ENVIRONMENTAL PROTECTION AGENCY OFFICE OF ENFORCEMENT

EPA-330/2-77-022

The Potential For Pollution

Of The Little Menomonee River

From The Kerr-McGee/Moss-American Plant Site

Milwaukee, Wisconsin

(September-October 1977)

NATIONAL ENFORCEMENT INVESTIGATIONS CENTER
DENVER, COLORADO

REGION V. CHICAGO, ILLINOIS

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ENVIRONMENTAL PROTECTION AGENCY Office of Enforcement EPA-330/2-77-022

THE POTENTIAL FOR POLLUTION OF THE LITTLE MENOMONEE RIVER FROM THE KERR-McGEE/MOSS-AMERICAN PLANT SITE MILWAUKEE, WISCONSIN

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National Enforcement Investigations Center - Denver and ${\sf Region\ V\ -\ Chicago}$

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I. INTRODUCTION

Creosote contamination of the Little Menomonee River from the operation of a Kerr-McGee/Moss-American creosote plant near Milwaukee, Wisconsin has been a long-standing problem in the area. Severe contamination occurred in the past as a result of the direct discharge of wastewater and now, even though the plant has ceased operations, continued contamination from residual creosote deposits is of concern.

The National Enforcement Investigations Center (NEIC) of the Environmental Protection Agency (EPA) was requested by EPA's Region V to conduct an investigation of the plant site and recommend measures to eliminate further pollution of the River. This investigation was conducted in September and October of 1977. A review of the history of the site and the resulting pollution is presented in an earlier report by NEIC.*

Due to the highly technical and complex nature of the subject matter concerning creosote contamination of the Little Menomonee River, the Honorable Myron L. Gordon, U.S. District Judge (Eastern District, Wisconsin) appointed David A. Saichek, of Gaines and Saichek, to serve as Special Master in the matter of United States v. Kerr-McGee/Moss-American, Inc. ** All drilling, sampling and other field observations conducted by NEIC were done in accordance with requirements specified in a letter from the Special Master dated April 29, 1977 to Mr. Frank J. Daily, Attorney for the defendant, and Mr. Charles H. Bohl, U.S.

** Case No. 75-C-277.

^{*} National Enforcement Investigations Center, June 1977. Impact of Creosote Deposits in the Little Menomonee River, Wisconsin (April 1977). Denver: Environmental Protection Agency, 330/2-77-016, 47 p.

Attorney. In addition, on-site field operations including well drilling, sampling, water level measurements, well casing removal, well backfilling and abandonment procedures were observed by the Special Master, the EPA Region V Attorney, and representatives of the defendant.

This report describes the field methods used during the September and October surveys. It also presents the results of the investigation and recommends measures to reduce or eliminate movement of creosote from the plant site into the Little Menomonee River.

II. SUMMARY AND CONCLUSIONS

As requested by EPA, Region V, the National Enforcement Investigations Center conducted an investigation of the abandoned Kerr-McGee/Moss-American plant site near Milwaukee, Wisconsin. The objectives of the investigation were to determine the extent of creosote contamination in soil and groundwater, to evaluate the potential for continued pollution of the Little Menomonee River from this site, and to determine the measures necessary to eliminate the source of pollution.

During a site visit on September 19 and 20, 1977, six wells were drilled, casings were installed, groundwater levels were determined and water samples were collected from four of the wells for analysis [Appendix A]. In addition, core samples were collected at depths over 4.6 m (15 ft) during well drilling. Transects were laid out in relation to the wells and to structures in the area, and more than 100 soil samples were collected at depths up to 90 cm (3 ft) for later analysis.

Another site visit was made on October 19, 1977 to collect a second series of water samples from the wells and to remove the well casings. The wells were backfilled and the property was restored to its presurvey condition to the satisfaction of Company representatives present on the site.

All soil and water samples were returned to the NEIC under chain-of-custody procedures [Appendix B] and analyzed for methylene chloride extractables [Appendix C]. The moisture content of the soil samples was also determined. The presence of creosote in the extracts was confirmed by gas chromatography on all of the extracts and the individual components of creosote were confirmed by mass spectrometry analyses of selected samples.

Groundwater level at each well was measured during both visits. These measurements plus general observations of the area and a knowledge of geology and hydrology indicate that the groundwater flows toward the Little Menomonee River.

The presence of creosote in water samples from two wells during both site visits and from a third well during the October visit shows that the groundwater is contaminated with creosote.

Analyses of core samples and soil samples taken from the transects show that the entire area is contaminated with creosote, in some cases to a depth of at least 4.6 m (15 ft) below ground level. In one case, the concentration of methylene chloride extractables in the soil was greater than 27% and it was greater than 10% in several more samples.

Such widespread and high concentrations of creosote at the Kerr-McGee/Moss-American plant site make it evident that contamination of the Little Menomonee River will continue unabated for years if no efforts are made to control surface runoff and mobility of the creosote-contaminated groundwater.

The provision of an interceptor ditch, however, would prevent the contaminated runoff, groundwater and soil from entering the River. A granular carbon adsorption column may be necessary if the ditch effluent contains dissolved contaminants.

III. RECOMMENDATIONS

The inflow to the Little Menomonee River of creosote-contaminated surface runoff, groundwater, and soil could be prevented by installing an interceptor ditch along the railroad track north of the site and between the contaminated area and the Little Menomonee River [Figure 1, Section IV].

The ditch should intercept the groundwater and surface runoff from the Kerr-McGee/Moss-American property. A system of skimmers and adsorption media (straw), placed in the ditch, would remove creosote from the interceptor discharge before it enters the Little Menomonee River. At 2.4 m (8 ft) deep, the ditch would collect any creosote floating on the water table and any soil washed off during a storm.

