell Pipe Line Corporation

ENVIRONMENTAL
SPILL CONTROL, INC.

NM GENERAL CONTRACTORS LIC. #55535
TX DRILLING LIC. #5005M
NM DRILLING LIC. #WD 1349

P.O. BOX 5890 ★ HOBBS, NM 88241
PHONE (505) 392-6167 ★ FAX (505) 397-5085

November 29, 1995

Mr. Neal D. Stidham
Environmental & Technical
Shell Oil Company
Room 1452, Two Shell Plaza
777 Walker Street
Houston, Texas 77002

RE: **SUBSURFACE INVESTIGATION**
FORMER B S & W PIT
NEW HOBBS STATION
LEA COUNTY, NEW MEXICO

Dear Mr. Stidham:

Environmental Spill Control, Inc. (ESC) has completed a Subsurface Investigation of the former basic sediment and water (BS & W) pit located at Shell Pipe Line Corporation's (SPLC) New Hobbs Station, Lea County, New Mexico. The purpose of the subsurface investigation was to delineate the extent and magnitude of hydrocarbon contamination, if present, within the former pit area to assist in the development of a final pit closure plan.

Additional objectives of this investigation included identifying site specific criteria needed to determine target remediation levels, documenting the variations in contaminant levels at various distances from their source (pit), and assessing the migration potential of the contaminants.

The subsurface investigation was conducted from June 29, 1995 to November 3, 1995 using a four phase approach:

> Phase I - Identify the general site characteristics required to determine a recommended remediation level in accordance with New Mexico Oil Conservation Division (NMOCD) guidelines.
>
> Phase II - Advance two soil borings in the floor of the former pit to determine if hydrocarbon contamination is present and provide a baseline of contaminant levels.
>
> Phase III - Advance six soil borings in the former pit area to explore the subsurface stratigraphy and to delineate the extent of hydrocarbon impact.
>
> Phase IV - Advance five soil borings and collect surface samples to access the magnitude of hydrocarbon impact and provide sufficient information on subsurface conditions to evaluate the migration potential of the contaminants.

Background

The former BS & W pit is located on the west side of an active crude oil pipeline pumping station operated by SPLC. The station is situated approximately 3 miles south of the city of Hobbs in a rural part of Lea County, New Mexico. The station contains a field office, pipe yard, aboveground crude oil storage tank and pipeline facilities and is surrounded by unimproved pasture land.

The initial phase of pit closure was conducted in June 1995. This work included removal and off site disposal of the contents of the pit. The previous assessment activities have characterized the pit contents as non-hazardous with the primary contaminant identified as petroleum hydrocarbons. The materials removed consisted of a hydrocarbon based semisolid (crude oil sludge) that exhibited characteristics typically found in oil field / pipeline waste stored in BS & W pits. The pit contents were reclaimed at a NMOCD permitted facility.

No record of construction is available, but the pit appears to have been constructed of native soil and lined with a one foot thick layer of gray clay or bentonite. Currently, the former pit excavation is approximately 185 feet long by 77 feet wide with the bottom of the excavation approximately four feet below the existing ground surface. The pit excavation is surrounded by a berm composed of native soil approximately three feet high by 10 feet wide.

Phase I - General Site Characteristics

A field survey of the site and surrounding area was conducted to identify the general site characteristics required to determine a recommended remediation level in accordance with the Unlined Surface Impoundment Closure Guidelines published by the NMOCD. The site characteristics of concern are depth to groundwater, the distance to fresh water sources, and the distance to the nearest surface body of water.

During the field survey no surface bodies of water were identified within 2,500 feet of the site. One rural residence (potential water source) is located approximately 800 feet north of the former pit. One on-site water well is located approximately 800 feet southeast of the former pit. This well is owned by SPLC and currently used as an industrial water source for the station. Althrough the well is frequently tested and meets Primary Drinking Water Standards, it not used as a potable water source. Drinking water for the facility is supplied by a bottled water service. Depth to groundwater in the station water well was gauged in July and November 1995 by ESC personnel and measured approximately 36 feet below ground surface.

According to the NMOCD Closure Guidelines the recommended remediation level is determined using a modified risk based approach based on the general site characteristics. Because the depth to groundwater in the former pit area is less than 50 feet below the bottom of the pit, the recommended remediation levels of the hydrocarbon impacted soils are as follows:

Benzene - 10 ppm
Total BTEX (benzene, toluene, eythlbenzene, and xylene) - 50 ppm
TPH (total petroleum hydrocarbon) - 100 ppm

The TPH remediation level of 100 ppm was based solely on the fact that the depth to groundwater beneath the site is less than 50 feet. If the depth to groundwater were greater than 50 feet a remediation level of 1,000 ppm or 5,000 ppm would have applied.

Phase II -Subsurface Assessment

A multiphase approach was used during this investigation to assure complete characterization of the subsurface while utilizing the information obtained from each phase to design the drilling and sampling plans for the subsequent phases.

During the initial drilling phase of this investigation, two soil borings (B-1 and B-2) were drilled on June 29, 1995 to determine if hydrocarbon contamination exists in the soils underlying the bottom of the pit excavation. Boring B-1 was placed near the center of the southeast quarter of the pit floor and drilled to a total depth of 17 feet. Boring B-2 was placed near the center of the northwest quarter of the pit floor and drilled to a total depth of 12 feet.

The soil borings were drilled using a truck-mounted air rotary drill rig with a 4 1/2 inch bit. Soil samples were obtained from the borings using a driven split spoon at approximately three foot intervals through the upper ten feet and at five foot intervals, thereafter. The total depth of each boring and any changes in sampling frequency were determined by the ESC on-site representative based on field screening results obtained during drilling.

A steam cleaner was used to clean drilling and sampling equipment after completion of each boring. A soapy water solution and rinse were used to clean sampling equipment between samples. These procedures minimize the possibility of cross-contamination.

The soil sample obtained from each interval was split into three sets of soil samples. One sample was screened for relative amounts of volatile organic constituents with a Foxboro Model 128 Organic Vapor Analyzer (OVA) using the head space procedure described in Unlined Surface Impoundment Closure Guidelines published by the NMOCD. Based on the OVA readings and visible staining observed in the field, a second sample from selected intervals was analyzed for TPH levels with a General Analysis Corporation (GAC) MEGA TPH analyzer using EPA Method 418.1.

The field OVA and TPH results were used to select the samples to be submitted to the laboratory for BTEX analysis using EPA Method 8020. The samples to be submitted for laboratory analysis were placed into glass jars with teflon-lined lids and zero head space, sealed with QA/QC seals, and preserved at 4C in accordance with EPA protocol for laboratory shipment.

Once drilling and sampling operations were completed, the borings were abandoned by filling each bore hole with non-shrinking grout containing 5 percent bentonite.

Analytical results from the soil samples retrieved from the borings identified hydrocarbon contamination in the soils underlying the former pit. However, the results indicated that the hydrocarbon concentrations decrease rapidly with depth. TPH levels ranged from 4,900 ppm in the 5 to 6 foot interval of B-1 to 65 ppm in the 10 to 11 foot sample of B-1. Total BTEX concentrations ranged from 1.446 ppm in the 5 to 6 foot interval of B-1 to 0.002 ppm in the 10 to 11 foot interval of B-2.

Phase III -Delineation of Hydrocarbon Impact

During this phase of this investigation, six soil borings (B-1T, B-2T, B-3, B-4, B-5, and B-6) were drilled on August 18, 1995 in order to explore the subsurface stratigraphy at the site and complete delineation of the vertical and horizontal extent of hydrocarbon impact. Two of the borings were drilled adjacent to B-1 and B-2 to complete the vertical delineation of the hydrocarbon impacted soils previously identified. Three additional borings (B-3, B-5, and B-6) were randomly located in the bottom of the pit area to assess subsurface conditions beneath the entire former pit area. Boring B-4 was placed on the center of the earthen berm surrounding the pit to assess the integrity of the berm. The borings were drilled to depths ranging from 21 to 40 feet using the same field procedures performed during the initial drilling phase. The approximate locations of the soil borings are shown on the site map in Appendix A.

Because the analytical results obtained during the phase II activities indicated that the primary constituents of concern (BTEX) make up a relatively small portion of the total amount of hydrocarbons (TPH) identified in the soils, the sampling plan was modified to include TPH analysis using the synthetic precipitation leaching procedure (SPLP; EPA Method 1312). The field OVA and TPH results were used to select the samples to be submitted to the laboratory for TPH analysis using SPLP. The SPLP test was developed to represent the leaching potential for materials under natural conditions, i.e. precipitation.

The SPLP procedure consists of immersing the sample in a slightly acidic extraction fluid and tumbling in a sealed container for 18 hours to dissolve any soluble constituents present. The extraction fluid was then filtered and the filtrate subjected to TPH analysis by EPA Method 418.1. The analytical results of this procedure represent the total amount of petroleum compounds (TPH) that could be leached from the impacted soils by acidic waters (simulated rain water).

The soil stratigraphy encountered during the boring operations beneath the pit area appears to be relatively uniform with minor variations noted in strata thickness. In general, the subsurface of the site consists of between 25 to 35 feet of multicolored fine-grained calcareous sands (caliche) containing scattered sandstone and limestone stringers overlying a brown non-calcareous to slightly calcareous fine-grained sand (SM). Groundwater was encountered in the basal sand at a depth of approximately 35 feet below the bottom of the excavation indicating that the depth to the water table is approximately 39 feet below ground surface. No soil staining, hydrocarbon odors, or free-floating hydrocarbons were observed near the water table. No water samples were obtained during this investigation because the loose sand collapsed the bore hole in both borings.

The drilling logs included in Appendix B provide a more detailed description of the subsurface conditions. These logs are composites of the soil descriptions, screening, and analytical results obtained from the boring operations performed at that location.

Analytical results from the soil samples retrieved during phase III activities from the borings confirmed that hydrocarbon contamination decreases rapidly with depth and indicate that the vertical extent of contamination is limited to the soils above the water table (vadose zone). TPH concentrations in the soil samples collected immediately above the water table at a depth of 33 to 35 feet ranged from 7 ppm to 5 ppm. TPH levels in the boring that penetrated the berm of the former pit measured 3 ppm and indicate that the horizontal extent of hydrocarbon impacted soils is confined to the area within the pit berm.

TPH analysis of the SPLP leachate from the split samples (samples containing TPH concentrations ranging from 845 ppm to 138 ppm) recorded levels less than the method detection limit of 1 ppm.

Phase IV - Characterization of Hydrocarbon Impact

Five additional soil borings were drilled on November 1, 1995 and five hand auger samples (HA-1, HA-2, HA-3, HA-5, and HA-6) were collected on November 3, 1995 to complete the drilling phase of the investigation. The purpose of this phase of the investigation was to access the magnitude of hydrocarbon contaminant concentrations in the shallow soils beneath the former pit and to obtain soil samples for laboratory testing to determine the migration potential of the contaminants. These borings were placed adjacent to borings B-1, B-2, B-3, B-5, and B-6 and drilled to a total depth of 6 feet using the same field procedures performed during the previous two phases. The hand auger samples were collected from the upper one foot of soil adjacent to each of the boring locations.

Because the previous TPH - SPLP analytical results indicated that little or no hydrocarbons could be leached from the hydrocarbon impacted soils by rain water, the sampling plan was modified to include TPH and BTEX analysis using SPLP.

The soil sample obtained from each sampled interval was split into four sets of soil samples. One sample was screened for VOCs, the second sample was analyzed for TPH levels, and the third sample was submitted to the laboratory for SPLP-TPH analysis. The forth sample obtained from all the sampled intervals where the OVA readings exceeded 100 ppm was submitted to the laboratory for BTEX analysis using the synthetic precipitation leaching procedure (SPLP; EPA Method 1312) in order to simulate a worst case scenario of hydrocarbon contaminants migrating through the soil by leaching.

A summary of the analytical results is presented in Table 1 and the laboratory reports and chain-of-custody are included in Appendix C.

Migration Potential of Contaminants

One of the main objectives of this investigation was to predict the potential for migration of any contaminants identified in the subsurface. In order to evaluate the potential for migration a knowledge of the contaminants of concern, physical properties of the site, transport mechanisms, and a method of evaluation is required.

While crude oil is a complex mixture of naturally occurring hydrocarbon compounds and other trace constituents, all crude oils are predominantly composed of widely varying amounts of three classes of hydrocarbons; cycloalkanes, alkanes, and aromatics. The bulk of hydrocarbons that make up crude oil, especially weathered crude, are not regarded as very toxic. The relative concentrations of known or suspected carcinogens (certain BTEX and polycyclic aromatic hydrocarbon (PAH) compounds) typically represents a very small portion of the crude oil. Weathering further reduces BTEX and PAH levels through biodegradation, oxidation, and selective dissolution.

The analytical results obtained during this investigation recorded relatively high concentrations of TPH and low levels of BTEX in each sampled interval indicating that the hydrocarbon contamination beneath the former pit is the result of weathered crude impact.

Contaminant migration through soil above the water table includes liquid and vapor phase transport mechanisms. The hydrocarbon contamination pattern exhibited by the impacted soils beneath the former pit shows a definite decrease in both volatile and non-volatile concentrations with depth (distance from source) and contained no free-phase hydrocarbon liquids. For hydrocarbon impacted soils containing no free-phase hydrocarbon liquids, the primary transport mechanism for the potential migration of the contaminants is leaching from the soils by rain water.

In order to simulate a worst case scenario of hydrocarbon contaminants leaching through the soil selected samples were submitted to the laboratory for TPH and BTEX analysis using the synthetic precipitation leaching procedure (SPLP).

TPH levels ranged from 9,780 ppm to 138 ppm in the sampled intervals selected for analysis using the SPLP procedure. TPH analysis of the SPLP leachate from the split samples recorded levels ranging from 1 ppm to less than the method detection limit (1 ppm to 0.5 ppm). BTEX analysis of the SPLP leachate from the split samples recorded levels ranging from 0.043 ppm to 0.001 ppm while the average BTEX concentration of the sampled soils was approximately 2.8 ppm.

The results indicate that the contamination identified in the soils beneath the former pit is composed of hydrocarbon compounds with a chemical make up similar to weathered crude. The SPLP analytical results indicate that a majority of the TPH concentrations identified in the soils is relatively inert and will release little or no hydrocarbons into the environment through leaching by acidic waters (rainfall).

Summary

The vadose zone beneath the site is approximately 36 feet thick and composed of fine-grained calcareous sands (caliche) containing scattered sandstone and limestone stringers. These soils are underlain by a slightly calcareous fine-grained sand representing the Ogallala aquifer with the top of the water table encountered at a depth of approximately 38 feet below ground surface.

The recommended remediation levels for the site as determined from the NMOCD Unlined Surface Impoundment Closure Guidelines are 10 ppm benzene, 50 ppm total BTEX, and 100 ppm TPH. The TPH remediation level of 100 ppm was based solely the fact that the depth to groundwater beneath the site is less than 50 feet. If the depth to groundwater were greater than 50 feet at remediation level of 1,000 ppm or 5,000 ppm would have applied.

The results of this investigation indicate that the horizontal extent of hydrocarbon impact is restricted to the soils within the former pit area surrounded by the earthen berm. The vertical extent of hydrocarbon impact (soils containing TPH levels greater than 100 ppm) is confined to the soils underlying the floor of the former pit and extends to a maximum depth of 25 feet.

Analytical results from the soils forming the bottom of the pit excavation recorded BTEX concentrations ranging from 3.59 ppm to 0.001 ppm. These concentrations are well below NMOCD recommended remediation levels (benzene < 10 ppm, BTEX < 50 ppm).

TPH levels ranged from 9,780 ppm to 3 ppm with the distribution of TPH concentrations indicating that the levels decrease rapidly with depth. TPH concentrations exceeding 5,000 ppm appear confined to the near surface soils above a depth of 3 feet below the pit floor. Soils containing TPH concentrations between 5,000 ppm to 1,000 ppm appear to be confined to a depth of less than 7 feet.

TPH levels ranged from 9,780 ppm to 138 ppm in the sampled intervals selected for analysis using the SPLP procedure. TPH analysis of the SPLP leachate from the split samples recorded levels ranging from 1 ppm to less than the method detection limit (1 ppm to 0.5 ppm). BTEX analysis of the SPLP leachate from the split samples recorded levels ranging from 0.043 ppm to 0.001 ppm while the average BTEX concentration of the sampled soils was approximately 2.8 ppm.

Conclusions

This investigation identified an area of hydrocarbon contamination beneath the former pit that is restricted to the soils above the water table. The hydrocarbon impacted soils exhibit characteristics similar to those of weathered crude and contained BTEX concentrations well below NMOCD recommended remediation levels with TPH levels exceeding the recommended remediation level in the shallower portion of the impact area. However, SPLP leachate results indicate that a large majority of the TPH concentrations are not water soluble and will release little or no hydrocarbons into the environment through leaching by acidic waters (rainfall).

Because the source of the release has been removed, the site is not located in a sensitive area, and the bulk of the hydrocarbon contamination is inert, in ESCs opinion, the remaining hydrocarbon impacted soils pose a low risk of contaminating the water table.

ESC appreciates the opportunity to be of service. If you have any questions or desire further information, please contact us at any time.