The ditch would have to be maintained regularly to remove accumulated skimmed material and solids that have settled.

If the water leaving the ditch is contaminated with soluble or emulsified components of creosote, it may be necessary to install a granular activated carbon column to treat the discharge from the ditch.

IV. STUDY METHODS

WELL AND SOIL SAMPLING SITE LOCATIONS

The investigated site consists of approximately 44,500 m² (11 acres) located in the NW 1/4, Section 8, T.8N., R.21E. near Brown Deer, Milwaukee County, Wisconsin [Figure 1]. From an aerial photograph of the site and an on-site inspection, the decision was made that six observation wells would be drilled to characterize the degree of contamination of the soil and the groundwater with creosote. One well was drilled near the northeast corner of a green shed close to the former retort area, and where it was assumed that the concentration of creosote would be the highest. Five other wells were drilled in relation to this "green shed" well, as described in Table 1; Figure 1 shows the approximate location of these wells. Because of surface characteristics (water, muck, etc.), it was not practicable to drill all of the wells at what was considered optimum locations.

Once the wells were located, transects were established in relation to the wells and to structures in the area [Table 2]. The transects enabled the location of sites for collection of samples of soil from the surface and down to depths of 90 cm (3 ft) below land surface. The analysis of the samples collected allowed a more complete evaluation of the extent of contamination in the area.

The six observation wells were drilled on the eastern half of the Kerr-McGee/Moss-American plant site on September 19 and 20, 1977 using a truck-mounted continuous flight auger drill rig. In the field, a geologic log was prepared of each hole, including observations made by studying cuttings brought from the wells, noting rig behavior and taking drive samples.

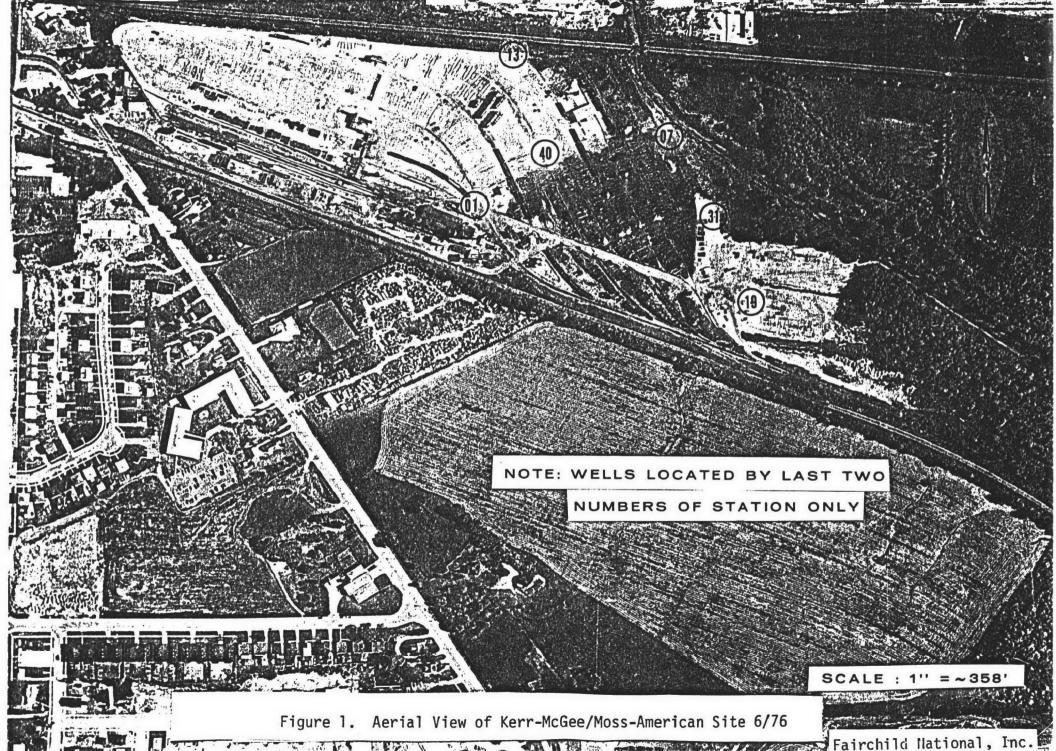


Table 1

LOCATION OF WELLS INSTALLED AT THE KERR-MCGEE/MOSS-AMERICAN PLANT SITE MILWAUKEE, WISCONSIN September 19-20, 1977

Well	Location Description [†]		
772401	<pre>2.4 m (8 ft) from NE corner of the green shed, bearing 045°</pre>		
772407	226 m (740 ft) from well 772401, bearing 045°		
772419	315 m (1,035 ft) from well 772401, bearing 080°		
772413	177 m (582 ft) from well 772401, bearing 335°		
772440	About 91 m (298 ft) from well 772401 and 29 m (96 ft) north of transect A		
772431	Halfway between wells 772407 and 772419 on transect F		

⁺ All bearings were taken with magnetic compass

Table 2

TRANSECTS USED IN LOCATING SOIL SAMPLING AND WELL SITES KERR-MCGEE/MOSS-AMERICAN PLANT SITE MILWAUKEE, WISCONSIN September, 1977

Transect	Description
Α	Line connecting wells 772401 and 772407
В	Line connecting wells 772401 and 772413
С	Line connecting wells 772407 and 772413
D	Line east from well 772413 and parallel to railroad tracks
E	Line connecting wells 772401 and 772419
F	Line connecting wells 772407 and 772419
G	Line parallel to and 1 m (3 ft) south of retorts
Н	Line parallel to and 0.3 m (1 ft) north of north wall of retort building

A drive sample was collected by removing the auger from the well and replacing it with a "split-spoon sampler" on the bottom end of the drill rod. The split spoon was driven into the undisturbed earth at the bottom of the well by dropping a 59 kg (130 lb) weight. The number of blows required to drive split-spoon sampler 46 cm (18 in) deep was recorded at the time of sampling. These data are recorded in the drill logs.

After the split-spoon sampler was removed, the auger was reinstalled in the hole, and drilling continued until the desired depth was reached. Upon reaching this depth, the auger was removed from the hole and a section of 5-cm (2-in) diameter PVC pipe, capped on the end and perforated with hacksaw slots, was installed in the hole. Backfill around the casing was obtained from cuttings which had been withdrawn from the hole.

During the drilling of the green-shed well (772401), it was noted that cores obtained from 3.0 to 3.5 m (10 to 11.5 ft) and from 4.6 to 5.0 m (15 to 16.5 ft) had layers of creosote ** filling fractures and partings. As drilling progressed to about 6 m (20 ft), the auger began to lift water to the surface. This water had globules of creosote floating on the surface. As the auger was withdrawn, it was noted that the bottom 4.6 m (15 ft) was not coated with clay as had been the experience in other holes on the site, but instead was coated with creosote. This apparently was the result of drilling through a layer of creosote which coated the auger and prevented any clay from adhering to the auger.

^{*} Polyvinyl Chloride

^{**} Identified on the basis of odor and appearance

SAMPLING AND ANALYTICAL PROCEDURES

Well Samples

Upon completing the six observation wells, the depth to groundwater was determined, and water sampling proceeded. Depth to water, obtained with a Powers' electric well sounder, was recorded as depth below the undisturbed land surface [Table 3].

After the depth was recorded, a 4.6 m (15 ft) long, 2.5 cm (1 in) diameter suction hose was installed in the casing and well was pumped until dry or until suction was broken. This was done twice at each well, then water samples were collected in glass containers prerinsed with water from each well using a Homelight gasoline-powered water pump. Water could not be obtained from two of the wells (772407 and 772419) due to the tight clay which comprises the bulk of the formation.

During each pumping of the green-shed well (772401), the water was noticed to contain globules of creosote, and as the pump broke suction a creosote froth was produced.

The soil samples from the wells were taken by opening the splitspoon sampler and placing the lower portions of the core in chemically clean glass jars capped with Teflon-lined lids. The upper portion of the core, which included material that fell into the hole as the auger was being removed, was not saved as a part of the sample.

All samples, soil and water, were taken to the NEIC Laboratory for analysis. Chain-of-custody procedures were followed throughout.

On October 19, the site was again visited by NEIC to re-sample the wells and return the site to pre-survey conditions. Several of

Table 3

WATER LEVEL MEASUREMENTS IN WELLS DRILLED AT THE KERR-MCGEE/MOSS-AMERICAN PLANT SITE MILWAUKEE, WISCONSIN September 19 - 20, 1977

	Date	Depth to water be	
Well	(Sept.)	(cm)	(ft)
772401	20	142	4.7
772407	19	27	0.9
772419	20	143	4.7
772413	20	33.5	1.1
772440	20	42.7	1.4
772431	20	79.2	2.6

the wells, all of which had been capped with a plastic cover, had been vandalized and the well casing was broken off at or just above the ground surface. As before, the depth to water level at each of the wells was measured, this time using a steel tape [Table 4]. Then the wells were purged to a depth of 4.6 m (15 ft) and left to recover before sampling.

Also as noted during the previous visit, the well nearest the green shed produced a black, greasy liquid during purging. Well 772407, which produced no water during the earlier attempt at sampling, produced a very muddy, thick liquid during purging.

During the October visit, samples were obtained by use of a hand-powered diaphragm pump which was rinsed with acetone between sampling at each well. Well 772407 was difficult to sample, probably because of mud and clay blocking the slots in the casing. To get enough sample for analysis, it was finally necessary to use a hand-operated bailer. The sample contained about 1,000 ml of water and 900 g of clayey sediment.

The sample from the well nearest the green shed was brownish with an iridescent sheen on the surface. The sample was not as dark or greasy as the material removed when the well had been purged earler in the day.

At sampling site 772452, about 31 m (100 ft) southwest of well 772440, the discharge from a small spring flowed through a small clump of vegetation and then toward the Little Menomonee River. A small pond had formed which was partially covered by an oily, iridescent sheen. As the water came up out of the ground, a sample was taken.

Table 4

WATER LEVEL MEASUREMENTS IN WELLS AND A SPRING AT THE

KERR-MCGEE/MOSS-AMERICAN PLANT SITE

MILWAUKEE, WISCONSIN

October 19, 1977

Well	Depth to water	below land surface
	cm	ft
772401	101.60	3.3
772407	36.48	1.0
772419	93.98	3.1
772413	36.83	1.2
772440	34.29	1.1
772431	76.20	2.5
772452	Surfa	ce

After sampling, all casings were pulled from the wells and removed from the site. The holes were backfilled with a mixture of sand and bentonite clay, and the survey stakes placed during the earlier site visit were removed.

Soil Samples

Soil samples were collected from sites on the transects described in Table 2. At each of the sites except the wells, a small coring device was used to collect a surface sample. An auger was then used to excavate a hole to an intermediate depth of 30 to 50 cm (1 to 1.6 ft) where another core sample was collected. The holes were then deepened with the auger to final depths varying between about 55 and 90 cm (1.8 to 3.0 ft) and again a small core sample was collected from the bottom of each hole.