Sincerely,

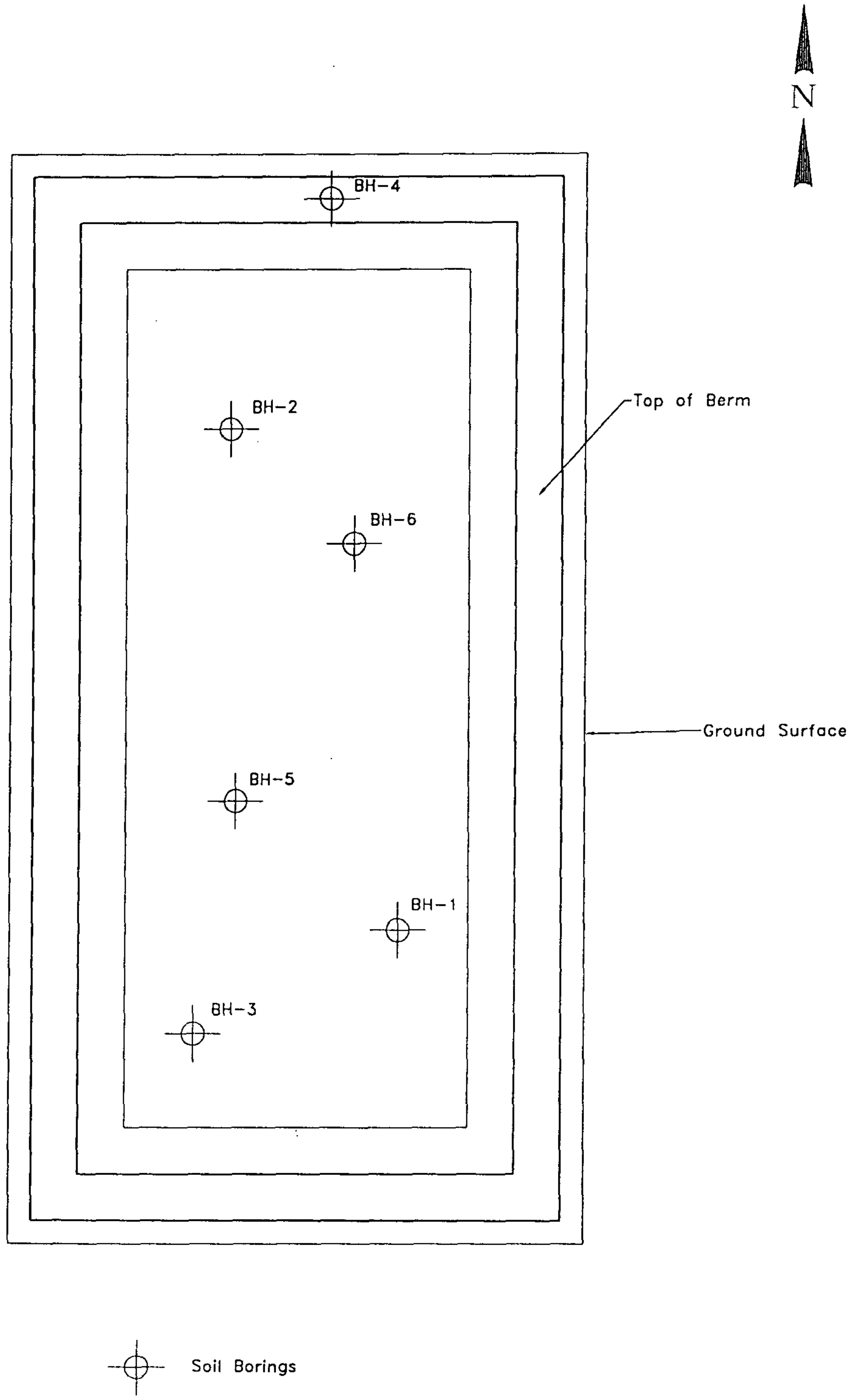
ENVIRONMENTAL SPILL CONTROL, INC.

F. Wesley Root

F. Wesley Root
Division Manager
Hydrology/Geology

cc: Mr. Allen Hodge
Attachments

APPENDIX A
FIGURES



SHELL PIPELINE

PIT EXCAVATION
NEW HOBBS STATION
SW4 SEC15, T19S, R38E
LEA Co., NEW MEXICO

DATE:8-3-95	DRAWN M.F.G.	REV. DATE	BY:
SCALE: 1" = 40'		JOB # 124	
DRAWING 1 OF 1		FILE:	

TABLE 1
NEW HOBBS STATION
SOIL SAMPLE ANALYTICAL RESULTS

Soil samples obtained during boring operations on June 29, August 18, and November 1, 1995

Boring	Sample Interval (feet)	Field OVA	Benzene	Toluene	Ethyl-benzene	Xylenes	Total BTEX	Field TPH	TPH (SPLP)	Total BTEX (SPLP)
HA-1	0 - 1	28	-	-	-	-	-	7,240	<0.5	-
B-1, B-1T	2 - 3	>1,000	0.480	0.210	0.700	2.200	3.590	3,380	0.5	0.011
B-1, B-1T	5 - 6	>1,000	0.036	0.110	0.100	1.200	1.446	4,900	<1	-
B-1, B-1T	10 - 11	130	<0.001	0.037	0.032	0.620	0.689	845	<1	-
B-1T	15 - 16	63	-	-	-	-	-	740	<1	-
B-1T	16 - 17	39	-	-	-	-	-	84	-	-
B-1T	23 - 24	4	-	-	-	-	-	6	-	-
B-1T	32 - 33	<1	-	-	-	-	-	<5	-	-
HA-2	0 -1	45	-	-	-	-	-	3,080	<0.5	-
B-2, B-2T	2 - 3	250	0.400	0.180	0.130	1.500	2.210	212	<0.5	0.001
B-2, B-2T	5 - 6	650	0.120	0.069	0.046	0.650	0.885	970	<1	-
B-2, B-2T	10 - 11	220	<0.001	<0.001	<0.001	0.002	0.002	65	-	-
HA-3	0 - 1	55	-	-	-	-	-	9,780	1.0	-
B-3, B-3T	2 - 3	470	-	-	-	-	-	6,070	<0.5	0.043
B-3, B-3T	5 - 6	300	<0.001	<0.001	<0.001	0.001	0.001	1,662	<0.5	0.020
B-3	25 - 26	5	-	-	-	-	-	160	<1	-
B-3	30 - 31	3	-	-	-	-	-	86	-	-
B-4	10 - 11	<1	-	-	-	-	-	3	-	-
B-4	20 - 21	<1	-	-	-	-	-	3	-	-

- Sample was not analyzed for this parameter.
Organic vapor analyzer (OVA) measurements were obtained using a Foxboro Model 128 OVA (headspace method).
Field total petroleum hydrocarbon (TPH) results were performed during boring operations using a MEGA-TPH analyzer (modified EPA Method 418.1).
Benzene, toluene, ethylbenzene, and xylenes (BTEX); TPH; TPH using the synthetic precipitation leaching procedure (SPLP); and BTEX using SPLP results are listed in mg/kg (parts per million; ppm). Analyses were conducted using EPA Method 8020 (BTEX), EPA Method 418.1 (TPH), EPA Method 1312 (SPLP).

TABLE 1
NEW HOBBS STATION
SOIL SAMPLE ANALYTICAL RESULTS

Soil samples obtained during boring operations on June 29, August 18, and November 1, 1995

Boring	Sample Interval (feet)	Field OVA	Benzene	Toluene	Ethyl-benzene	Xylenes	Total BTEX	Field TPH	TPH (SPLP)	Total BTEX (SPLP)
HA-5	0 - 1	15	-	-	-	-	-	9,410	<0.5	-
B-5, B-5T	2 - 3	60	-	-	-	-	-	7,650	<0.5	-
B-5, B-5T	5 - 6	45	-	-	-	-	-	724	<0.5	-
B-5	17 - 18	40	-	-	-	-	-	289	<1	-
B-5	20 - 21	15	-	-	-	-	-	63	-	-
HA-6	0 -1	22	-	-	-	-	-	8,740	1.0	-
B-6, B-6T	2 -3	47	-	-	-	-	-	3,630	0.8	-
B-6, B-6T	5 - 6	25	-	-	-	-	-	470	<0.5	-
B-6	15 - 16	8	-	-	-	-	-	138	<1	-
B-6	20 - 21	30	-	-	-	-	-	59	-	-
B-6	35 - 36	5	-	-	-	-	-	7	-	-

- Sample was not analyzed for this parameter.

Organic vapor analyzer (OVA) measurements were obtained using a Foxboro Model 128 OVA (headspace method).

Field total petroleum hydrocarbon (TPH) results were performed during boring operations using a MEGA-TPH analyzer (modified EPA Method 418.1).

Benzene, toluene, ethylbenzene, and xylenes (BTEX); TPH; TPH using the synthetic precipitation leaching procedure (SPLP); and BTEX using SPLP results are listed in mg/kg (parts per million; ppm). Analyses were conducted using EPA Method 8020 (BTEX), EPA Method 418.1 (TPH), EPA Method 1312 (SPLP).

APPENDIX B

DRILLING LOGS

Company Drilled for: Shell Pipeline Corporation	ENVIRONMENTAL SPILL CONTROL, Inc. PHONE (505) 392-6167 FAX (505) 397-5085	# Drilling Log
Location: Former BS&W Pit, Hobbs Station SW4 Sec 15, T19S, R37E Lea Co., New Mexico	Well/Bore Number: B-1 Dates Drilled: Composite 6-29-95, 8-18-95, 11-1-95	Driller: AH/JT Logged By: F. Wesley Root

Drilling Method: Air Rotary	Depth of Boring: 17 Feet	Depth of Well: N/A	Length of Casing: N/A	Length of Screen: N/A
Bore Diameter: 5 Inch	Casing Diameter: N/A	Screen Diameter: N/A	Slot Size: N/A	Well Material: Plugged w/Grout

Depth	Lithology
0	Pit Surface
	Light gray and brown silty, calcareous, fine-grained sand (caliche).
	Light gray calcareous fine-grained sand (caliche) containing silicious streaks.
	Light brown silty calcareous fine-grained sand (caliche).
	Brown slightly calcareous fine-grained sand (SM).
40	Bottom Of Boring @40'

Depth: 0, 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, 80, 85, 90, 95, 100, 105

Sample Type	OVA (PPM)	TPH	TPH SPLP	BTEX SPLP	Remarks
Hand Auger	28	7,240	≤0.5		
Split Spoon	≥1000	3,380	0.5	0.011	
Split Spoon	≥1000	4,900	<1		
Split Spoon	≥1000	845	≤1		
Split Spoon	130				
Split Spoon	63 39	740 84	<1		
Split Spoon	30				
Split Spoon	4	6			
Split Spoon	9				
Split Spoon	10	<5			
Split Spoon	<1				
					1

Well Design

Remarks
1. Ground water encountered during drilling.

Company Drilled for: Shell Pipeline Corporation		ENVIRONMENTAL SPILL CONTROL, Inc. PHONE (505) 392-6167 FAX (505) 397-5085		
Location: Former BS&W Pit, Hobbs Station SW4 Sec 15, T19S, R37E Lea Co., New Mexico		Well/Bore Number: B-2	Date Drilled: Composite 6-29-95, 8-18-95, 11-1-95	Driller: AH/BK
				Logged By: F. Wesley Root
Drilling Method: Air Rotary	Depth of Boring: 26 Feet	Depth of Well: N/A	Length of Casing: N/A	Length of Screen: N/A
Bore Diameter: 5 Inch	Casing Diameter: N/A	Screen Diameter: N/A	Slot Size: N/A	Well Material: Plugged w/Grout

Drilling Log

Depth	Lithology	Sample Type	OVA (PPM)	TPH	TPH SPLP	BTEX SPLP	Remarks	Well Design	Depth
0	Pit Surface								0
	Gray and brown mottled, calcareous, fine-grained sands and clays (caliche).	Hand Auger	45	3,080	<0.5				
		Split Spoon	250	212	<0.5	0.001			
5		Split Spoon	650	970	<1				5
10		Split Spoon	87	65					10
15	Light pink calcareous fine-grained sand (caliche).	Split Spoon	65						15
		Cuttings	30						
20	Light brown, silty, calcareous fine-grained sand. (caliche).	Split Spoon	28						20
25		Cuttings	18						25
		Split Spoon	2						
30	Bottom Of Boring @26'								30
35									35
40									40
45									45
50									50
55									55
60									60
65									65
70									70
75									75
80									80
85									85
90									90
95									95
100									100
105									105

Company Drilled for: Shell Pipeline Corporation		ENVIRONMENTAL SPILL CONTROL, Inc. PHONE (505) 392-6167 FAX (505) 397-5085		
Location: Former BS&W Pit, Hobbs Station SW4 Sec 15, T19S, R37E Lea Co., New Mexico		Well/Bore Number: B-3	Date Drilled: Composite 8-18-95, 11-1-95	Driller: AH/JT
				Logged By: F. Wesley Root
Drilling Method: Air Rotary	Depth of Boring: 31 Feet	Depth of Well: N/A	Length of Casing: N/A	Length of Screen: N/A
Bore Diameter: 5 Inch	Casing Diameter: N/A	Screen Diameter: N/A	Slot Size: N/A	Well Material: Plugged w/Grout

Drilling Log

Depth	Lithology	Sample Type	OVA (PPM)	TPH	TPH SPLP	BTEX SPLP	Remarks	Well Design	Depth
0	Pit Surface								0
	Gray and brown silty, calcareous, fine-grained sand (caliche).	Hand Auger	55	9,780	1.0				
		Split Spoon	470	6,070	<0.5	0.043			
5		Split Spoon	300	1,662	<0.5	0.020			5
10		Split Spoon	30						10
15		Split Spoon	45						15
20	Light brown calcareous fine-grained sand (caliche).	Split Spoon	20						20
25		Split Spoon	5	160	<1.0				25
30		Split Spoon	3	86					30
35	Bottom Of Boring @31'								35
40									40
45									45
50									50
55									55
60									60
65									65
70									70
75									75
80									80
85									85
90									90
95									95
100									100
105									105

ENVIRONMENTAL SPILL CONTROL, Inc.
PHONE (505) 392-6167
FAX (505) 397-5085

Drilling Log

Company Drilled for: Shell Pipeline Corporation			
Location: Former BS&W Pit, Hobbs Station SW4 Sec 15, T19S, R37E Lea Co., New Mexico			
Well/Bore Number: B-4	Date Drilled: 8-18-95	Driller: AH/JT	Logged By: F. Wesley Root

Drilling Method: Air Rotary	Depth of Boring: 21 Feet	Depth of Well: N/A	Length of Casing: N/A	Length of Screen: N/A
Bore Diameter: 5 Inch	Casing Diameter: N/A	Screen Diameter: N/A	Slot Size: N/A	Well Material: Plugged w/Grout

Depth	Lithology	Sample Type	OVA (PPM)	TPH	TPH SPLP	BTEX SPLP	Remarks	Well Design	Depth
0	Top of Pit Berm								0
	Gray limestone.								
5	Light gray silty, calcareous, fine-grained sand (caliche).								5
		Split Spoon	<1						
	Pit bottom 7' below top of Berm.								
10	White, silty, calcareous, fine-grained sand (caliche).								10
		Split Spoon	<1	3					
15		Split Spoon	<1						15
20		Split Spoon	<1	3					20
	Bottom Of Boring @21'								
25									25
30									30
35									35
40									40
45									45
50									50
55									55
60									60
65									65
70									70
75									75
80									80
85									85
90									90
95									95
100									100
105									105

ENVIRONMENTAL SPILL CONTROL, Inc.
PHONE (505) 392-6167
FAX (505) 397-5085

Drilling Log

Company Drilled for: Shell Pipeline Corporation

Location: Former BS&W Pit, Hobbs Station
SW4 Sec 15, T19S, R37E
Lea Co., New Mexico

Well/Bore Number: B-5
Date Drilled: Composite 8-18-95, 11-1-95
Driller: AH/JT
Logged By: F. Wesley Root

Drilling Method: Air Rotary
Depth of Boring: 30 Feet
Depth of Well: N/A
Length of Casing: N/A
Length of Screen: N/A

Bore Diameter: 5 Inch
Casing Diameter: N/A
Screen Diameter: N/A
Slot Size: N/A
Well Material: Plugged w/Grout

Depth	Lithology	Sample Type	OVA (PPM)	TPH	TPH SPLP	BTEX SPLP	Remarks	Well Design	Depth
0	Pit Surface								0
	Gray and white, silty, calcareous, fine-grained sand (caliche).	Hand Auger	15	9,410	<0.5				
5		Split Spoon	60	7,650	<0.5				5
		Split Spoon	45	724	<0.5				
10		Split Spoon	30						10
15	Light brown silty calcareous fine-grained sand (caliche).	Split Spoon	15						15
	White and light brown silty calcareous fine-grained sand (caliche). Containing minor amounts of sand.	Split Spoon	40	289	<1				
20		Split Spoon	15	63					20
25	Light brown silty calcareous fine-grained sand (caliche).	Split Spoon	2						25
30		Split Spoon	1						30
	Bottom Of Boring @30'								

Company Drilled for: Shell Pipeline Corporation	ENVIRONMENTAL SPILL CONTROL, Inc. PHONE (505) 392-6167 FAX (505) 397-5085
Location: Former BS&W Pit, Hobbs Station SW4 Sec 15, T19S, R37E Lea Co., New Mexico	

Drilling Log

Well/Bore Number: B-6	Date Drilled: Composite 8-18-95, 11-1-95	Driller: AH/JT	Logged By: F. Wesley Root

Drilling Method: Air Rotary	Depth of Boring: 40 Feet	Depth of Well: N/A	Length of Casing: N/A	Length of Screen: N/A
Bore Diameter: 5 Inch	Casing Diameter: N/A	Screen Diameter: N/A	Slot Size: N/A	Well Material: Plugged w/Grout