Analytical Procedures

The soil and water samples were analyzed for methylene chloride extractables. In addition, the moisture content of the soils was determined. The methylene chloride extracts were also analyzed for the presence of creosote compounds by gas chromatography.

Individual components of creosote were also confirmed in selected samples by mass spectrometry. In all cases, unless otherwise indicated, most of the methylene chloride extractable material was creosote.

V. GEOLOGIC OBSERVATIONS AND ANALYTICAL RESULTS

The entire Kerr-McGee/Moss-American plant site is underlain by a thick section [at least 17 m (55 ft)] of plastic, clayey glacial till with a greater percentage of gravel material in the till section along the eastern side of the plant site near the Little Menomonee River. till is locally overlain by a man-made fill and/or by a section of organic peat. In the Kerr-McGee/Moss-American plant site, the till showed evidence of horizontal openings filled with creosote to a depth of at least 4.6 m (15 ft) in the undisturbed core samples. Groundwater occurs in the till section throughout the entire area of the Kerr-McGee/Moss-American plant site investigated. In humid areas such as in the Milwaukee vicinity, the groundwater table configuration is characteristically a subdued replica of the land surface which, at the subject site, slopes north and east across the Kerr-McGee/Moss-American site toward the Little Menomonee River. Groundwater and soil samples obtained from the plant site indicated evidence of severe creosote contamination, especially in the area of the former retort building. As the groundwater and soil are known to contain creosote and the water table slopes toward the Little Menomonee River, any movement will be in this direction.

As reported in Table 5, the water samples collected from the observation well (772401) near the retort contained 4,910 mg/l of methylene chloride extractables in the September sample and 99 mg/l during the October sampling. Even higher concentrations of creosote were noted in the water standing in the well. The suction line from the well to the pump was coated with creosote adhering to the inside of the pipe. Cores obtained from the surface 0.5 m (0 to 1.5 ft) and from depths within the hole -- 3.0 to 3.5 m and 4.6 to 5.0 m (10 to 11.5 ft

Table 5

ANALYTICAL RESULTS - WELL WATER SAMPLES COLLECTED AT THE KERR-MCGEE/MOSS-AMERICAN PLANT SITE MILWAUKEE, WISCONSIN

Well	Location Description	Methylene Chloride E Sept. 20, 1977 Oct mg/l	
772401	NE corner of green shed	4,190	99
772407	NE corner of area near RR	NS [†]	600 ^{††}
772413	Northside center	ND ^{† † †}	<2 ^{†††}
772419	SE corner of area	${\sf NS}^{\dagger}$	<2 ^{†††}
772431	Eastside center	ND ⁺⁺⁺	<2 ^{†††}
772440	Center of area	20	9
772452	Spring sample, ∿100 ft sou of 772440	uthwest -	<2 ^{†††}

† No sample obtained due to slow water level recovery after well development and purging.

***None detected above the detection limit of methylene chloride extractables or no identification as creosote by gas chromatography.

tt Value rounded off to nearest 100 mg/l. This sample contained a considerable amount of very finely divided clay-like mud. An 1,800 ml slurry of this material was extracted. The slurry consisted of about 1,100 ml of water and 900 g of mud. The moisture content of the mud was 45%. A small amount of the methylene chloride extractables in this sample was creosote. The majority of the extractable material resembled a highly weathered heavy fuel oil.

and 15 to 16.5 ft) -- contained large amounts (2,400 to 7,900 mg/kg) of methylene chloride extractables [Table 6].

It appears that there is a creosote layer, probably several inches thick, at shallow depth in the retort area. In addition, heavier fractions of creosote are apparently sinking through the groundwater to greater depths.

Much of the creosote may be temporarily sorbed on the soil material but a finite portion does move toward the Little Menomonee River. A sample of groundwater obtained from well 772440, near the center of the storage area, was found to contain 20 mg/l of methylene chloride extractables in the September sample and 9 mg/l during the October sample. A core sample, taken at a depth of 3.0 to 3.5 m (10 to 11.5 ft) from this well showed no detectable level of creosote. Creosote was not detected in water samples from any of the other wells except that during the October sampling, the mud-water mixture from well 772407 contained a high concentration of methylene chloride extractables which resembled highly weathered heavy fuel oil. A fraction of this extract was found to contain creosote components. This may be a result of the strong tendency for creosote to adhere to the well casing and to the pump suction line.

Analysis of the well core samples [Table 6] showed creosote at depths more than 4.6 m (15 ft) below the surface and concentrations of methylene chloride extractables as high as 23,700 mg/kg (2.37%). As previously discussed, this creosote was visible and detected by appearance and odor as the cores were removed from the split-spoon sampler. The creosote had evidently percolated through the soil and the ground-water surface to fill voids in the soil to at least the 4.6 m (15 ft) depth.

The presence of creosote from soil samples [Table 6] is randomly distributed throughout the area investigated. It reached a high of

Table 6

ANALYTICAL RESULTS - SOIL AND WELL CORE SAMPLES AT THE KERR-MCGEE/MOSS-AMERICAN PLANT SITE MILWAUKEE, WISCONSIN September 19-20, 1977

Description	Depth	% Moisture	Methylene Chloride Extractable (Dry Weight) mg/kg
Green shed, well	772401 0-1.5 ft 5-6.5 ft 10-11.5 ft 15-16.5 ft	8.7 24.2 16.5 10.6	2,500 ND 7,900 2,400
STATION NO. Transect A, 105 ft from Station 772401	772402 Surface 62 cm 90 cm	11.9 17.5 14.9	9,800 15,400 1,700
STATION NO. Transect A, 228 ft from Station 772401	772403 Surface 35 cm 50 cm	13.8 27.4 35.5	9,200 2,100 11,100
Transect A, 351 ft from Station 772401	772404 Surface 32 cm 82 cm	27.0 53.5 30.0	8,500 4,300 1,000
STATION NO. Transect A, 474 ft from Station 772401	772405 Surface 35 cm 65 cm	39.0 51.3 45.5	5,900 13,100 4,200
Transect A, 597 ft from Station 772401	772406 Surface Mid-Depth 73 cm	40.0 64.5 60.4	9,000 31,800 3,800
NE corner of site, well	772407 0-1.5 ft 5-6.5 ft 10-11.5 ft	20.1 22.1 12.7	23,700 ND [†] ND [†]

Description	Depth	% Moisture	Methylene Chloride Extractable (Dry Weight) mg/kg
STATION NO. 7 Transect B, 145 ft from Station 772401	Surface 50 cm	7.8 12.6	4,500 4,400
STATION NO. 7 Transect B, 291 ft from Station 772401	69 cm 72409 Surface 40 cm 69 cm	7.5 22.7 21.6	3,500 800 4,800 4,300
Transect B, 436.5 ft from Station 77240	72410	16.0 29.5 29.8	5,300 3,100 1,500
North side, well		21.0	1,700 ND
12 ft north of Transect E, 207 ft from Station 772401	Surface	13.3	71,000
	38 cm 55 cm	20.5 18.2	10,400 34,300
12 ft north of Transect E, 414 ft from Station 772401	72415 Surface	12.2	12,900
	50 cm	24.4	ND
	60 cm	22.6	ND^\dagger
12 ft north of Transect E, 611 ft	72416		
from Station 772401	Surface	12.0	20,000
	40 cm	15.1	5,300

Description	Depth	% Moisture	Methylene Chloride Extractable (Dry Weight) mg/kg
STATION NO. 7	72417		
12 ft north of Transect E, 808 ft from Station 772401	Surface	4.9	1,300
	30 cm	11.5	2,400
	60 cm	13.9	2,200
SE corner of site, well STATION NO. 7	72419 0-1.5 ft 5-6.5 ft 10-11.5 f	9.8	ND † ND † ND †
Transect H, NW corner of retort bldg	72420 Surface 50 cm 75 cm	18.3 22.7 18.5	40,200 19,300 13,600
Transect H, Midpoint of retort bldg 71 ft from Station 772401	72421 Surface	17.1	10,500
	47 cm	21.3	3,800
	72 cm	24.4	2,200
Transect H, NE corner of retort bldg	72422 Surface 33 cm 73 cm	8.1 21.1 22.9	4,900 6,200 2,200
	72423		
Transect G, Midpoint between 5th & 6th retorts on south facing side	Surface	13.0	41,900
	40 cm	19.7	2,900
	72 cm	23.5	13,900

Description	Depth	% Moisture	Methylene Chloride Extractable (Dry Weight) mg/kg
STATION NO. 7	72424		
Transect G, Midpoint of 4th retort from left on south facing side	Surface	11.9	92,700
	38 cm	25.8	127,000
	63 cm	25.9	36,400
STATION NO. 7	772425		•
Transect G, Midpoint of 2nd retort from left on south facing side	Surface	2.4	104,000
	38 cm	43.2	133,000
	70 cm	38.7	89,900
STATION NO. 7	772426		
Inside retort room, 40.3 ft from NE corner, 2 ft from north wall	Surface	5.0	72,900
	70 cm	26.0	7,100
STATION NO.	772427		
Inside retort room, 20 ft from NW corner, 4 ft in from north wall	Surface	3.3	279,000
	60 cm	20.2	5,600
STATION NO. Transect F, 168 ft from Station 772419		8.4 23.9 22.4	2,400 4,100 ND
STATION NO. Transect F, 336 ft from Station 772419		9.9 41.0 38.5	1,900 2,8 <u>0</u> 0 ND

Description	Depth N	% Moisture	Methylene Chloride Extractable (Dry Weight) mg/kg
STATION NO. 77 Eastside center, well	2431 5-6.5 ft 10-11.5 ft		ND [†] ND
STATION NO. 77 Transect F, 504 ft from Station 772419	2432 Surface 36 cm 58 cm	27.0 50.8 47.2	11,200 35,8 <u>0</u> 0 ND
STATION NO. 77 Transect D, 75 ft from Station 772413	2433 Surface 40 cm -	24.2 31.8 41.8	8,8 <u>0</u> 0 ND: ND
STATION NO. 77 Transect D, 168 ft from Station 772413	Surface 40 cm 70 cm	23.3 32.2 50.0	9,100 1,700 3,700
STATION NO. 77 Transect D, 261 ft from Station 772413	2435 Surface 40 cm 70 cm	33.3 50.8 55.3	19,700 1,000 1,000
STATION NO. 77 Transect D, 354 ft from Station 772413	72436 Surface 50 cm 80 cm	30.2 51.0 58.5	10,200 1,100 15,600
STATION NO. 77 Transect C, 152 ft from Station 772407	22437 Surface 32 cm 65 cm	26.0 74.5 73.6	8,600 11,000 2,800
STATION NO. 77 Transect C, 304 ft from Station 772407	7 <u>2438</u> Surface 52 cm 70 cm	25.0 59.9 58.5	1,900 3,900 3,100

Table 6 (Continued)
KERR-MCGEE/MOSS-AMERICAN PLANT SITE

Description	Depth	% Moisture	Methylene Chloride Extractable (Dry Weight) mg/kg
STATION NO. Transect C, 456 ft from Station 772407		14.9	900
Transect C, 456 It Troil Station 772407	38 cm 70 cm	38.9 60.4	25,300 21,100
Center of site, well	772440 10-11.5	ft 13.5	ND [†]
50 ft at 280° magnetic from a	772441 Surface	21.9	1,600
point in the RR ditch 23 ft downstream of the confluence	37 cm	24.2	1,100
with the dock ditch	72 cm	34.6	1,400

[†] ND - None detected above the detection limit of 500 mg/kg or no identification as creosote by gas chromatography.