Depth	Lithology	Sample Type	OVA (PPM)	TPH	TPH SPLP	BTEX SPLP	Remarks	Well Design	Depth
0	Pit Surface								0
	Light gray and brown, silty, calcareous, fine-grained sand (caliche).	Hand Auger	22	8,740	1.0				
		Split Spoon	47	3,630	0.8				
5	Brown and white limestone marl.	Split Spoon	25	470	<0.5				5
10	Light gray and brown, calcareous, fine-grained sand (caliche).	Split Spoon	6						10
15		Split Spoon	8	138	<1.0				15
20	Light brown, silty, calcareous, fine-grained sand (caliche).	Split Spoon	30	59					20
25		Split Spoon	14						25
30		Split Spoon	10						30
	Gray, sandy, limestone stringer.								
35	Red-brown, silty, fine-grained sand (SM) containing scattered caliche rock fragments.	Split Spoon	5	7			1		35
40	Bottom Of Boring @40'								40
45									45
50									50
55									55
60									60
65									65
70									70
75									75
80									80
85									85
90									90
95									95
100									100
105									105

Remarks

1. Ground water encountered during drilling

APPENDIX C

SOIL ANALYTICAL RESULTS

ENVIRONMENTAL SPILL CONTROL, INC.
123 West Dunnam
P.O. Box 5890
Hobbs, NM 88241
(505) 392-6167 (800) 390-6167

SOIL ANALYSIS REPORT

DATE: 06-29-95
CLIENT: Shell Pipe Line Corp.
Houston, Texas
SAMPLED BY: F. Wesley Root

FACILITY: New Hobbs Station
LOCATION: Lea County, New Mexico

DATE SAMPLED: 06-29-95
SAMPLE MATRIX: soil

Sample ID	Time Collected	OVA (ppm)	TPH (ppm)	Description
B-1 (5-6)	09:00	>1,000	4,900	Split-spoon sample
B-1 (10-11)	09:20	130	845	Split-spoon sample
B-1 (15-16)	09:35	63	740	Split-spoon sample
B-1 (16-17)	09:45	39	84	Split-spoon sample
B-2 (5-6)	15:25	650	970	Split-spoon sample
B-2 (10-11)	15:40	220	65	Split-spoon sample

COMMENTS: Total petroleum hydrocarbon (TPH) analysis was performed during boring operations using a General Analysis Corporation MEGA TPH Analyzer (EPA Method 418.1). Organic vapor readings were obtained using a Century Model 128 Organic Vapor Analyzer (OVA) by the headspace procedure as described by the New Mexico Oil Conservation Division in Guidelines for Remediation of Leaks, Spills, and Releases (August 13, 1993)

F. Wesley Root
Signature of Analyst

S01H7.SAM

ENVIRONMENTAL SPILL CONTROL, INC.
123 West Dunnam
P.O. Box 5890
Hobbs, NM 88241
(505) 392-6167 (800) 390-6167

SOIL ANALYSIS REPORT

DATE: 08-18-95
CLIENT: Shell Pipe Line Corp.
Houston, Texas
SAMPLED BY: F. Wesley Root

FACILITY: New Hobbs Station
LOCATION: Lea County, New Mexico

DATE SAMPLED: 08-18-95
SAMPLE MATRIX: soil

Sample ID	Time Collected	OVA (ppm)	TPH (ppm)	Description
B-1 (23-24)	10:00	4	6	Split-spoon sample
B-1 (32-33)	10:10	<1	<5	Split-spoon sample
B-3 (25-26)	14:50	5	160	Split-spoon sample
B-3 (30-31)	15:00	3	86	Split-spoon sample
B-4 (10-11)	12:10	<1	3	Split-spoon sample
B-4 (20-21)	12:25	<1	3	Split-spoon sample
B-5 (5-6)	12:40	45	724	Split-spoon sample
B-5 (17-18)	12:55	40	289	Split-spoon sample
B-5 (20-21)	13:10	15	63	Split-spoon sample
B-6 (5-6)	10:20	25	470	Split-spoon sample
B-6 (15-16)	10:40	8	138	Split-spoon sample
B-6 (20-21)	10:50	30	59	Split-spoon sample
B-6 (35-36)	11:25	5	7	Split-spoon sample

COMMENTS: Total petroleum hydrocarbon (TPH) analysis was performed during boring operations using a General Analysis Corporation MEGA TPH Analyzer (EPA Method 418.1). Organic vapor readings were obtained using a Century Model 128 Organic Vapor Analyzer (OVA) by the headspace procedure as described by the New Mexico Oil Conservation Division in Guidelines for Remediation of Leaks, Spills, and Releases (August 13, 1993)

F. Wesley Root
Signature of Analyst

S01H8.SAM

ENVIRONMENTAL SPILL CONTROL, INC.
123 West Dunnam
P.O. Box 5890
Hobbs, NM 88241
(505) 392-6167 (800) 390-6167

SOIL ANALYSIS REPORT

DATE: 11-01-95
CLIENT: Shell Pipe Line Corp.
Houston, Texas

FACILITY: New Hobbs Station
LOCATION: Lea County, New Mexico

SAMPLED BY: F. Wesley Root

DATE SAMPLED: 11-01-95
SAMPLE MATRIX: soil

Sample ID	Time Collected	OVA (ppm)	TPH (ppm)	Description
B-1 (2-3)	12:55	>1,000	3,380	Split-spoon sample
B-2 (2-3)	10:15	250	212	Split-spoon sample
B-3 (2-3)	13:15	470	6,070	Split-spoon sample
B-3 (5-6)	13:30	300	1,662	Split-spoon sample
B-5 (2-3)	12:40	60	7,650	Split-spoon sample
B-6 (2-3)	11:15	25	470	Split-spoon sample

COMMENTS: Total petroleum hydrocarbon (TPH) analysis was performed during boring operations using a General Analysis Corporation MEGA TPH Analyzer (EPA Method 418.1). Organic vapor readings were obtained using a Century Model 128 Organic Vapor Analyzer (OVA) by the headspace procedure as described by the New Mexico Oil Conservation Division in Guidelines for Remediation of Leaks, Spills, and Releases (August 13, 1993)

F. Wesley Root
Signature of Analyst

ENVIRONMENTAL SPILL CONTROL, INC.
123 West Dunnam
P.O. Box 5890
Hobbs, NM 88241
(505) 392-6167 (800) 390-6167

SOIL ANALYSIS REPORT

DATE: 11-03-95
CLIENT: Shell Pipe Line Corp.
Houston, Texas

FACILITY: New Hobbs Station
LOCATION: Lea County, New Mexico

SAMPLED BY: F. Wesley Root

DATE SAMPLED: 11-03-95
SAMPLE MATRIX: soil

Sample ID	Time Collected	OVA (ppm)	TPH (ppm)	Description
HA-1 (0-1)	10:00	28	7,240	Hand auger sample
HA-2 (0-1)	09:45	45	3,080	Hand auger sample
HA-3 (0-1)	10:10	55	9,780	Hand auger sample
HA-5 (0-1)	10:15	15	9,410	Hand auger sample
HA-6 (0-1)	09:30	22	8,740	Hand auger sample

COMMENTS: Total petroleum hydrocarbon (TPH) analysis was performed during boring operations using a General Analysis Corporation MEGA TPH Analyzer (EPA Method 418.1). Organic vapor readings were obtained using a Century Model 128 Organic Vapor Analyzer (OVA) by the headspace procedure as described by the New Mexico Oil Conservation Division in Guidelines for Remediation of Leaks, Spills, and Releases (August 13, 1993)

F. Wesley Root
Signature of Analyst

SPL®

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

SPL, INC.

REPORT APPROVAL SHEET

WORK ORDER NUMBER: 95 - 08 - A90

Approved for release by:

M. Scott Sample

M. Scott Sample, Laboratory Director *Date:* 9/11/95

Brent Barron, Project Manager *Date:* 9/11/95

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

Certificate of Analysis No. H9-9508A90-01

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 09/07/95

PROJECT: ESC Project No.124
SITE: LEA County, NM
SAMPLED BY: Envir. Spill
SAMPLE ID: B-1 (15-16)

PROJECT NO: H 16158
MATRIX: SOIL
DATE SAMPLED: 08/18/95
DATE RECEIVED: 08/29/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
Synthetic Precip. Leaching Procedure METHOD 1312 *** Analyzed by: CM Date: 08/30/95	08/30/95		
Petroleum extractables METHOD 418.1* Analyzed by: LD Date: 09/05/95 14:30:00	ND	1	mg/L

ND - Not detected.

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 17th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

SPL, Inc., - Project Manager

SPL®

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

Certificate of Analysis No. H9-9508A90-02

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 09/07/95

PROJECT: ESC Project No.124
SITE: LEA County, NM
SAMPLED BY: Envir. Spill
SAMPLE ID: B-3 (25-26)

PROJECT NO: H 16158
MATRIX: SOIL
DATE SAMPLED: 08/18/95
DATE RECEIVED: 08/29/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
Synthetic Precip. Leaching Procedure METHOD 1312 *** Analyzed by: CM Date: 08/30/95	08/30/95		
Petroleum extractables METHOD 418.1* Analyzed by: LD Date: 09/05/95 14:30:00	ND	1	mg/L

ND - Not detected.

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 17th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

SPL, Inc., - Project Manager

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

Certificate of Analysis No. H9-9508A90-03

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 09/07/95

PROJECT: ESC Project No.124
SITE: LEA County, NM
SAMPLED BY: Envir. Spill
SAMPLE ID: B-5 (17-18)

PROJECT NO: H 16158
MATRIX: SOIL
DATE SAMPLED: 08/18/95
DATE RECEIVED: 08/29/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
Synthetic Precip. Leaching Procedure METHOD 1312 *** Analyzed by: CM Date: 08/30/95	08/30/95		
Petroleum extractables METHOD 418.1* Analyzed by: LD Date: 09/05/95 14:30:00	ND	1	mg/L

ND - Not detected.

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 17th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

SPL, Inc., - Project Manager

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

Certificate of Analysis No. H9-9508A90-04

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 09/07/95

PROJECT: ESC Project No.124
SITE: LEA County, NM
SAMPLED BY: Envir. Spill
SAMPLE ID: B-6 (15-16)

PROJECT NO: H 16158
MATRIX: SOIL
DATE SAMPLED: 08/18/95
DATE RECEIVED: 08/29/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
Synthetic Precip. Leaching Procedure METHOD 1312 *** Analyzed by: CM Date: 08/30/95	08/30/95		
Petroleum extractables METHOD 418.1* Analyzed by: LD Date: 09/05/95 14:30:00	ND	1	mg/L

ND - Not detected.

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 17th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

SPL, Inc., - Project Manager

QUALITY CONTROL DOCUMENTATION

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

** SPL QUALITY CONTROL REPORT **

Matrix: Aqueous

Reported on: 09/05/95
Analyzed on: 09/05/95
Analyst: LD

This sample was randomly selected for use in the SPL quality control program. Samples chosen are fortified with a known concentration in duplicate. The results are as follows:

Petroleum extractables
METHOD 418.1*

SPL Sample ID Number	Blank Value mg/l	Amt Added mg/l	Matrix Spike Recovery %	Matrix Spike Duplicate Recovery %	Relative Percent Difference %	QC Limits Recovery	RPD Max.
BLANK	ND	4.0	92.5	95.0	2.7	82. - 112	9.8

9509051200RS -9509020

Samples in batch:

9508A19-02B
9508A90-02B
9508B22-02B
9508B33-01B
9508B33-05B
9508A19-03B
9508A90-03B
9508B22-03B
9508B33-02B
9508B33-06B
9508A19-05B
9508A90-04B
9508B22-04B
9508B33-03B
9508B53-01B
9508A90-01B
9508B22-01B
9508B22-05B
9508B33-04B
9508B85-02B

COMMENTS:

SPL, Incorporated

Cynthia Schreiner, QC Officer

CHAIN OF CUSTODY
AND
SAMPLE RECEIPT CHECKLIST

9508A90

SHELL OIL COMPANY
RETAIL ENVIRONMENTAL ENGINEERING

CHAIN OF CUSTODY RECORD NO. H 16158

Date: 8-28-95
Page 1 of 1

SITE ADDRESS: Hobbs Station Shell Pipe Line Corp, Lea County, New Mexico

WIC #: ESC Project No. 124

CONSULTANT NAME & ADDRESS: Environmental Spill
1203 West Dunnam P.O. Box 5890
Hobbs, New Mexico 88241

CONSULTANT CONTACT: F. Wesley Root

PHONE: 505-397-6167 FAX: 397-5085

SAMPLED BY: F. Wesley Root

CHECK ONE BOX ONLY CT/DT

- QUARTERLY MONITORING ☐ 5461
- SITE INVESTIGATION ☒ 5441
- SOIL FOR DISPOSAL ☐ 5442
- WATER FOR DISPOSAL ☐ 5443
- AIR SAMPLER - SYS O+M ☐ 5452
- WATER SAMPLE - SYS O+M ☐ 5453
- OTHER ______ ☐

SAMPLE I.D.	DATE	TIME	COMP.	GRAB	MATRIX				OTHER	METHOD PRESERVED				OTHER	NO. OF CONTAINERS	CONTAINER SIZE	ANALYSIS REQUEST: (CHECK APPROPRIATE BOX)										OTHER			REMARKS
					H2O	SOIL	AIR	SLUDGE		HCl	HNO3	H2SO4	NONE				BTEX 602 ☐ 8020 ☐ WITH MTBE ☐	BTEX/GAS HYDROCARBONS PID/FID ☐ WITH MTBE	VOL 624/PPL ☐ 8240/TAL ☐ NBS (+15) ☐	PNA/PAH 8310 ☐ 8100 ☐ 610 ☐	SEMI-VOL 625/PPL ☐ 8270/TAL ☐ NBS (+25) ☐	TPH/IR 418.1 ☐ SM503 ☐ ___ ☐ ___ ☐ ___ ☐	TPH/GC 8015 Mod. GAS ☐ 8015 Mod DIESEL ☐	TCLP METALS ☐ VOL ☐ SEMI-VOL ☐ PEST ☐ HERB ☐	EP TOX METALS ☐ PESTICIDES ☐ HERBICIDES ☐	REACTIVITY ☐ CORROSIVITY ☐ IGNITABILITY ☐	TPH Leachate Method			
B-1 (15-16')	8-18-95			✓		✓								ICE													✓			
B-3 (25-26')	8-18-95			✓		✓								ICE													✓			
B-5 (17-18')	8-18-95			✓		✓								ICE													✓			
B-6 (15-16')	8-18-95			✓		✓								ICE													✓			

RELINQUISHED BY: (SIGNATURE)	DATE	TIME	RECEIVED BY: (SIGNATURE)	DATE	TIME
F. Wesley Root	8-28-95	6:00 PM			
RELINQUISHED BY: (SIGNATURE)	DATE	TIME	RECEIVED BY: (SIGNATURE)	DATE	TIME
			3°C INTACT		
RELINQUISHED BY: (SIGNATURE)	DATE	TIME	RECEIVED BY: (SIGNATURE)	DATE	TIME
			S. West	8/29/95	1015

BILL NO.:

LABORATORY: SPL - Houston

SHELL CONTACT: Neal Stidham PHONE: FAX:

TURN AROUND TIME (CHECK ONE)

7 DAYS ☐ (NORMAL) 14 DAYS ☐

48 HOURS ☐ OTHER ☒ per SPLC contract

THE LABORATORY MUST PROVIDE A COPY OF THIS CHAIN OF CUSTODY WITH INVOICE AND RESULTS

DISTRIBUTION: PINK Sampling Coordinator - WHITE & YELLOW Accompanies Shipment - WHITE Returned with Report

SPL HOUSTON ENVIRONMENTAL LABORATORY

SAMPLE LOGIN CHECKLIST

DATE: 8/29/95 TIME: ________ CLIENT NO. ________
LOT NO. ________ CONTRACT NO. ________

CLIENT SAMPLE NOS. ________

SPL SAMPLE NOS.: 9508A90

		YES	NO
1.	Is a Chain-of-Custody form present?	✓	
2.	Is the COC properly completed?	✓	
	If no, describe what is incomplete:		
	If no, has the client been contacted about it? (Attach subsequent documentation from client about the situation)		
3.	Is airbill/packing list/bill of lading with shipment? If yes, ID#: by Fed Ex	✓	
4.	Is a USEPA Traffic Report present?		✓
5.	Is a USEPA SAS Packing List present?		✓
6.	Are custody seals present on the package?	✓	
	If yes, were they intact upon receipt?	✓	
7.	Are all samples tagged or labeled?	✓	
	Do the sample tags/labels match the COC?	✓	
	If no, has the client been contacted about it? (Attach subsequent documentation from client about the situation)		
8.	Do all shipping documents agree?	✓	
	If no, describe what is in nonconformity:		

9. Condition/temperature of shipping container: 3°C INTACT
10. Condition/temperature of sample bottles: Good
11. Sample Disposal?: SPL disposal ✓ Return to client ____

NOTES (reference item number if applicable): ________

ATTEST: ________ DATE: 8/29/95
DELIVERED FOR RESOLUTION: REC'D ________ DATE: ________
RESOLVED: ________ DATE: ________

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

Certificate of Analysis No. H9-9508A19-01

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 09/07/95

PROJECT: Hobbs Station
SITE: Lea County, New Mexico
SAMPLED BY: Shell Pipe Line
SAMPLE ID: B-1 2-3'

PROJECT NO: H 16157
MATRIX: SOIL
DATE SAMPLED: 08/18/95 08:50:00
DATE RECEIVED: 08/26/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
BENZENE	480	50 P	µg/Kg
TOLUENE	210	50 P	µg/Kg
ETHYLBENZENE	700	50 P	µg/Kg
TOTAL XYLENE	2200	50 P	µg/Kg
TOTAL VOLATILE AROMATIC HYDROCARBONS	3590		µg/Kg

Surrogate	**% Recovery**
1,4-Difluorobenzene	97
4-Bromofluorobenzene	148

METHOD 8020***
Analyzed by: KA
Date: 08/29/95

(P) - Practical Quantitation Limit

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 17th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

Certificate of Analysis No. H9-9508A19-02

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 09/07/95

PROJECT: Hobbs Station
SITE: Lea County, New Mexico
SAMPLED BY: Shell Pipe Line
SAMPLE ID: B-1 5-6'

PROJECT NO: H 16157
MATRIX: SOIL
DATE SAMPLED: 08/18/95 09:00:00
DATE RECEIVED: 08/26/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
BENZENE	36	10 P	µg/Kg
TOLUENE	110	10 P	µg/Kg
ETHYLBENZENE	100	10 P	µg/Kg
TOTAL XYLENE	1200	10 P	µg/Kg
TOTAL VOLATILE AROMATIC HYDROCARBONS	1446		µg/Kg
Surrogate	**% Recovery**		
1,4-Difluorobenzene	99		
4-Bromofluorobenzene	CI		

METHOD 8020***
Analyzed by: KA
Date: 08/29/95

Synthetic Precip. Leaching Procedure	08/30/95		

METHOD 1312 ***
Analyzed by: CM
Date: 08/30/95

Petroleum extractables	ND	1	mg/L

METHOD 418.1*
Analyzed by: LD
Date: 09/05/95 14:30:00

(P) - Practical Quantitation Limit CI - Coeluting interference.
ND - Not detected.