279,000 mg/kg (27.9%) in a sample taken inside the retort room and was greater than 100,000 mg/kg (10%) at depths up to 38 cm (15 in) below the ground surface at sites outside the buildings.

The absence of creosote in some of the water samples collected and the relatively low concentrations found in wells other than the one nearest the green shed may be an indication of the low solubility of creosote or the strong tendency for creosote to adhere to the well casing and to the pump suction line. It may also be an indication that the wells were placed at the edge of a plane of higher concentration of contaminants. This latter possibility is thought most likely because the wells could not be drilled in optimum locations due to poor surface conditions which precluded ready access.

The high concentrations of creosote found in the groundwater and in the soil at all levels, particularly at the surface, indicate that the pollution of the Little Menomonee River will continue if corrective measures are not taken. It seems likely that a ditch dug along the north and east boundaries of the study area would intercept both groundwater and surface runoff. The creosote thus intercepted could be removed by skimming and sediment removal prior to release of the water to the River. If soluble materials in the water orove to be a problem, it may be necessary to treat the water with activated carbon prior to discharge.

APPENDIX A

WELL LOGS, CASING AND PERFORATION RECORD AND FIELD NOTES 9/19-20/77

APPENDIX A

WELL LOGS, CASING AND PERFORATION RECORD AND FIELD NOTES, 9/19-20/77

KEY TO WELL LOGS



Fill, Cinders, gravel, clay and silt

Till, gray, clayey silt, peat, vari-colored clay

Gravel and sand, dense, sandy

Peat, dark gray

Total Depth



Record of casing depth and perforations.

Record of surface of water level. Other notes on field logs are self-explanatory.

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APPENDIX B CHAIN-OF-CUSTODY PROCEDURES

ENVIRONMENTAL PROTECTION AGENCY NATIONAL ENFORCEMENT INVESTIGATIONS CENTER

CHAIN OF CUSTODY PROCEDURES June 1, 1975

GENERAL

The evidence gathering portion of a survey should be characterized by the minimum number of samples required to give a fair representation of the effluent or water body from which taken. To the extent possible, the quantity of samples and sample locations will be determined prior to the survey.

Chain of Custody procedures must be followed to maintain the documentation necessary to trace sample possession from the time taken until the evidence is introduced into court. A sample is in your "custody" if:

- 1. It is in your actual physical possession, or
- 2. It is in your view, after being in your physical possession, or
- 3. It was in your physical possession and then you locked it up in a manner so that no one could tamper with it.

All survey participants will receive a copy of the survey study plan and will be knowledgeable of its contents prior to the survey. A pre-survey briefing will be held to re-appraise all participants of the survey objectives, sample locations and Chain of Custody procedures. After all Chain of Custody samples are collected, a de-briefing will be held in the field to determine adherence to Chain of Custody procedures and whether additional evidence type samples are required.

SAMPLE COLLECTION

- 1. To the maximum extent achievable, as few people as possible should handle the sample.
- 2. Stream and effluent samples shall be obtained, using standard field sampling techniques.
- 3. Sample tags (Exhibit I) shall be securely attached to the sample container at the time the complete sample is collected and shall contain, at a minimum, the following information: station number, station location, data taken, time taken, type of sample, sequence number (first sample of the day sequence No. 1, second sample sequence No. 2, etc.), analyses required and samplers. The tags must be legibly filled out in ballpoint (waterproof ink).
- 4. Blank samples shall also be taken with preservatives which will be analyzed by the laboratory to exclude the possibility of container or preservative contamination.
- 5. A pre-printed, bound Field Data Record logbook shall be maintained to record field measurements and other pertinent information necessary to refresh the sampler's memory in the event he later takes the stand to testify regarding his actions during the evidence gathering activity. A separate set of field notebooks shall be maintained for each survey and stored in a safe place where they could be protected and accounted for at all times. Standard formats (Exhibits II and III) have been established to minimize field entries and include the date, time, survey, type of samples taken, volume of each sample, type of analysis, sample numbers, preservatives, sample location and field measurements such as temperature, conductivity,

- DO, pH, flow and any other pertinent information or observations. The entries shall be signed by the field sampler. The preparation and conservation of the field logbooks during the survey will be the responsibility of the survey coordinator. Once the survey is complete, field logs will be retained by the survey coordinator, or his designated representative, as a part of the permanent record.
- 6. The field sampler is responsible for the care and custody of the samples collected until properly dispatched to the receiving laboratory or turned over to an assigned custodian. He must assure that each container is in his physical possession or in his view at all times, or locked in such a place and manner that no one can tamper with it.
- 7. Colored slides or photographs should be taken which would visually show the outfall sample location and any water pollution to substantiate any conclusions of the investigation. Written documentation on the back of the photo should include the signature of the photographer, time, date and site location. Photographs of this nature, which may be used as evidence, shall be handled recognizing Chain of Custody procedures to prevent alteration.