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 17th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

Certificate of Analysis No. H9-9508A19-03

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 09/07/95

PROJECT: Hobbs Station
SITE: Lea County, New Mexico
SAMPLED BY: Shell Pipe Line
SAMPLE ID: B-1 10-11'

PROJECT NO: H 16157
MATRIX: SOIL
DATE SAMPLED: 08/18/95 09:20:00
DATE RECEIVED: 08/26/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
BENZENE	ND	10 P	μg/Kg
TOLUENE	37	10 P	μg/Kg
ETHYLBENZENE	32	10 P	μg/Kg
TOTAL XYLENE	620	10 P	μg/Kg
TOTAL VOLATILE AROMATIC HYDROCARBONS	689		μg/Kg
Surrogate	**% Recovery**		
1,4-Difluorobenzene	110		
4-Bromofluorobenzene	CI		
METHOD 8020***			
Analyzed by: KA			
Date: 08/29/95			
Synthetic Precip. Leaching Procedure	08/30/95		
METHOD 1312 ***			
Analyzed by: CM			
Date: 08/30/95			
Petroleum extractables	ND	1	mg/L
METHOD 418.1*			
Analyzed by: LD			
Date: 09/05/95 14:30:00			

ND - Not detected.
CI - Coeluting interference.
(P) - Practical Quantitation Limit

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 17th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

SPL®

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

Certificate of Analysis No. H9-9508A19-04

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 09/07/95

PROJECT: Hobbs Station
SITE: Lea County, New Mexico
SAMPLED BY: Shell Pipe Line
SAMPLE ID: B-2 2-3'

PROJECT NO: H 16157
MATRIX: SOIL
DATE SAMPLED: 08/18/95 15:15:00
DATE RECEIVED: 08/26/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
BENZENE	400	50 P	µg/Kg
TOLUENE	180	50 P	µg/Kg
ETHYLBENZENE	130	50 P	µg/Kg
TOTAL XYLENE	1500	50 P	µg/Kg
TOTAL VOLATILE AROMATIC HYDROCARBONS	2210		µg/Kg

Surrogate	**% Recovery**
1,4-Difluorobenzene	106
4-Bromofluorobenzene	135

METHOD 8020***
Analyzed by: KA
Date: 08/29/95

(P) - Practical Quantitation Limit

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 17th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

Certificate of Analysis No. H9-9508A19-05

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 09/07/95

PROJECT: Hobbs Station
SITE: Lea County, New Mexico
SAMPLED BY: Shell Pipe Line
SAMPLE ID: B-2 5-6'

PROJECT NO: H 16157
MATRIX: SOIL
DATE SAMPLED: 08/18/95 15:25:00
DATE RECEIVED: 08/26/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
BENZENE	120	10 P	µg/Kg
TOLUENE	69	10 P	µg/Kg
ETHYLBENZENE	46	10 P	µg/Kg
TOTAL XYLENE	650	10 P	µg/Kg
TOTAL VOLATILE AROMATIC HYDROCARBONS	885		µg/Kg

Surrogate	**% Recovery**
1,4-Difluorobenzene	100
4-Bromofluorobenzene	CI

METHOD 8020***
Analyzed by: KA
Date: 08/29/95

Synthetic Precip. Leaching Procedure	08/30/95		

METHOD 1312 ***
Analyzed by: CM
Date: 08/30/95

Petroleum extractables	ND	1	mg/L

METHOD 418.1*
Analyzed by: LD
Date: 09/05/95 14:30:00

(P) - Practical Quantitation Limit CI - Coeluting interference.
ND - Not detected.

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 17th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

Certificate of Analysis No. H9-9508A19-06

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 09/07/95

PROJECT: Hobbs Station
SITE: Lea County, New Mexico
SAMPLED BY: Shell Pipe Line
SAMPLE ID: B-2 10-11'

PROJECT NO: H 16157
MATRIX: SOIL
DATE SAMPLED: 08/18/95 15:40:00
DATE RECEIVED: 08/26/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
BENZENE	ND	1 P	µg/Kg
TOLUENE	ND	1 P	µg/Kg
ETHYLBENZENE	ND	1 P	µg/Kg
TOTAL XYLENE	2	1 P	µg/Kg
TOTAL VOLATILE AROMATIC HYDROCARBONS	2		µg/Kg

Surrogate	**% Recovery**
1,4-Difluorobenzene	103
4-Bromofluorobenzene	76

METHOD 8020***
Analyzed by: KA
Date: 08/29/95

ND - Not detected.

(P) - Practical Quantitation Limit

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 17th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

Certificate of Analysis No. H9-9508A19-07

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 09/07/95

PROJECT: Hobbs Station
SITE: Lea County, New Mexico
SAMPLED BY: Shell Pipe Line
SAMPLE ID: B-3 5-6'

PROJECT NO: H 16157
MATRIX: SOIL
DATE SAMPLED: 08/18/95 14:30:00
DATE RECEIVED: 08/26/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
BENZENE	ND	1 P	µg/Kg
TOLUENE	ND	1 P	µg/Kg
ETHYLBENZENE	ND	1 P	µg/Kg
TOTAL XYLENE	1	1 P	µg/Kg
TOTAL VOLATILE AROMATIC HYDROCARBONS	1		µg/Kg

Surrogate	% Recovery
1,4-Difluorobenzene	109
4-Bromofluorobenzene	56

METHOD 8020***
Analyzed by: KA
Date: 08/29/95

ND - Not detected. (P) - Practical Quantitation Limit

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 17th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

QUALITY CONTROL DOCUMENTATION

** SPL BATCH QUALITY CONTROL REPORT **
METHOD 8020***

PAGE

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

Matrix: Soil
Units: μg/Kg

Batch Id: HP_0950828124800

LABORATORY CONTROL SAMPLE

SPIKE COMPOUNDS	Method Blank Result <2>	Spike Added <3>	Blank Spike Result <1>	Blank Spike Recovery %	QC Limits(**) (Mandatory) % Recovery Range
Benzene	ND	50	45	90.0	51 - 125
Toluene	ND	150	140	93.3	52 - 126
EthylBenzene	ND	50	49	98.0	53 - 125
O Xylene	ND	100	98	98.0	32 - 160
M & P Xylene	ND	200	200	100	32 - 160

MATRIX SPIKES

SPIKE COMPOUNDS	Sample Results <2>	Spike Added <3>	Matrix Spike Result <1>	Matrix Spike Recovery <4>	Matrix Spike Duplicate Result <1>	Matrix Spike Duplicate Recovery <5>	MS/MSD Relative % Difference	QC Limits(***) (Advisory) RPD Max.	QC Limits(***) (Advisory) Recovery Range
BENZENE	ND	50	48	96.0	50	100	4.08	33	47 - 143
TOLUENE	ND	150	140	93.3	140	93.3	0	35	46 - 148
ETHYLBENZENE	ND	50	48	96.0	50	100	4.08	40	32 - 151
O XYLENE	ND	100	94	94.0	96	96.0	2.11	24	18 - 144
M & P XYLENE	ND	100	110	110	110	110	0	23	25 - 139

Analyst: KA
Sequence Date: 08/28/95
SPL ID of sample spiked: 9508995-08A
Sample File ID: OO__082.TX0
Method Blank File ID:
Blank Spike File ID: OO__064.TX0
Matrix Spike File ID: OO__069.TX0
Matrix Spike Duplicate File ID: OO__070.TX0

* = Values Outside QC Range
NC = Not Calculated (Sample exceeds spike by factor of 4 or more)
ND = Not Detected/Below Detection Limit
% Recovery = [(<1> - <2>) / <3>] x 100
LCS % Recovery = (<1> / <3>) x 100
Relative Percent Difference = |(<4> - <5> | / [(<4> + <5>) x 0.5] x 100
(**) = Source: SPL-Houston Historical Data
(***) = Source:

<u>SAMPLES IN BATCH(SPL ID):</u>

9508897-05A 9508897-06A 9508995-01A 9508995-02A
9508995-03A 9508995-04A 9508995-05A 9508995-06A
9508995-07A 9508995-08A 9508995-09A 9508995-10A
9508A19-06A 9508A19-07A 9508A19-02A 9508A19-03A
9508A19-05A 9508A19-01A 9508A19-04A 9508897-01A

Cynthia Schreiner, QC Officer

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

** SPL QUALITY CONTROL REPORT **

Matrix: Aqueous

Reported on: 09/05/95
Analyzed on: 09/05/95
Analyst: LD

This sample was randomly selected for use in the SPL quality control program. Samples chosen are fortified with a known concentration in duplicate. The results are as follows:

Petroleum extractables
METHOD 418.1*

SPL Sample ID Number	Blank Value mg/l	Amt Added mg/l	Matrix Spike Recovery %	Matrix Spike Duplicate Recovery %	Relative Percent Difference %	QC Limits Recovery	RPD Max.
BLANK	ND	4.0	92.5	95.0	2.7	82. - 112	9.8

9509051200RS -9509020

Samples in batch:

9508A19-02B 9508A19-03B 9508A19-05B 9508A90-01B
9508A90-02B 9508A90-03B 9508A90-04B 9508B22-01B
9508B22-02B 9508B22-03B 9508B22-04B 9508B22-05B
9508B33-01B 9508B33-02B 9508B33-03B 9508B33-04B
9508B33-05B 9508B33-06B 9508B53-01B 9508B85-02B

COMMENTS:

SPL, Incorporated

Cynthia Schreiner, QC Officer

CHAIN OF CUSTODY
AND
SAMPLE RECEIPT CHECKLIST

9508A19 B3

SHELL OIL COMPANY
RETAIL ENVIRONMENTAL ENGINEERING

CHAIN OF CUSTODY RECORD NO. H 16157

Date: 8-25-95
Page 1 of 1

Hobbs Station
SITE ADDRESS: Shell Pipe Line Corp.
Lea County, New Mexico
WIC #: ESC No. 124
CONSULTANT NAME & ADDRESS: Environmental Spill Control
1203 W. Dunnam, P.O. 5890, Hobbs, NM 88241
CONSULTANT CONTACT: F. Wesley Root
PHONE: 505-392-6167 FAX: 505-397-3085
SAMPLED BY: F. Wesley Root

CHECK ONE BOX ONLY CT/DT
QUARTERLY MONITORING ☐ 5461
SITE INVESTIGATION ☒ 5441
SOIL FOR DISPOSAL ☐ 5442
WATER FOR DISPOSAL ☐ 5443
AIR SAMPLER - SYS O+M ☐ 5452
WATER SAMPLE - SYS O+M ☐ 5453
OTHER ______ ☐

SAMPLE I.D.	DATE	TIME	COMP.	GRAB	MATRIX H2O	MATRIX SOIL	MATRIX AIR	MATRIX SLUDGE	OTHER	METHOD PRESERVED HCl	METHOD PRESERVED HNO3	METHOD PRESERVED H2SO4	METHOD PRESERVED NONE	OTHER	NO. OF CONTAINERS	CONTAINER SIZE	BTEX 602 ☐ 8020 ☒ WITH MTBE ☐	BTEX/GAS HYDROCARBONS PID/FID ☐ WITH MTBE	VOL 624/PPL ☐ 8240/TAL ☐ NBS (+15) ☐	PNA/PAH 8310 ☐ 8100 ☐ 610 ☐	SEMI - VOL 625/PPL ☐ 8270/TAL ☐ NBS (+25) ☐	TPH/IR 418.1 ☐ SM503 ☐ ___ ☐ ___ ☐ ___ ☐	TPH/GC 8015 Mod. GAS ☐ 8015 Mod DIESEL ☐	TCLP METALS ☐ VOL ☐ SEMI-VOL ☐ PEST ☐ HERB ☐	EP TOX METALS ☐ PESTICIDES ☐ HERBICIDES ☐	REACTIVITY ☐ CORROSIVITY ☐ IGNITABILITY ☐	OTHER	OTHER	OTHER	REMARKS
B-1 2-~~3~~'	8-18-95	8:50		✓		✓								ICE			✓													
B-1 5-6'	8-18-95	9:00		✓		✓								ICE			✓													
B-1 10-11'	8-18-95	9:20		✓		✓								ICE			✓													
B-2 2-3'	8-18-95	3:15		✓		✓								ICE			✓													
B-2 5-6'	8-18-95	3:25		✓		✓								ICE			✓													
B-2 10-11'	8-18-95	3:40		✓		✓								ICE			✓													
B-3 5-6'	8-18-95	2:30		✓		✓								ICE			✓													

RELINQUISHED BY: (SIGNATURE)	DATE	TIME	RECEIVED BY: (SIGNATURE)	DATE	TIME
F. Wesley Root	8-25-95	6:00 PM			
			E. Brown	8/26/95	0945

BILL NO.: ______
LABORATORY: SPL — Houston
SHELL CONTACT: Nac Stidham PHONE: ______ FAX: ______

TURN AROUND TIME (CHECK ONE)
7 DAYS ☐ (NORMAL)
48 HOURS ☐
14 DAYS ☐
OTHER ☒ per SPLC contract

intact 4 C

THE LABORATORY MUST PROVIDE A COPY OF THIS CHAIN OF CUSTODY WITH INVOICE AND RESULTS

DISTRIBUTION: PINK Sampling Coordinator - WHITE & YELLOW Accompanies Shipment - WHITE Returned with Report

SPL HOUSTON ENVIRONMENTAL LABORATORY

SAMPLE LOGIN CHECKLIST

DATE: 8/26/95 TIME: 0945 CLIENT NO. ____

LOT NO. ____ CONTRACT NO. ____

CLIENT SAMPLE NOS. ____

SPL SAMPLE NOS.: 9508A19

		YES	NO
1.	Is a Chain-of-Custody form present?	✓	
2.	Is the COC properly completed?	✓	
	If no, describe what is incomplete:		
	If no, has the client been contacted about it? (Attach subsequent documentation from client about the situation)		
3.	Is airbill/packing list/bill of lading with shipment?	✓	
	If yes, ID#: FedEx		
4.	Is a USEPA Traffic Report present?		✓
5.	Is a USEPA SAS Packing List present?		✓
6.	Are custody seals present on the package?	✓	
	If yes, were they intact upon receipt?	✓	
7.	Are all samples tagged or labeled?	✓	
	Do the sample tags/labels match the COC?	✓	
	If no, has the client been contacted about it? (Attach subsequent documentation from client about the situation)		
8.	Do all shipping documents agree?	✓	
	If no, describe what is in nonconformity:		

9. Condition/temperature of shipping container: intact, 4°C
10. Condition/temperature of sample bottles: Good
11. Sample Disposal?: SPL disposal ✓ Return to client ____

NOTES (reference item number if applicable): ____

ATTEST: E. Brown DATE: 8/26/95

DELIVERED FOR RESOLUTION: REC'D ____ DATE: ____

RESOLVED: ____ DATE: ____

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

Certificate of Analysis No. H9-9511198-01

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 12/06/95

PROJECT: ESC No. 124
SITE: Lea County, NM
SAMPLED BY: Environmental Spill Control
SAMPLE ID: B-1 (0-1)

PROJECT NO: H 23512
MATRIX: SOIL
DATE SAMPLED: 11/03/95 10:00:00
DATE RECEIVED: 11/04/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
Synthetic Precip. Leaching Procedure METHOD 1312 *** Analyzed by: CM Date: 11/06/95	11/06/95		
Petroleum extractables METHOD 418.1* Analyzed by: SW Date: 11/08/95 13:00:00	ND	0.5	mg/L

ND - Not detected.

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 18th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

SPL®

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

Certificate of Analysis No. H9-9511198-02

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 12/06/95

PROJECT: ESC No. 124
SITE: Lea County, NM
SAMPLED BY: Environmental Spill Control
SAMPLE ID: B-1 (2-3)

PROJECT NO: H 23512
MATRIX: SOIL
DATE SAMPLED: 11/01/95 12:55:00
DATE RECEIVED: 11/04/95

ANALYTICAL DATA

PARAMETER	**RESULTS**	**DETECTION LIMIT**	**UNITS**
MTBE, SPLP	ND	1 P	ug/L
BENZENE, SPLP	ND	1 P	ug/L
TOLUENE, SPLP	ND	1 P	ug/L
ETHYLBENZENE, SPLP	7	1 P	ug/L
TOTAL XYLENE, SPLP	4	1 P	ug/L
SPLP BTEX	11		ug/L
Surrogate	**% Recovery**		
1,4-Difluorobenzene	101		
4-Bromofluorobenzene	113		
METHOD 1312/8020***			
Analyzed by: DAO			
Date: 11/08/95			
Synthetic Precip. Leaching Procedure	11/06/95		
METHOD 1312 ***			
Analyzed by: CM			
Date: 11/06/95			
Petroleum extractables	0.5	0.5	mg/L
METHOD 418.1*			
Analyzed by: SW			
Date: 11/08/95 13:00:00			
Synthetic Precipitation Leaching - ZHE	11/07/95		
METHOD 1312 ***			
Analyzed by: CM			
Date: 11/07/95			

ND - Not detected. (P) - Practical Quantitation Limit

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 18th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

SPL®

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

Certificate of Analysis No. H9-9511198-03

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 12/06/95

PROJECT: ESC No. 124
SITE: Lea County, NM
SAMPLED BY: Environmental Spill Control
SAMPLE ID: B-2 (0-1)

PROJECT NO: H 23512
MATRIX: SOIL
DATE SAMPLED: 11/03/95 09:45:00
DATE RECEIVED: 11/04/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
Synthetic Precip. Leaching Procedure METHOD 1312 *** Analyzed by: CM Date: 11/06/95	11/06/95		
Petroleum extractables METHOD 418.1* Analyzed by: SW Date: 11/08/95 13:00:00	ND	0.5	mg/L

ND - Not detected.

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 18th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

Certificate of Analysis No. H9-9511198-04

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 12/06/95

PROJECT: ESC No. 124
SITE: Lea County, NM
SAMPLED BY: Environmental Spill Control
SAMPLE ID: B-2 (2-3)

PROJECT NO: H 23512
MATRIX: SOIL
DATE SAMPLED: 11/01/95 10:15:00
DATE RECEIVED: 11/04/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
MTBE, SPLP	ND	1 P	ug/L
BENZENE, SPLP	ND	1 P	ug/L
TOLUENE, SPLP	ND	1 P	ug/L
ETHYLBENZENE, SPLP	ND	1 P	ug/L
TOTAL XYLENE, SPLP	ND	1 P	ug/L
SPLP BTEX	ND		ug/L
Surrogate	**% Recovery**		
1,4-Difluorobenzene	95		
4-Bromofluorobenzene	99		
METHOD 1312/8020***			
Analyzed by: DAO			
Date: 11/08/95			
Synthetic Precip. Leaching Procedure	11/06/95		
METHOD 1312 ***			
Analyzed by: CM			
Date: 11/06/95			
Petroleum extractables	ND	0.5	mg/L
METHOD 418.1*			
Analyzed by: SW			
Date: 11/08/95 13:00:00			
Synthetic Precipitation Leaching - ZHE	11/07/95		
METHOD 1312 ***			
Analyzed by: CM			
Date: 11/07/95			

ND - Not detected. (P) - Practical Quantitation Limit

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 18th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

Certificate of Analysis No. H9-9511198-05

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 12/06/95

PROJECT: ESC No. 124
SITE: Lea County, NM
SAMPLED BY: Environmental Spill Control
SAMPLE ID: B-3 (2-3)

PROJECT NO: H 23512
MATRIX: SOIL
DATE SAMPLED: 11/01/95 13:15:00
DATE RECEIVED: 11/04/95

ANALYTICAL DATA

PARAMETER	**RESULTS**	**DETECTION LIMIT**	**UNITS**
MTBE, SPLP	ND	1 P	ug/L
BENZENE, SPLP	ND	1 P	ug/L
TOLUENE, SPLP	ND	1 P	ug/L
ETHYLBENZENE, SPLP	39	1 P	ug/L
TOTAL XYLENE, SPLP	4	1 P	ug/L
SPLP BTEX	43		ug/L
Surrogate	**% Recovery**		
1,4-Difluorobenzene	94		
4-Bromofluorobenzene	117		
METHOD 1312/8020*** Analyzed by: DAO Date: 11/08/95			
Synthetic Precip. Leaching Procedure METHOD 1312 *** Analyzed by: CM Date: 11/06/95	11/06/95		
Petroleum extractables METHOD 418.1* Analyzed by: SW Date: 11/08/95 13:00:00	ND	0.5	mg/L
Synthetic Precipitation Leaching - ZHE METHOD 1312 *** Analyzed by: CM Date: 11/07/95	11/07/95		

ND - Not detected. (P) - Practical Quantitation Limit

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 18th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

HOUSTON LABORATORY
8880 INTERCHANGE DRIVE
HOUSTON, TEXAS 77054
PHONE (713) 660-0901

Certificate of Analysis No. H9-9511198-06

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 12/06/95

PROJECT: ESC No. 124
SITE: Lea County, NM
SAMPLED BY: Environmental Spill Control
SAMPLE ID: B-3 (0-1)

PROJECT NO: H 23512
MATRIX: SOIL
DATE SAMPLED: 11/03/95 10:10:00
DATE RECEIVED: 11/04/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
Synthetic Precip. Leaching Procedure METHOD 1312 *** Analyzed by: CM Date: 11/06/95	11/06/95		
Petroleum extractables METHOD 418.1* Analyzed by: SW Date: 11/08/95 13:00:00	1	0.5	mg/L

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 18th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

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Certificate of Analysis No. H9-9511198-07

Shell Pipeline Corporation
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Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 12/06/95

PROJECT: ESC No. 124
SITE: Lea County, NM
SAMPLED BY: Environmental Spill Control
SAMPLE ID: B-3 (5-6)

PROJECT NO: H 23512
MATRIX: SOIL
DATE SAMPLED: 11/01/95 13:30:00
DATE RECEIVED: 11/04/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
MTBE, SPLP	ND	1 P	ug/L
BENZENE, SPLP	ND	1 P	ug/L
TOLUENE, SPLP	ND	1 P	ug/L
ETHYLBENZENE, SPLP	18	1 P	ug/L
TOTAL XYLENE, SPLP	2	1 P	ug/L
SPLP BTEX	20		ug/L

Surrogate	**% Recovery**
1,4-Difluorobenzene	94
4-Bromofluorobenzene	107

METHOD 1312/8020***
Analyzed by: DAO
Date: 11/08/95

Synthetic Precip. Leaching Procedure	11/06/95		

METHOD 1312 ***
Analyzed by: CM
Date: 11/06/95

Petroleum extractables	ND	0.5	mg/L

METHOD 418.1*
Analyzed by: SW
Date: 11/08/95 13:00:00

Synthetic Precipitation Leaching - ZHE	11/07/95		

METHOD 1312 ***
Analyzed by: CM
Date: 11/07/95

ND - Not detected.
(P) - Practical Quantitation Limit

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 18th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

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Certificate of Analysis No. H9-9511198-08

Shell Pipeline Corporation
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ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 12/06/95

PROJECT: ESC No. 124
SITE: Lea County, NM
SAMPLED BY: Environmental Spill Control
SAMPLE ID: B-5 (0-1)

PROJECT NO: H 23512
MATRIX: SOIL
DATE SAMPLED: 11/03/95 10:15:00
DATE RECEIVED: 11/04/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
Synthetic Precip. Leaching Procedure METHOD 1312 *** Analyzed by: CM Date: 11/06/95	11/06/95		
Petroleum extractables METHOD 418.1* Analyzed by: SW Date: 11/08/95 13:00:00	ND	0.5	mg/L

ND - Not detected.

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 18th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

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Certificate of Analysis No. H9-9511198-09

Shell Pipeline Corporation
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ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 12/06/95

PROJECT: ESC No. 124
SITE: Lea County, NM
SAMPLED BY: Environmental Spill Control
SAMPLE ID: B-5 (2-3)

PROJECT NO: H 23512
MATRIX: SOIL
DATE SAMPLED: 11/01/95 12:40:00
DATE RECEIVED: 11/04/95

ANALYTICAL DATA

PARAMETER	**RESULTS**	**DETECTION LIMIT**	**UNITS**
Synthetic Precip. Leaching Procedure METHOD 1312 *** Analyzed by: CM Date: 11/06/95	11/06/95		
Petroleum extractables METHOD 418.1* Analyzed by: SW Date: 11/08/95 13:00:00	ND	0.5	mg/L

ND - Not detected.

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 18th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

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Certificate of Analysis No. H9-9511198-10

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 12/06/95

PROJECT: ESC No. 124
SITE: Lea County, NM
SAMPLED BY: Environmental Spill Control
SAMPLE ID: B-5 (5-6)

PROJECT NO: H 23512
MATRIX: SOIL
DATE SAMPLED: 11/01/95 12:45:00
DATE RECEIVED: 11/04/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
Synthetic Precip. Leaching Procedure METHOD 1312 *** Analyzed by: CM Date: 11/06/95	11/06/95		
Petroleum extractables METHOD 418.1* Analyzed by: SW Date: 11/08/95 13:00:00	ND	0.5	mg/L

ND - Not detected.

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 18th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

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Certificate of Analysis No. H9-9511198-11

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 12/06/95

PROJECT: ESC No. 124
SITE: Lea County, NM
SAMPLED BY: Environmental Spill Control
SAMPLE ID: B-6 (0-1)

PROJECT NO: H 23512
MATRIX: SOIL
DATE SAMPLED: 11/03/95 09:30:00
DATE RECEIVED: 11/04/95

ANALYTICAL DATA

PARAMETER	**RESULTS**	**DETECTION LIMIT**	**UNITS**
Synthetic Precip. Leaching Procedure METHOD 1312 *** Analyzed by: CM Date: 11/06/95	11/06/95		
Petroleum extractables METHOD 418.1* Analyzed by: SW Date: 11/08/95 13:00:00	1	0.5	mg/L

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 18th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

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Certificate of Analysis No. H9-9511198-12

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ATTN: Neal Stidham

P.O.# MESA-CAO-B-1312-01-PX-4204-NS
DATE: 12/06/95

PROJECT: ESC No. 124
SITE: Lea County, NM
SAMPLED BY: Environmental Spill Control
SAMPLE ID: B-6 (2-3)

PROJECT NO: H 23512
MATRIX: SOIL
DATE SAMPLED: 11/01/95 11:15:00
DATE RECEIVED: 11/04/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
Synthetic Precip. Leaching Procedure METHOD 1312 *** Analyzed by: CM Date: 11/06/95	11/06/95		
Petroleum extractables METHOD 418.1* Analyzed by: SW Date: 11/08/95 13:00:00	0.8	0.5	mg/L

Notes: *Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 18th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

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PHONE (713) 660-0901

Certificate of Analysis No. H9-9511198-13

Shell Pipeline Corporation
P.O.Box 2648
Houston, TX 77252
ATTN: Neal Stidham

P.O.#
MESA-CAO-B-1312-01-PX-4204-NS
DATE: 12/06/95

PROJECT: ESC No. 124
SITE: Lea County, NM
SAMPLED BY: Environmental Spill Control
SAMPLE ID: B-6 (5-6)

PROJECT NO: H 23512
MATRIX: SOIL
DATE SAMPLED: 11/01/95 11:45:00
DATE RECEIVED: 11/04/95

ANALYTICAL DATA

PARAMETER	RESULTS	DETECTION LIMIT	UNITS
Synthetic Precip. Leaching Procedure METHOD 1312 *** Analyzed by: CM Date: 11/06/95	11/06/95		
Petroleum extractables METHOD 418.1* Analyzed by: SW Date: 11/08/95 13:00:00	ND	0.5	mg/L

ND - Not detected.

Notes: .*Ref: Methods for Chemical Analysis of Water and Wastes, 1983, EPA
**Ref: Standard Methods for Examination of Water & Wastewater, 18th ed.
***Ref: Test Methods for Evaluating Solid Waste, EPA SW846, 3rd Ed.

QUALITY ASSURANCE: These analyses are performed in accordance with EPA guidelines for quality assurance.

METHOD 1312

SYNTHETIC PRECIPITATION LEACHING PROCEDURE

1.0 SCOPE AND APPLICATION

1.1 Method 1312 is designed to determine the mobility of both organic and inorganic analytes present in liquids, soils, and wastes.

2.0 SUMMARY OF METHOD

2.1 For liquid samples (i.e., those containing less than 0.5 % dry solid material), the sample, after filtration through a 0.6 to 0.8 µm glass fiber filter, is defined as the 1312 extract.

2.2 For samples containing greater than 0.5 % solids, the liquid phase, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. If the sample is a waste or wastewater, the extraction fluid employed is a pH 4.2 solution. A special extractor vessel is used when testing for volatile analytes (see Table 1 for a list of volatile compounds). Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

2.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 INTERFERENCES

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 APPARATUS AND MATERIALS

4.1 Agitation apparatus: The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion (see Figure 1) at 30 ± 2 rpm. Suitable devices known to EPA are identified in Table 2.

4.2 Extraction Vessels

4.2.1 Zero Headspace Extraction Vessel (ZHE). This device is for use only when the sample is being tested for the mobility of volatile analytes (i.e., those listed in Table 1). The ZHE (depicted in Figure 2) allows for liquid/solid separation within the device and effectively precludes headspace. This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel (see Step 4.3.1). These vessels shall have an internal volume of 500-600 mL and be equipped to accommodate a 90-110 mm filter. The devices contain VITON®[1] O-rings which should be replaced frequently. Suitable ZHE devices known to EPA are identified in Table 3.

For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 15 psig or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced. If this does not solve the problem, the ZHE is unacceptable for 1312 analyses and the manufacturer should be contacted.

The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psig, allow it to stand unattended for 1 hour, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psig, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer should be contacted.

Some ZHEs use gas pressure to actuate the ZHE piston, while others use mechanical pressure (see Table 3). Whereas the volatiles procedure (see Step 7.3) refers to pounds-per-square-inch (psig), for the mechanically actuated piston, the pressure applied is measured in torque-inch-pounds. Refer to the manufacturer's instructions as to the proper conversion.

4.2.2 Bottle Extraction Vessel. When the sample is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed. Headspace is allowed in this vessel.

The extraction bottles may be constructed from various materials, depending on the analytes to be analyzed and the nature of the waste (see Step 4.3.3). It is recommended that borosilicate glass bottles be used instead of other types of glass, especially when inorganics are of concern. Plastic bottles, other than polytetrafluoroethylene, shall not be used if organics are to be investigated. Bottles are available from a number of laboratory suppliers. When this type of extraction vessel is used, the filtration device discussed in Step 4.3.2 is used for initial liquid/solid separation and final extract filtration.

4.3 Filtration Devices: It is recommended that all filtrations be performed in a hood.

4.3.1 Zero-Headspace Extraction Vessel (ZHE): When the sample is evaluated for volatiles, the zero-headspace extraction vessel described in Step 4.2.1 is used for filtration. The device shall be capable of

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[1] VITON® is a trademark of Du Pont.

supporting and keeping in place the glass fiber filter and be able to withstand the pressure needed to accomplish separation (50 psig).

> NOTE: When it is suspected that the glass fiber filter has been ruptured, an in-line glass fiber filter may be used to filter the material within the ZHE.

4.3.2 Filter Holder: When the sample is evaluated for other than volatile analytes, a filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation may be used. Suitable filter holders range from simple vacuum units to relatively complex systems capable of exerting pressures of up to 50 psig or more. The type of filter holder used depends on the properties of the material to be filtered (see Step 4.3.3). These devices shall have a minimum internal volume of 300 mL and be equipped to accommodate a minimum filter size of 47 mm (filter holders having an internal capacity of 1.5 L or greater, and equipped to accommodate a 142 mm diameter filter, are recommended). Vacuum filtration can only be used for wastes with low solids content (<10 %) and for highly granular, liquid-containing wastes. All other types of wastes should be filtered using positive pressure filtration. Suitable filter holders known to EPA are listed in Table 4.

4.3.3 Materials of Construction: Extraction vessels and filtration devices shall be made of inert materials which will not leach or absorb sample components of interest. Glass, polytetrafluoroethylene (PTFE), or type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components. Devices made of high-density polyethylene (HDPE), polypropylene (PP), or polyvinyl chloride (PVC) may be used only when evaluating the mobility of metals. Borosilicate glass bottles are recommended for use over other types of glass bottles, especially when inorganics are analytes of concern.

4.4 Filters: Filters shall be made of borosilicate glass fiber, shall contain no binder materials, and shall have an effective pore size of 0.6 to 0.8-μm . Filters known to EPA which meet these specifications are identified in Table 5. Pre-filters must not be used. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1N nitric acid followed by three consecutive rinses with reagent water (a minimum of 1-L per rinse is recommended). Glass fiber filters are fragile and should be handled with care.

4.5 pH Meters: The meter should be accurate to ± 0.05 units at 25°C.

4.6 ZHE Extract Collection Devices: TEDLAR®[2] bags or glass, stainless steel or PTFE gas-tight syringes are used to collect the initial liquid phase and the final extract when using the ZHE device. These devices listed are recommended for use under the following conditions:

4.6.1 If a waste contains an aqueous liquid phase or if a waste does not contain a significant amount of nonaqueous liquid (i.e., <1 % of

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[2] TEDLAR® is a registered trademark of Du Pont.

total waste), the [illegible]EDLAR® bag or a 600 mL syringe sh[illegible]d be used to collect and combine the initial liquid and solid extract.

4.6.2 If a waste contains a significant amount of nonaqueous liquid in the initial liquid phase (i.e., >1 % of total waste), the syringe or the TEDLAR® bag may be used for both the initial solid/liquid separation and the final extract filtration. However, analysts should use one or the other, not both.

4.6.3 If the waste contains no initial liquid phase (is 100 % solid) or has no significant solid phase (is <0.5% solid), either the TEDLAR® bag or the syringe may be used. If the syringe is used, discard the first 5 mL of liquid expressed from the device. The remaining aliquots are used for analysis.