TRANSFER OF CUSTODY AND SHIPMENT

- 1. Samples will be accompanied by a Chain of Custody Record which includes the name of the survey, samplers' signatures, station number, station location, date, time, type of sample, sequence number, number of containers and analyses required (Fig. IV). When turning over the possession of samples, the transferor and transferee will sign, date and time the sheet. This record sheet allows transfer of custody of a group of samples in the field, to the mobile laboratory or when samples are dispatched to the NEIC Denver laboratory. When transferring a portion of the samples identified on the sheet to the field mobile laboratory, the individual samples must be noted in the column with the signature of the person relinquishing the samples. The field laboratory person receiving the samples will acknowledge receipt by signing in the appropriate column.
- 2. The field custodian or field sampler, if a custodian has not been assigned, will have the responsibility of properly packaging and dispatching samples to the proper laboratory for analysis. The "Dispatch" portion of the "Chain of Custody Record shall be properly filled out, dated, and signed.
- Samples will be properly packed in shipment containers such as ice chests, to avoid breakage. The shipping containers will be padlocked for shipment to the receiving laboratory.
- 4. All packages will be accompanied by the Chain of Custody Record showing identification of the contents. The original will accompany the shipment, and a copy will be retained by the survey coordinator.
- 5. If sent by mail, register the package with return receipt requested. If sent by common carrier, a Government Bill of Ladıng should be obtained. Receipts from post offices, and bills of ladıng will be retained as part of the permanent Chain of Custody documentation.
- 6. If samples are delivered to the laboratory when appropriate personnel are not there to receive them, the samples must be locked in a designated area within the laboratory in a manner so that no one can tamper with them. The same person must then return to the laboratory and unlock the samples and deliver custody to the appropriate custodian.

LABORATORY CUSTODY PROCEDURES

- 1. The laboratory shall designate a "sample custodian." An alternate will be designated in his absence. In addition, the laboratory shall set aside a "sample storage security area." This should be a clean, dry, isolated room which can be securely locked from the outside.
- 2. All samples should be handled by the minimum possible number of persons.
- 3. All incoming samples shall be received only by the custodian, who will indicate receipt by signing the Chain of Custody Sheet accompanying the samples and retaining the sheet as permanent records. Couriers picking up samples at the airport, post office, etc. shall sign jointly with the laboratory custodian.
- 4. Immediately upon receipt, the custodian will place the sample in the sample room, which will be locked at all times except when samples are removed or replaced by the custodian. To the maximum extent possible, only the custodian should be permitted in the sample room.
- 5. The custodian shall ensure that heat-sensitive or light-sensitive samples, or other sample materials having unusual physical characteristics, or requiring special handling, are properly stored and maintained.
- Only the custodian will distribute samples to personnel who are to perform tests.
- 7. The analyst will record in his laboratory notebook or analytical worksheet, identifying information describing the sample, the procedures performed and the results of the testing. The notes shall be dated and indicate who performed the tests. The notes shall be retained as a permanent record in the laboratory and should note any abnormalties which occurred during the testing procedure. In the event that the person who performed the tests is not available as a witness at time of trial, the government may be able to introduce the notes in evidence under the Federal Business Records Act.
- 8. Standard methods of laboratory analyses shall be used as described in the "Guidelines Establishing Test Procedures for Analysis of Pollutants," 38 F.R. 28758, October 16, 1973. If laboratory personnel deviate from standard procedures, they should be prepared to justify their decision during cross-examination.
- 9. Laboratory personnel are responsible for the care and custody of the sample once it is handed over to them and should be prepared to testify that the sample was in their possession and view or secured in the laboratory at all times from the moment it was received from the custodian until the tests were run.
- 10. Once the sample testing is completed, the unused portion of the sample together with all identifying tags and laboratory records, should be returned to the custodian. The returned tagged sample will be retained in the sample room until it is required for trial. Strip charts and other documentation of work will also be turned over to the custodian.
- 11. Samples, tags and laboratory records of tests may be destroyed only upon the order of the laboratory director, who will first confer with the Chief, Enforcement Specialist Office, to make certain that the information is no longer required or the samples have deteriorated.

EXHIBIT I

Station No.	Date	Time	Sequence No.
Station No. Station Lo	cation	_ !	Grab
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ENVIRONMENTAL PROTECTION AGENCY

OFFICE OF ENFORCEMENT

NATIONAL ENFORCEMENT INVESTIGATIONS CENTER

BUILDING 53, BOX 25227, DENVER FEDERAL CENTER

DENVER, COLORADO 80225



Back

EXHIBIT II

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EXHIBIT III

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ENVIRONMENTAL PROTECTION AGENCY Office Of Enforcement

NATIONAL ENFORCEMENT INVESTIGATIONS CENTER
Building 53, Box 25227, Denver Federal Center
Denver, Colorado 80225

CHAIN OF CUSTODY RECORD

SURVEY				SAMPLERS: (Signature)									
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APPENDIX C SOIL AND WATER SAMPLE ANALYSES

CONFIRMATION OF CREOSOTE IN WATER AND SOIL SAMPLES FROM THE KERR-MCGEE/MOSS-AMERICAN PLANT SITE MILWAUKEE, WISCONSIN BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY

Analyses of the water extract and soil extract from Kerr-McGee confirming creosote were completed. The samples were screened initially on the flame ionization gas chromatograph, then analyzed utilizing combined gas chromatography/mass spectrometry (GC/MS). The gas chromatograms readily confirmed the presence of creosote when the chromatogram of the standard was compared to the water sample extract and the soil extract. The major sample components in samples and standard all occurred in the same ratio. The mass spectra of the individual compounds in the standard creosote mix and standard spectra of individual compounds run using the same instrumental conditions as the samples were perfect matches and all were confirmed by mass spectrometry and matching gas chromatography retention times. Table I gives the results for the samples and the standard mix.