4.7 ZHE Extraction Fluid Transfer Devices: Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a positive displacement or peristaltic pump, a gas-tight syringe, pressure filtration unit (see Step 4.3.2), or other ZHE device).

4.8 Laboratory Balance: Any laboratory balance accurate to within ± 0.01 grams may be used (all weight measurements are to be within ± 0.1 grams).

4.9 Beaker or Erlenmeyer flask, glass, 500 mL.

4.10 Watchglass, appropriate diameter to cover beaker or Erlenmeyer flask.

4.11 Magnetic stirrer.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Reagent Water. Reagent water is defined as water in which an interferant is not observed at or above the method's detection limit of the analyte(s) of interest. For nonvolatile extractions, ASTM Type II water or equivalent meets the definition of reagent water. For volatile extractions, it is recommended that reagent water be generated by any of the following methods. Reagent water should be monitored periodically for impurities.

5.2.1 Reagent water for volatile extractions may be generated by passing tap water through a carbon filter bed containing about 500 grams of activated carbon (Calgon Corp., Filtrasorb-300 or equivalent).

5.2.2 A water purification system (Millipore Super-Q or equivalent) may also be used to generate reagent water for volatile extractions.

5.2.3 Reagent water for volatile extractions may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the water temperature at 90 ± 5 degrees C, bubble a contaminant-free inert gas (e.g. nitrogen) through the water for 1 hour. While still hot, transfer the water to a narrow mouth screw-cap bottle under zero-headspace and seal with a Teflon-lined septum and cap.

5.3 Sulfuric acid/nitric acid (60/40 weight percent mixture) H_2SO_4/HNO_3. Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid. If preferred, a more dilute H_2SO_4/HNO_3 acid mixture may be prepared and used in steps 5.4.1 and 5.4.2 making it easier to adjust the pH of the extraction fluids.

5.4 Extraction fluids.

5.4.1 Extraction fluid #1: This fluid is made by adding the 60/40 weight percent mixture of sulfuric and nitric acids (or a suitable dilution) to reagent water (Step 5.2) until the pH is 4.20 ± 0.05. The fluid is used to determine the leachability of soil from a site that is east of the Mississippi River, and the leachability of wastes and wastewaters.

<u>NOTE</u>: Solutions are unbuffered and exact pH may not be attained.

5.4.2 Extraction fluid #2: This fluid is made by adding the 60/40 weight percent mixture of sulfuric and nitric acids (or a suitable dilution) to reagent water (Step 5.2) until the pH is 5.00 ± 0.05. The fluid is used to determine the leachability of soil from a site that is west of the Mississippi River.

5.4.3 Extraction fluid #3: This fluid is reagent water (Step 5.2) and is used to determine cyanide and volatiles leachability.

<u>NOTE</u>: These extraction fluids should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

5.5 Analytical standards shall be prepared according to the appropriate analytical method.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples shall be collected using an appropriate sampling plan.

6.2 There may be requirements on the minimal size of the field sample depending upon the physical state or states of the waste and the analytes of concern. An aliquot is needed for the preliminary evaluations of the percent solids and the particle size. An aliquot may be needed to conduct the nonvolatile analyte extraction procedure. If volatile organics are of concern, another aliquot may be needed. Quality control measures may require additional aliquots. Further, it is always wise to collect more sample just in case something goes wrong with the initial attempt to conduct the test.

6.3 Preservatives shall not be added to samples before extraction.

6.4 Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

6.5 When the sample is to be evaluated for volatile analytes, care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon-lined septum capped vials and stored at 4°C. Samples should be opened only immediately prior to extraction).

6.6 1312 extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH < 2, unless precipitation occurs (see Step 7.2.14 if precipitation occurs). Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses. See Step 8.0 (Quality Control) for acceptable sample and extract holding times.

7.0 PROCEDURE

7.1 Preliminary Evaluations

Perform preliminary 1312 evaluations on a minimum 100 gram aliquot of sample. This aliquot may not actually undergo 1312 extraction. These preliminary evaluations include: (1) determination of the percent solids (Step 7.1.1); (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration (Step 7.1.2); and (3) determination of whether the solid portion of the waste requires particle size reduction (Step 7.1.3).

7.1.1 Preliminary determination of percent solids: Percent solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

7.1.1.1 If the sample will obviously yield no free liquid when subjected to pressure filtration (i.e., is 100% solid), weigh out a representative subsample (100 g minimum) and proceed to Step 7.1.3.

7.1.1.2 If the sample is liquid or multiphasic, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device discussed in Step 4.3.2, and is outlined in Steps 7.1.1.3 through 7.1.1.9.

7.1.1.3 Pre-weigh the filter and the container that will receive the filtrate.

7.1.1.4 Assemble filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

7.1.1.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight.

7.1.1.6 Allow slurries to stand to permit the solid phase to settle. Samples that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.1.1.7 Quantitatively transfer the sample to the filter holder (liquid and solid phases). Spread the sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

Gradually apply vacuum or gentle pressure of 1-10 psig, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psig, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10 psig increments to a maximum of 50 psig. After each incremental increase of 10 psig, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psig increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psig (i.e., filtration does not result in any additional filtrate within any 2-minute period), stop the filtration.

NOTE: If sample material (>1 % of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Step 7.1.1.5 to determine the weight of the sample that will be filtered.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.1.1.8 The material in the filter holder is defined as the solid phase of the sample, and the filtrate is defined as the liquid phase.

<u>NOTE</u>: Some samples, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid, but even after applying vacuum or pressure filtration, as outlined in Step 7.1.1.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.1.1.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see Step 7.1.1.3) from the total weight of the filtrate-filled container. Determine the weight of the solid phase of the sample by subtracting the weight of the liquid phase from the weight of the total sample, as determined in Step 7.1.1.5 or 7.1.1.7.

Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

$$\text{Percent solids} = \frac{\text{Weight of solid (Step 7.1.1.9)}}{\text{Total weight of waste (Step 7.1.1.5 or 7.1.1.7)}} \times 100$$

7.1.2 If the percent solids determined in Step 7.1.1.9 is equal to or greater than 0.5%, then proceed either to Step 7.1.3 to determine whether the solid material requires particle size reduction or to Step 7.1.2.1 if it is noticed that a small amount of the filtrate is entrained in wetting of the filter. If the percent solids determined in Step 7.1.1.9 is less than 0.5%, then proceed to Step 7.2.9 if the nonvolatile 1312 analysis is to be performed, and to Step 7.3 with a fresh portion of the waste if the volatile 1312 analysis is to be performed.

7.1.2.1 Remove the solid phase and filter from the filtration apparatus.

7.1.2.2 Dry the filter and solid phase at 100 ± 20°C until two successive weighings yield the same value within ± 1 %. Record the final weight.

<u>Caution</u>: The drying oven should be vented to a hood or other appropriate device to eliminate the possibility of fumes from the sample escaping into the laboratory. Care should be taken to ensure that the sample will not flash or violently react upon heating.

7.1.2.3 Calculate the percent dry solids as follows:

$$\text{Percent dry solids} = \frac{\text{(Weight of dry sample + filter) - tared weight of filter}}{\text{Initial weight of sample (Step 7.1.1.5 or 7.1.1.7)}} \times 100$$

7.1.2.4 If the percent dry solids is less than 0.5%, then proceed to Step 7.2.9 if the nonvolatile 1312 analysis is to be performed, and to Step 7.3 if the volatile 1312 analysis is to be performed. If the percent dry solids is greater than or equal to 0.5%, and if the nonvolatile 1312 analysis is to be performed, return to the beginning of this Step (7.1) and, with a fresh portion of sample, determine whether particle size reduction is necessary (Step 7.1.3).

7.1.3 Determination of whether the sample requires particle-size reduction (particle-size is reduced during this step): Using the solid portion of the sample, evaluate the solid for particle size. Particle-size reduction is required, unless the solid has a surface area per gram of material equal to or greater than 3.1 cm^2, or is smaller than 1 cm in its narrowest dimension (i.e., is capable of passing through a 9.5 mm (0.375 inch) standard sieve). If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the sample for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described above. If the solids are prepared for organic volatiles extraction, special precautions must be taken (see Step 7.3.6).

NOTE: Surface area criteria are meant for filamentous (e.g., paper, cloth, and similar) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet the criteria, sample-specific methods would need to be developed and employed to measure the surface area. Such methodology is currently not available.

7.1.4 Determination of appropriate extraction fluid:

7.1.4.1 For soils, if the sample is from a site that is east of the Mississippi River, extraction fluid #1 should be used. If the sample is from a site that is west of the Mississippi River, extraction fluid #2 should be used.

7.1.4.2 For wastes and wastewater, extraction fluid #1 should be used.

7.1.4.3 For cyanide-containing wastes and/or soils, extraction fluid #3 (reagent water) must be used because leaching of cyanide-containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.

7.1.5 If the aliquot of the sample used for the preliminary evaluation (Steps 7.1.1 - 7.1.4) was determined to be 100% solid at Step 7.1.1.1, then it can be used for the Step 7.2 extraction (assuming at least 100 grams remain), and the Step 7.3 extraction (assuming at least 25 grams remain). If the aliquot was subjected to the procedure in Step 7.1.1.7, then another aliquot shall be used for the volatile extraction procedure in Step 7.3. The aliquot of the waste subjected to the procedure in Step 7.1.1.7 might be appropriate for use for the Step 7.2 extraction if an adequate amount of solid (as determined by Step 7.1.1.9) was obtained. The amount of solid necessary is dependent upon whether a sufficient amount of extract will be produced to support the analyses. If an adequate amount of solid remains, proceed to Step 7.2.10 of the nonvolatile 1312 extraction.

7.2 Procedure When Volatiles Are Not Involved

A minimum sample size of 100 grams (solid and liquid phases) is recommended. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample (percent solids, See Step 7.1.1), whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of 1312 extract will be sufficient to support all of the analyses required. If the amount of extract generated by a single 1312 extraction will not be sufficient to perform all of the analyses, more than one extraction may be performed and the extracts from each combined and aliquoted for analysis.

7.2.1 If the sample will obviously yield no liquid when subjected to pressure filtration (i.e., is 100 % solid, see Step 7.1.1), weigh out a subsample of the sample (100 gram minimum) and proceed to Step 7.2.9.

7.2.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device described in Step 4.3.2 and is outlined in Steps 7.2.3 to 7.2.8.

7.2.3 Pre-weigh the container that will receive the filtrate.

7.2.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (see Step 4.4).

NOTE: Acid washed filters may be used for all nonvolatile extractions even when metals are not of concern.

7.2.5 Weigh out a subsample of the sample (100 gram minimum) and record the weight. If the waste contains <0.5 % dry solids (Step 7.1.2), the liquid portion of the waste, after filtration, is defined as the 1312 extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required of the 1312 extract. For wastes containing >0.5 % dry solids (Steps 7.1.1 or 7.1.2), use the percent solids information obtained in Step 7.1.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the 1312 extract.

7.2.6 Allow slurries to stand to permit the solid phase to settle. Samples that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If the sample is centrifuged, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.2.7 Quantitatively transfer the sample (liquid and solid phases) to the filter holder (see Step 4.3.2). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

Gradually apply vacuum or gentle pressure of 1-10 psig, until air or pressurizing gas moves through the filter. If this point if not reached under 10 psig, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psig increments to maximum of 50 psig. After each incremental increase of 10 psig, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psig increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psig (i.e., filtration does not result in any additional filtrate within a 2-minute period), stop the filtration.

> NOTE: If waste material (>1 % of the original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Step 7.2.5, to determine the weight of the waste sample that will be filtered.

> NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.2.8 The material in the filter holder is defined as the solid phase of the sample, and the filtrate is defined as the liquid phase. Weigh the filtrate. The liquid phase may now be either analyzed (see Step 7.2.12) or stored at 4°C until time of analysis.

> NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material which appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Step 7.2.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid, and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.2.9 If the sample contains <0.5% dry solids (see Step 7.1.2), proceed to Step 7.2.13. If the sample contains >0.5 % dry solids (see Step 7.1.1 or 7.1.2), and if particle-size reduction of the solid was needed in Step 7.1.3, proceed to Step 7.2.10. If the sample as received passes a 9.5 mm sieve, quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase, and proceed to Step 7.2.11.

7.2.10 Prepare the solid portion of the sample for extraction by crushing, cutting, or grinding the waste to a surface area or particle-size as described in Step 7.1.3. When the surface area or particle-size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

NOTE: Sieving of the waste is not normally required. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. If sieving is necessary, a Teflon-coated sieve should be used to avoid contamination of the sample.

7.2.11 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \%\ \text{solids (Step 7.1.1)} \times \text{weight of waste filtered (Step 7.2.5 or 7.2.7)}}{100}$$

Slowly add this amount of appropriate extraction fluid (see Step 7.1.4) to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape be used to ensure a tight seal), secure in rotary extractor device, and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction takes place) shall be maintained at 23 ± 2°C during the extraction period.

NOTE: As agitation continues, pressure may build up within the extractor bottle for some types of sample (e.g., limed or calcium carbonate-containing sample may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

7.2.12 Following the 18 ± 2 hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in Step 7.2.7. For final filtration of the 1312 extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed (see Step 4.4) if evaluating the mobility of metals.

7.2.13 Prepare the 1312 extract as follows:

7.2.13.1 If the sample contained no initial liquid phase, the filtered liquid material obtained from Step 7.2.12 is defined as the 1312 extract. Proceed to Step 7.2.14.

7.2.13.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Step 7.2.12 with the initial liquid phase of the sample obtained in Step 7.2.7. This combined liquid is defined as the 1312 extract. Proceed to Step 7.2.14.

7.2.13.3 If the initial liquid phase of the waste, as obtained from Step 7.2.7, is not or may not be compatible with the filtered liquid resulting from Step 7.2.12, do not combine these liquids. Analyze these liquids, collectively defined as the 1312 extract, and combine the results mathematically, as described in Step 7.2.14.

7.2.14 Following collection of the 1312 extract, the pH of the extract should be recorded. Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH < 2. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible. All other aliquots must be stored under refrigeration (4°C) until analyzed. The 1312 extract shall be prepared and analyzed according to appropriate analytical methods. 1312 extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to ± 0.5 %), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)\ (C_1) + (V_2)\ (C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phase (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.2.15 Compare the analyte concentrations in the 1312 extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality assurance requirements.

7.3 Procedure When Volatiles Are Involved

Use the ZHE device to obtain 1312 extract for analysis of volatile compounds only. Extract resulting from the use of the ZHE shall not be used to evaluate the mobility of non-volatile analytes (e.g., metals, pesticides, etc.).

The ZHE device has approximately a 500 mL internal capacity. The ZHE can thus accommodate a maximum of 25 grams of solid (defined as that fraction of a sample from which no additional liquid may be forced out by an applied pressure of 50 psig), due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase.

Charge the ZHE with sample only once and do not open the device until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted.

Do not allow the sample, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold (4°C) to minimize loss of volatiles.

7.3.1 Pre-weigh the (evacuated) filtrate collection container (see Step 4.6) and set aside. If using a TEDLAR® bag, express all liquid from the ZHE device into the bag, whether for the initial or final liquid/solid separation, and take an aliquot from the liquid in the bag for analysis. The containers listed in Step 4.6 are recommended for use under the conditions stated in Steps 4.6.1-4.6.3.

7.3.2 Place the ZHE piston within the body of the ZHE (it may be helpful first to moisten the piston O-rings slightly with extraction fluid). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample (based upon sample size requirements determined from Step 7.3, Step 7.1.1 and/or 7.1.2). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

7.3.3 If the sample is 100% solid (see Step 7.1.1), weigh out a subsample (25 gram maximum) of the waste, record weight, and proceed to Step 7.3.5.

7.3.4 If the sample contains <0.5% dry solids (Step 7.1.2), the liquid portion of waste, after filtration, is defined as the 1312 extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required. For samples containing ≥0.5% dry solids (Steps 7.1.1 and/or 7.1.2), use the percent solids information obtained in Step 7.1.1 to determine the optimum sample size to charge into the ZHE. The recommended sample size is as follows:

7.3.4.1 For samples containing <5% solids (see Step 7.1.1), weigh out a 500 gram subsample of waste and record the weight.

7.3.4.2 For wastes containing >5% solids (see Step 7.1.1), determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge ZHE} = \frac{25}{\text{percent solids (Step 7.1.1)}} \times 100$$

Weigh out a subsample of the waste of the appropriate size and record the weight.

7.3.5 If particle-size reduction of the solid portion of the sample was required in Step 7.1.3, proceed to Step 7.3.6. If particle-size reduction was not required in Step 7.1.3, proceed to Step 7.3.7.

7.3.6 Prepare the sample for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle size as described in Step 7.1.3.1. Wastes and appropriate reduction equipment should be refrigerated, if possible, to 4°C prior to particle-size reduction. The means used to effect particle-size reduction must not generate heat in and of itself. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

> <u>NOTE</u>: Sieving of the waste is not recommended due to the possibility that volatiles may be lost. The use of an appropriately graduated ruler is recommended as an acceptable alternative. Surface area requirements are meant for filamentous (<u>e.g.</u>, paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended.

When the surface area or particle-size has been appropriately altered, proceed to Step 7.3.7.

7.3.7 Waste slurries need not be allowed to stand to permit the solid phase to settle. Do not centrifuge samples prior to filtration.

7.3.8 Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens into the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extraction collection device to the top plate.

> <u>Note</u>: If sample material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Step 7.3.4 to determine the weight of the waste sample that will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psig (or more if necessary) to force all headspace slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. If the waste is 100 % solid (see Step 7.1.1), slowly increase the pressure to a maximum of 50 psig to force most of the headspace out of the device and proceed to Step 7.3.12.

7.3.9 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10 psig to force the liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psig increments to a maximum of 50 psig. After each incremental increase of 10 psig, if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psig increment. When liquid flow has ceased such that continued pressure filtration at 50 psig does not result in any additional filtrate within a 2-minute period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect and weigh the filtrate collection container.

<u>NOTE</u>: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.3.10 The material in the ZHE is defined as the solid phase of the sample and the filtrate is defined as the liquid phase.

<u>NOTE</u>: Some samples, such as oily wastes and some paint wastes, will obviously contain some material which appears to be a liquid. Even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid, and is carried through the 1312 extraction as a solid.

If the original waste contained <0.5 % dry solids (see Step 7.1.2), this filtrate is defined as the 1312 extract and is analyzed directly. Proceed to Step 7.3.15.

7.3.11 The liquid phase may now be either analyzed immediately (see Steps 7.3.13 through 7.3.15) or stored at 4°C under minimal headspace conditions until time of analysis. Determine the weight of extraction fluid #3 to add to the ZHE as follows:

$$\text{Weight of extraction fluid} = \frac{\begin{array}{c}20 \times \%\ \text{solids (Step 7.1.1)} \times \text{weight} \\ \text{of waste filtered (Step 7.3.4 or 7.3.8)}\end{array}}{100}$$

7.3.12 The following steps detail how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #3 is used in all cases (see Step 5.4.3).

7.3.12.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be preflushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.

7.3.12.2 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Pressurize the ZHE to 5-10 psig (if necessary) and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psig and check all ZHE fittings to ensure that they are closed.

7.3.12.3 Place the ZHE in the rotary extractor apparatus (if it is not already there) and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction occurs) shall be maintained at 23 ± 2°C during agitation.

7.3.13 Following the 18 ± 2 hour agitation period, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (i.e., no gas release observed), the ZHE is leaking. Check the ZHE for leaking as specified in Step 4.2.1, and perform the extraction again with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (i.e., TEDLAR® bag) holding the initial liquid phase of the waste. A separate filtrate collection container must be used if combining would create multiple phases, or there is not enough volume left within the filtrate collection container. Filter through the glass fiber filter, using the ZHE device as discussed in Step 7.3.9. All extracts shall be filtered and collected if the TEDLAR® bag is used, if the extract is multiphasic, or if the waste contained an initial liquid phase (see Steps 4.6 and 7.3.1).

NOTE: An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured

7.3.14 If the original sample contained no initial liquid phase, the filtered liquid material obtained from Step 7.3.13 is defined as the 1312 extract. If the sample contained an initial liquid phase, the filtered liquid material obtained from Step 7.3.13 and the initial liquid phase (Step 7.3.9) are collectively defined as the 1312 extract.

7.3.15 Following collection of the 1312 extract, immediately prepare the extract for analysis and store with minimal headspace at 4°C until analyzed. Analyze the 1312 extract according to the appropriate analytical methods. If the individual phases are to be analyzed separately (<u>i.e.</u>, are not miscible), determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume- weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)\ (C_1) + (V_2)\ (C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phases (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.3.16 Compare the analyte concentrations in the 1312 extract with the levels identified in the appropriate regulations. Refer to Step 8.0 for quality assurance requirements.

8.0 QUALITY CONTROL

8.1 A minimum of one blank (using the same extraction fluid as used for the samples) for every 20 extractions that have been conducted in an extraction vessel. Refer to Chapter One for additional quality control protocols.

8.2 A matrix spike shall be performed for each waste type (<u>e.g.</u>, wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data is being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. As a minimum, follow the matrix spike addition guidance provided in each analytical method.

8.2.1 Matrix spikes are to be added after filtration of the 1312 extract and before preservation. Matrix spikes should not be added prior to 1312 extraction of the sample.

8.2.2 In most cases, matrix spike levels should be added at a concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be less than five times the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of 1312 extract as that which was analyzed for the unspiked sample.

8.2.3 The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. Use of other internal calibration methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration in the 1312 extract when the recovery of the matrix spike is below the expected analytical method performance.

8.2.4 Matrix spike recoveries are calculated by the following formula:

$$\%R\ (\%\ \text{Recovery}) = 100\ (X_s - X_u)\ /\ K$$

where:

X_s = measured value for the spiked sample
X_u = measured value for the unspiked sample, and
K = known value of the spike in the sample.

8.3 All quality control measures described in the appropriate analytical methods shall be followed.

8.4 The use of internal calibration quantitation methods shall be employed for a metallic contaminant if: (1) Recovery of the contaminant from the 1312 extract is not at least 50% and the concentration does not exceed the appropriate regulatory level, and (2) The concentration of the contaminant measured in the extract is within 20% of the appropriate regulatory level.

8.4.1. The method of standard additions shall be employed as the internal calibration quantitation method for each metallic contaminant.

8.4.2 The method of standard additions requires preparing calibration standards in the sample matrix rather than reagent water or blank solution. It requires taking four identical aliquots of the solution and adding known amounts of standard to three of these aliquots. The forth aliquot is the unknown. Preferably, the first addition should be prepared so that the resulting concentration is approximately 50% of the expected concentration of the sample. The second and third additions should be prepared so that the concentrations are approximately 100% and 150% of the expected concentration of the sample. All four aliquots are maintained at the same final volume by adding reagent water or a blank solution, and may need dilution adjustment to maintain the signals in the linear range of the instrument technique. All four aliquots are analyzed.

8.4.3 Prepare a plot, or subject data to linear regression, of instrument signals or external-calibration-derived concentrations as the dependant variable (y-axis) versus concentrations of the additions of standards as the independent variable (x-axis). Solve for the intercept of the abscissa (the independent variable, x-axis) which is the concentration in the unknown.

8.4.4 Alternately, subtract the instrumental signal or external-calibration-derived concentration of the unknown (unspiked) sample from the instrumental signals or external-calibration-derived concentrations of the standard additions. Plot or subject to linear regression of the corrected instrument signals or external-calibration-derived concentrations as the dependant variable versus the independent variable. Derive concentrations for the unknowns using the internal calibration curve as if it were an external calibration curve.

8.5 Samples must undergo 1312 extraction within the following time periods:

SAMPLE MAXIMUM HOLDING TIMES (days)

	From: Field Collection To: 1312 extraction	From: 1312 extraction To: Preparative extraction	From: Preparative extraction To: Determinative analysis	Total Elapsed Time
Volatiles	14	NA	14	28
Semi-volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except mercury	180	NA	180	360
NA = Not Applicable				

If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

9.0 METHOD PERFORMANCE

9.1 Precision results for semi-volatiles and metals: An eastern soil with high organic content and a western soil with low organic content were used for the semi-volatile and metal leaching experiments. Both types of soil were analyzed prior to contaminant spiking. The results are shown in Table 6. The concentration of contaminants leached from the soils were reproducible, as shown by the moderate relative standard deviations (RSDs) of the recoveries (averaging 29% for the compounds and elements analyzed).

9.2 Precision results for volatiles: Four different soils were spiked and tested for the extraction of volatiles. Soils One and Two were from western and eastern Superfund sites. Soils Three and Four were mixtures of a western soil with low organic content and two different municipal sludges. The results are shown in Table 7. Extract concentrations of volatile organics from the eastern soil were lower than from the western soil. Replicate leachings of Soils

Three and Four showed lower precision than the leachates from the Superfund soils.

10.0 REFERENCES

1. Environmental Monitoring Systems Laboratory, "Performance Testing of Method 1312; QA Support for RCRA Testing: Project Report". EPA/600/4-89/022. EPA Contract 68-03-3249 to Lockheed Engineering and Sciences Company, June 1989.

2. Research Triangle Institute, "Interlaboratory Comparison of Methods 1310, 1311, and 1312 for Lead in Soil". U.S. EPA Contract 68-01-7075, November 1988.

Table 1. Volatile Analytes[1]

Compound	CAS No.
Acetone	67-64-1
Benzene	71-43-2
n-Butyl alcohol	71-36-3
Carbon disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroform	67-66-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethylene	75-35-4
Ethyl acetate	141-78-6
Ethyl benzene	100-41-4
Ethyl ether	60-29-7
Isobutanol	78-83-1
Methanol	67-56-1
Methylene chloride	75-09-2
Methyl ethyl ketone	78-93-3
Methyl isobutyl ketone	108-10-1
Tetrachloroethylene	127-18-4
Toluene	108-88-3
1,1,1,-Trichloroethane	71-55-6
Trichloroethylene	79-01-6
Trichlorofluoromethane	75-69-4
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1
Vinyl chloride	75-01-4
Xylene	1330-20-7

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[1] When testing for any or all of these analytes, the zero-headspace extractor vessel shall be used instead of the bottle extractor.

Table 2. Suitable Rotary Agitation Apparatus[1]

Company	Location	Model No.
Analytical Testing and Consulting Services, Inc.	Warrington, PA (215) 343-4490	4-vessel extractor (DC20S); 8-vessel extractor (DC20); 12-vessel extractor (DC20B)
Associated Design and Manufacturing Company	Alexandria, VA (703) 549-5999	2-vessel (3740-2); 4-vessel (3740-4); 6-vessel (3740-6); 8-vessel (3740-8); 12-vessel (3740-12); 24-vessel (3740-24)
Environmental Machine and Design, Inc.	Lynchburg, VA (804) 845-6424	8-vessel (08-00-00) 4-vessel (04-00-00)
IRA Machine Shop and Laboratory	Santurce, PR (809) 752-4004	8-vessel (011001)
Lars Lande Manufacturing	Whitmore Lake, MI (313) 449-4116	10-vessel (10VRE) 5-vessel (5VRE)
Millipore Corp.	Bedford, MA (800) 225-3384	4-ZHE or 4 1-liter bottle extractor (YT300RAHW)

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[1] Any device that rotates the extraction vessel in an end-over-end fashion at 30 ±2 rpm is acceptable.

Table 3. Suitable Zero-Headspace Extractor Vessels[1]

Company	Location	Model No.
Analytical Testing & Consulting Services, Inc.	Warrington, PA (215) 343-4490	C102, Mechanical Pressure Device
Associated Design and Manufacturing Company	Alexandria, VA (703) 549-5999	3745-ZHE, Gas Pressure Device
Lars Lande Manufacturing[2]	Whitmore Lake, MI (313) 449-4116	ZHE-11, Gas Pressure Device
Millipore Corporation	Bedford, MA (800) 225-3384	YT30090HW, Gas Pressure Device
Environmental Machine and Design, Inc.	Lynchburg, VA (804) 845-6424	VOLA-TOX1, Gas Pressure Device

[1] Any device that meets the specifications listed in Step 4.2.1 of the method is suitable.

[2] This device uses a 110 mm filter.

Table 4. Suitable Filter Holders[1]

Company	Location	Model/ Catalogue #	Size
Nucleopore Corporation	Pleasanton, CA (800) 882-7711	425910 410400	142 mm 47 mm
Micro Filtration Systems	Dublin, CA (800) 334-7132 (415) 828-6010	302400 311400	142 mm 47 mm
Millipore Corporation	Bedford, MA (800) 225-3384	YT30142HW XX1004700	142 mm 47 mm

[1] Any device capable of separating the liquid from the solid phase of the waste is suitable, providing that it is chemically compatible with the waste and the constituents to be analyzed. Plastic devices (not listed above) may be used when only inorganic analytes are of concern. The 142 mm size filter holder is recommended.

Table 5. Suitable Filter Media[1]

Company	Location	Model	Pore Size (μm)
Millipore Corporation	Bedford, MA (800) 225-3384	AP40	0.7
Nucleopore Corporation	Pleasanton, CA (415) 463-2530	211625	0.7
Whatman Laboratory Products, Inc.	Clifton, NJ (201) 773-5800	GFF	0.7
Micro Filtration Systems	Dublin, CA (800) 334-7132 (415) 828-6010	GF75	0.7

[1] Any filter that meets the specifications in Step 4.4 of the Method is suitable.

TABLE 6 - METHOD 1312 PRECISION RESULTS FOR SEMI-VOLATILES AND METALS

		Eastern Soil (pH 4.2)		Western Soil (pH 5.0)	
	Amount Spiked (µg)	Amount Recovered* (µg)	% RSD	Amount Recovered* (µg)	% RSD
FORTIFIED ANALYTES					
bis(2-chloroethyl)-ether	1040	834	12.5	616	14.2
2-Chlorophenol	1620	1010	6.8	525	54.9
1,4-Dichlorobenzene	2000	344	12.3	272	34.6
1,2-Dichlorobenzene	8920	1010	8.0	1520	28.4
2-Methylphenol	3940	1860	7.7	1130	32.6
Nitrobenzene	1010	812	10.0	457	21.3
2,4-Dimethylphenol	1460	200	18.4	18	87.6
Hexachlorobutadiene	6300	95	12.9	280	22.8
Acenaphthene	3640	210	8.1	310**	7.7
2,4-Dinitrophenol	1300	896**	6.1	23**	15.7
2,4-Dinitrotoluene	1900	1150	5.4	585	54.4
Hexachlorobenzene	1840	3.7	12.0	10	173.2
gamma BHC (Lindane)	7440	230	16.3	1240	55.2
beta BHC	640	35	13.3	65.3	51.7
METALS					
Lead	5000	70	4.3	10	51.7
Cadmium	1000	387	2.3	91	71.3

* - Triplicate analyses.
** - Duplicate analyses; one value was rejected as an outlier at the 90% confidence level using the Dixon Q test.

TABLE 7 - METHOD 1312 PRECISION RESULTS FOR VOLATILES

	Soil No. 1 (Western)		Soil No. 2 (Eastern)		Soil No. 3 (Western and Sludge)		Soil No. 4 (Western and Sludge)	
Compound Name	Avg. %Rec.*	%RSD	Avg. %Rec.*	%RSD	Avg. %Rec.**	%RSD	Avg. %Rec.***	%RSD
Acetone	44.0	12.4	43.8	2.25	116.0	11.5	21.3	71.4
Acrylonitrile	52.5	68.4	50.5	70.0	49.3	44.9	51.8	4.6
Benzene	47.8	8.29	34.8	16.3	49.8	36.7	33.4	41.1
n-Butyl Alcohol (1-Butanol)	55.5	2.91	49.2	14.6	65.5	37.2	73.0	13.9
Carbon disulfide	21.4	16.4	12.9	49.5	36.5	51.5	21.3	31.5
Carbon tetrachloride	40.6	18.6	22.3	29.1	36.2	41.4	24.0	34.0
Chlorobenzene	64.4	6.76	41.5	13.1	44.2	32.0	33.0	24.9
Chloroform	61.3	8.04	54.8	16.4	61.8	29.1	45.8	38.6
1,2-Dichloroethane	73.4	4.59	68.7	11.3	58.3	33.3	41.2	37.8
1,1-Dichloroethane	31.4	14.5	22.9	39.3	32.0	54.4	16.8	26.4
Ethyl acetate	76.4	9.65	75.4	4.02	23.0	119.8	11.0	115.5
Ethylbenzene	56.2	9.22	23.2	11.5	37.5	36.1	27.2	28.6
Ethyl ether	48.0	16.4	55.1	9.72	37.3	31.2	42.0	17.6
Isobutanol (4-Methyl-1-propanol)	0.0	ND	0.0	ND	61.8	37.7	76.0	12.2
Methylene chloride	47.5	30.3	42.2	42.9	52.0	37.4	37.3	16.6
Methyl ethyl ketone (2-Butanone)	56.7	5.94	61.9	3.94	73.7	31.3	40.6	39.0
Methyl isobutyl ketone	81.1	10.3	88.9	2.99	58.3	32.6	39.8	40.3
1,1,1,2-Tetrachloroethane	69.0	6.73	41.1	11.3	50.8	31.5	36.8	23.8
1,1,2,2-Tetrachloroethane	85.3	7.04	58.9	4.15	64.0	25.7	53.6	15.8
Tetrachloroethene	45.1	12.7	15.2	17.4	26.2	44.0	18.6	24.2
Toluene	59.2	8.06	49.3	10.5	45.7	35.2	31.4	37.2
1,1,1-Trichloroethane	47.2	16.0	33.8	22.8	40.7	40.6	26.2	38.8
1,1,2-Trichloroethane	76.2	5.72	67.3	8.43	61.7	28.0	46.4	25.4
Trichloroethene	54.5	11.1	39.4	19.5	38.8	40.9	25.6	34.1
Trichlorofluoromethane	20.7	24.5	12.6	60.1	28.5	34.0	19.8	33.9
1,1,2-Trichlorotrifluoroethane	18.1	26.7	6.95	58.0	21.5	67.8	15.3	24.8
Vinyl chloride	10.2	20.3	7.17	72.8	25.0	61.0	11.8	25.4

* Triplicate analyses
** Six replicate analyses
*** Five replicate analyses

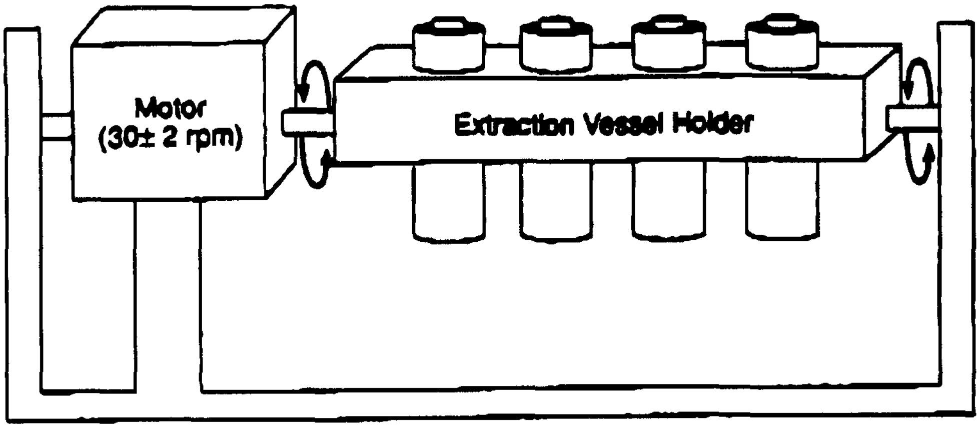


Figure 1. Rotary Agitation Apparatus

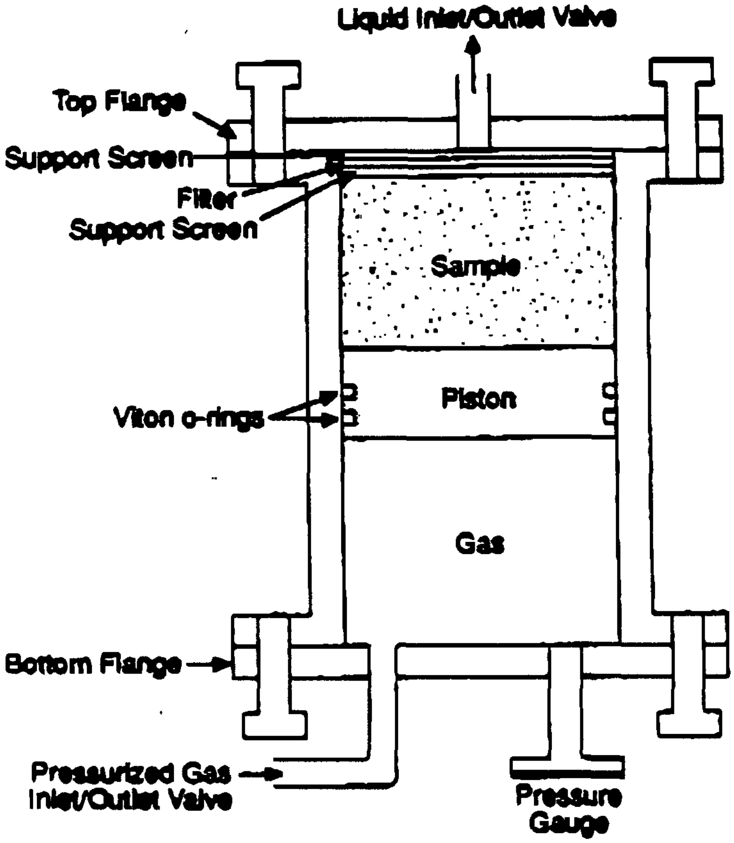
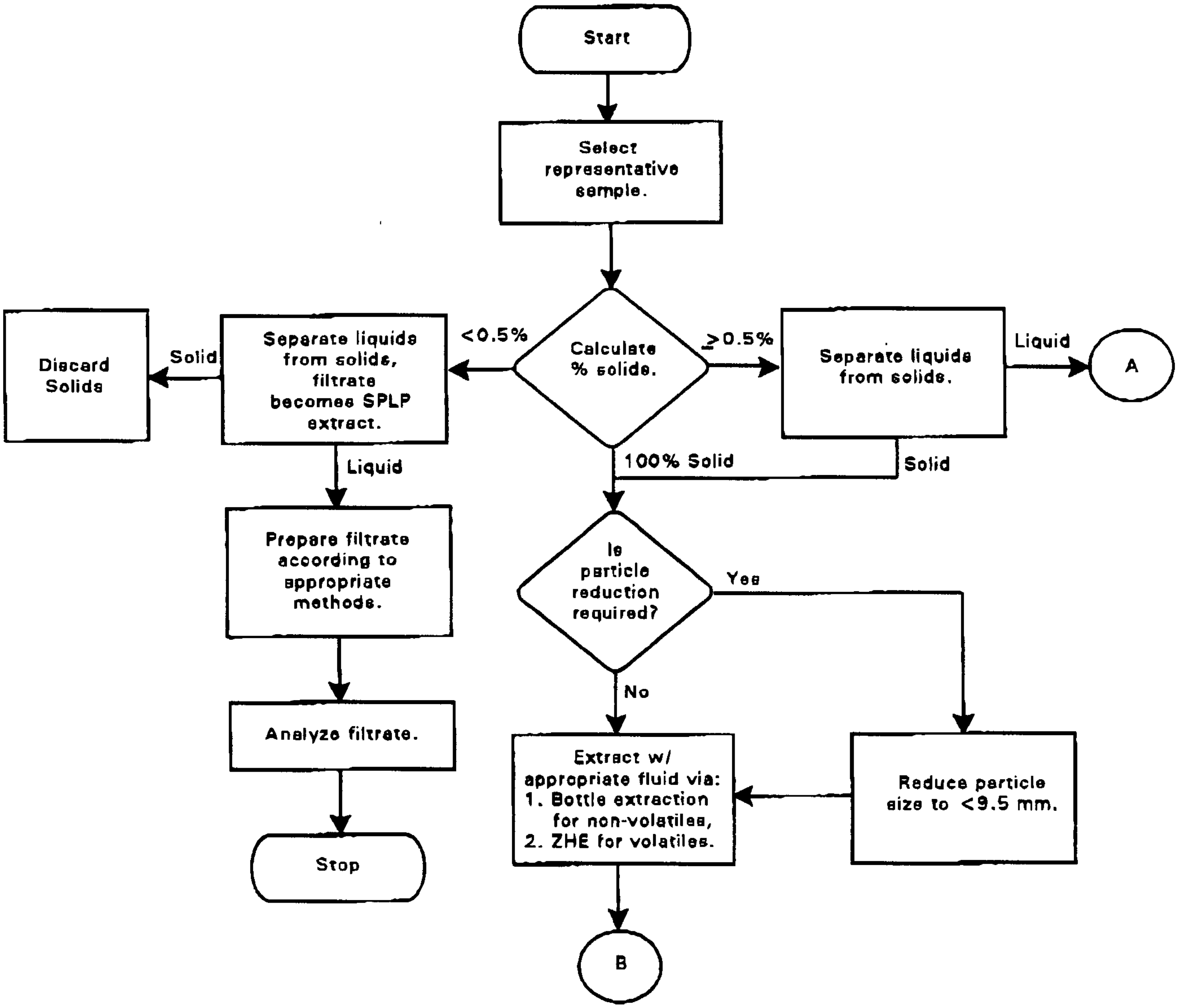


Figure 2. Zero-Headspace Extractor (ZHE)

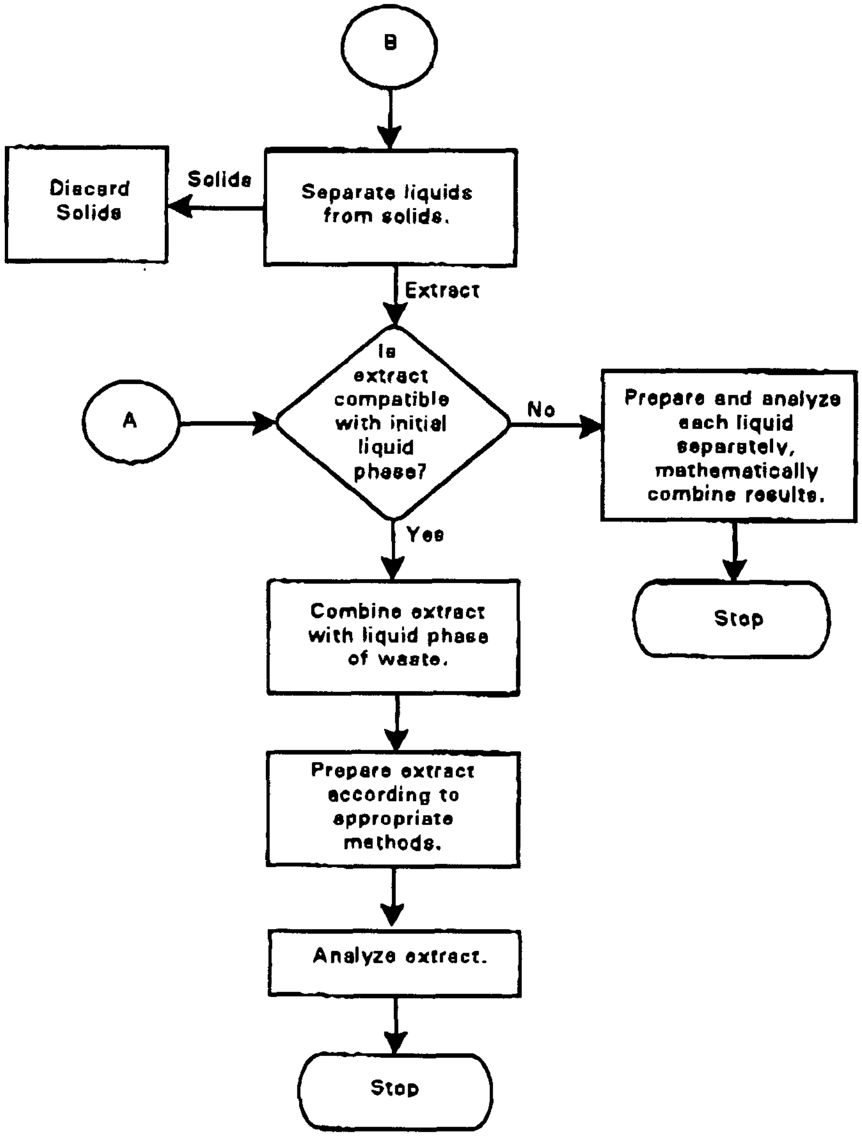
METHOD 1312

SYNTHETIC PRECIPITATION LEACHING PROCEDURE



METHOD 1312

SYNTHETIC PRECIPITATION LEACHING PROCEDURE (continued)



OIL CONSERVATION DIVISION
RECEIVED
'95 APR 20 PM 8 52

Shell Oil Products Company

Two Shell Plaza
P. O. Box 2099
Houston, Texas 77252-2099

April 13, 1995

William Olson
State of New Mexico Oil Conservation Division
Environmental Bureau
2040 S. Pacheco St.
Santa Fe, New Mexico 87504

SUBJECT: SHELL PIPE LINE CORPORATION'S HOBBS STATION PIT CLEAN UP
HOBBS, NEW MEXICO

Dear Mr. Olson,

As we discussed yesterday, Shell Pipe Line Corporation is planning to initiate action leading to the remediation and closure of the pit at Hobbs Station. The hydrocarbon material in the pit is non-exempt tank bottoms from pipeline crude oil storage. All of the pit contents will be transported to Controlled Recovery Inc., OCD Permit #R-9166, for processing to recover and reclaim the hydrocarbon product. A hydraulic excavator will be used to load the material into dump trucks equipped with plastic bed liners. Any free liquid that accumulates in the pit will be collected and transported by vacuum truck to CRI. Once the pit is empty we will review the site and develop a subsurface investigation plan in accordance with the New Mexico Oil Conservation Division "Unlined Surface Impoundment Closure Guidelines". Upon review of the investigation results we will develop and submit a site remediation plan. Assuming no major delays during the removal process I feel we can submit a site investigation to you by June 2. I respectfully request your concurrence with this approach.

If you have any questions please call me at 713-241-2961.

Sincerely

Neal Stidham

Neal Stidham
Shell Oil Products Company
Representing Shell Pipe Line Corporation

cc: C.H.Kerby-SPLC Midland
L.F.Sotomayor-SPLC Hobbs
B.J.Rush-SPLC Denver City

Submit 5 copies to Appropriate District Office
DISTRICT I
P.O.Box 1980, Hobbs, NM 88241-1980
DISTRICT II
P.O. Drawer DD, Artesia, NM 88211-0719
DISTRICT III
1000 Rio Brazos Rd, Aztec, NM 87410

State of New Mexico
[illegible]gy, Minerals and Natural Resources Departm[illegible]

OIL CONSERVATION DIVISION

P.O. Box 2088
Santa Fe, New Mexico 87504-2088

Form C-117 A
Revised 4-1-91

PERMIT NO.__________

TANK CLEANING, SEDIMENT OIL REMOVAL, TRANSPORTATION OF MISCELLANEOUS HYDROCARBONS AND DISPOSAL PERMIT

Operator or Owner Shell Pipe Line Corporation Address 831 Sunrise Circle, Hobbs, NM 88240

Lease or Facility Name Hobbs Station Location Sec. 15, T19S, R36E
U.L. - Sec. - Twp. - Rge.

OPERATION TO BE PERFORMED:

☐ Tank Cleaning ☒ Sediment Oil Removal ☒ Transportation of Miscellaneous Hydrocarbons

Operator or Owner Representative authorizing work Shell Pipe Line Corporation

Date Work to be Performed Upon permit approval

TANK CLEANING DATA Tank Number__________ Volume__________

Tank Type__________ Volume Below Load Line__________

SEDIMENT OIL OR MISCELLANEOUS HYDROCARBON DATA

Sediment Oil from: ☒ Pit ☐ Cellar ☐ Other

MISCELLANEOUS OIL

Tank Bottoms From: ☐ Pipeline Station ☐ Crude Terminal ☐ Refinery ☐ Other*

Catchings From: ☐ Gasoline Plant ☐ Gathering Lines ☐ Salt Water Disposal System ☐ Other*

Pipeline Break Oil or Spill ☐

*Other (Explain)__________

VOLUME AND DESTINATION: Estimated Volume 19,000 Bbls. Field test volume of good oil Est. 40-50% Bbls.
(Not required prior to Division approval)

Destination (Name and Location of treating plant or other facility) Controlled Recovery Inc. (CRI)
P. O. Box 369, Hobbs, NM 88241

DESTRUCTION OF SEDIMENT OIL BY: ☐ Burning ☐ Pit Disposal ☐ Use on Roads or firewalls ☐ Other

(Explain)__________

Location of Destruction__________

Justification of Destruction__________

CERTIFICATION : (APPLICATION MAY BE MADE BY EITHER OF THE FOLLOWING)
I hereby certify that the infomation above is true and complete to the best of my knowledge and belief.

Owner Shell Pipe Line Corporation	Transporter__________
By	Address__________
Title Staff Engineer	Signature__________
Date April 12, 1995	Title__________ Date__________

OIL CONSERVATION DIVISION

Approved By Bill Olson Title Geologist IV Santa Fe Date 4/28/95

A COPY OF THIS FORM MUST BE ON LOCATION DURING TANK CLEANING, REMOVAL OF SEDIMENT OIL OR MISCELLANEOUS HYDROCARBONS, AND MUST BE PRESENTED WITH TANK BOTTOMS, SEDIMENT OIL OR MISCELLANEOUS HYDROCARBONS AT THE TREATING PLANT TO WHICH IT IS DELIVERED.

DISTRIBUTION BY OCD
✓ ~~Santa Fe~~ Hobbs
✓ File
Operator
Transporter (2)

Bill Olson

From: Wayne Price
To: Bill Olson
Cc: Wayne Price; Jerry Sexton
Subject: Shell Pit Closure
Date: Wednesday, April 12, 1995 11:03AM
Priority: High

<<File Attachment: SHELL.PIT>>

Dear Bill,

Per our telephone conversation, I am sending you a copy of the letter that I sent Shell concerning the pit closure (see E-mail attachment). Please review and call or write me on your comments. If this pit closure needs to be handled out of Santa Fe, then as soon as Shell responds I will forward it up to you.

Thanks Bill, I will appreciate any feedback on the proper procedures to be used on these type of closures.

Wayne Price.

April 11, 1995

Mr. Neal Stidham
Staff Engineer
Shell Oil Products Company
P.O. 2099
Houston, Texas 77252-2099

Dear Mr. Stidham,

Per our telephone conversation today, I understand that Shell Oil Products Company (SOPC) wishes to close an old pit located just south of Hobbs, New Mexico. I also understand that you indicated this material generated from the pit would possibly be classified as a RCRA Non-exempt waste due to its association with previous crude oil pipeline activities.

Therefore, please submit to this office a closure plan which includes locations, a summary of your anticipated work, disposal plans for any material to be shipped off-site and/or site assessment plan. We will review the plan and approve as is or with conditions.

I have included a form C-103 and will send you a set of "Pit Closure Guidelines" with a new pit closure form to be filled out at the end of the project.

Please note, any disposal of any RCRA non-exempt waste that is offered to a New Mexico permitted facility must be approved by the NMOCD on a special form called "REQUEST FOR APPROVAL TO ACCEPT SOLID WASTE". The DISPOSAL company that you are dealing with can provide you all the necessary information and paper work that is required.

If the material contained in the pit is classified as a solid waste as defined in EPA CFR 40 part 261 then SOPC must make a waste determination to determine if it is RCRA hazardous waste. If the material is determined to be hazardous then SOPC should notify the New Mexico Environmental Department, Hazardous & Radioactive Material Bureau (505-827-4300).

However, if the material located within the pit in question is not classified as a RCRA solid waste, or is otherwise excluded or exempted from such definitions as defined in EPA CFR 40 part 261 then the requirement of using a "REQUEST FOR APPROVAL TO ACCEPT SOLID WASTE" form is not required. The proof of this waste determination should be included in the submitted work plan mentioned above.

Please find enclosed a C-103 form to be filled out by SOPC and sent in to this NMOCD office for approval for the above mentioned pit closure.

If you have any further questions please do not hesitate to call or write.

Sincerely yours,

Wayne Price-Environmental Engineer

CC: Roger Anderson-Environmental Bureau Chief
Jerry Sexton-District I Supervisor

Attachments-2