TABLE I

	GC/MS Identification						
	Commercial	Sample	Sample				
	Creosote	2311	2240				
Compound	<u>Local</u>	<u>Soil</u>	<u>Water</u>				
Napthalene	CF(1)	CF	CF				
2-methylnaphthalene	CF	CF	CF				
l-methylnaphthalene	CF	CF	CF				
Bipheny1	CF	CF	CF				
1,3-dimethylnaphthalene	CF	CF	CF				
Acenaphthene	CF	CF	CF				
Dibenzofuran	CF	CF	CF				
Fluorene	CF	CF	CF				
Anthracene	CF	CF	CF				
(Phenanthrene)	CF(2)	CF	CF				
Carbazole	CF	CF	CF				
2-methylphenanthrene	Good (3)	Good	Good				
Fluoranthene	CF	CF	CF				
Pyrene	CF	CF	CF				

Note: CF - confirmed

- (1) These compounds confirmed by GC/MS identification, GC/MS of standards, and coincidence of retention times with those of standards.
- (2) Phenanthrene and anthracene have identical mass spectra and the gas chromatography retention times are only slightly different, with phenanthrene eluting later. It appears as a definite shoulder on the anthacene peak with the GC conditions used when more dilute solutions are run. Both compounds are obviously present.
- (3) This compound was identified by mass spectrometry only. A standard was not available.

ANALYTICAL METHODOLOGY - SOIL, WELL CORE AND GROUNDWATER SAMPLES COLLECTED SEPTEMBER 19-20, 1977 AT THE KERR-MCGEE/MOSS-AMERICAN PLANT SITE MILWAUKEE, WISCONSIN

On September 23, 1977, 6 water and 113 soil samples were received at the NEIC Laboratory for creosote analysis. All samples were handled according to chain-of-custody procedures. The waters and soils were analyzed for methylene chloride extractables and the soils for moisture content. The methylene chloride extracts were also analyzed for the presence of creosote compounds by gas chromatography. The results are summarized in Tables 5 and 7 in the body of the report.

Methodology:

A. Moisture

About 10 grams of thoroughly mixed sample was accurately weighed in a tared 50 ml beaker and dried overnight in an oven at 105°C. The water loss was determined by reweighing the cooled and desiccated beakers. Calculations:

Wt. of water loss x 100 = % moisture
Wt. of sample wet

B. Methylene Chloride Extractables

Soils - 10 grams of thoroughly mixed soil was weighed into a 150 ml beaker. Large stones, twigs, leaves, etc. were avoided. 30 grams of granular anhydrous sodium sulfate was added to the beaker and the soil and sodium sulfate were mixed thoroughly to obtain a coarse granular consistency. The mixture was then transferred to a 33 x 80 mm cellulose extraction thimble and placed in a Soxhlet extractor. 200 ml of methylene chloride was placed in a 500 ml flat-bottomed flask and attached to the extractor. The extractor was allowed to cycle for 2 to 3 hours, with a rate of about 10 cycles per hour. Each flask was then placed on a rotary evaporator and the solution was concentrated to a volume of about 20 ml. The remaining solvent was quantitatively transferred to a tared 50 ml beaker and evaporated to dryness on a warm hot plate under a gentle stream of carbon-filtered air. Each beaker was reweighed and the residue determined. Results were calculated on a dry weight basis using % moisture values.

Wt. of residue in mg
Wt. of sediment extracted wet (gm) x wt fraction solids
mg/kg dry basis

wt fraction solids = 1.00 - wt fraction moisture

Waters - From 100 ml to 3 liters of the water samples were extracted twice with 100 ml of methylene chloride. The extracts were combined and evaporated to dryness in tared beakers on a warm hot plate under a gentle stream of carbon-filtered air. Each beaker was reweighed and the residue determined.

C. Creosote Compounds

Creosote is a complex mixture of compounds, some of which can be detected on a flame ionization gas chromatograph. These compounds give a distinctive pattern of peaks. This pattern was used to identify the presence of creosote.

The methylene chloride extracts were dissolved in 10 ml of acetone and an aliquot was injected on a Hewlett-Packard 5700A gas chromatograph equipped with an automatic sampler. Chromatograms were compared with a "standard" creosote sample that was purchased locally. The standard creosote sample was labelled as containing 60% creosote by weight. The sample was cut with a light refined petroleum-based oil.

Some samples with methylene chloride residues as high as 7,000 mg/kg showed no creosote pattern by gas chromatography. These residues could be caused by either "natural background" material, noncreosote compounds which have been extracted by methylene chloride, or creosote compounds which are not chromatographable. The samples which contained detectable amounts of methylene chloride residue, but which exhibited no creosote pattern, were reported as None Detected.

ANALYTICAL METHODOLOGY GROUNDWATER SAMPLES COLLECTED OCTOBER 19, 1977 AT THE KERR-MCGEE/MOSS-AMERICAN PLANT SITE MILWAUKEE, WISCONSIN

On October 21, 1977 seven plant site well samples were delivered to the laboratory in accordance with NEIC chain-of-custody procedures. The samples were analyzed for total methylene chloride extractables and the extracts screened by flame ionization detection - gas chromatography (FID-GC) to determine the presence of creosote material.

FID-GC analysis showd that samples from Stations 772413, 772419, 772431, and 772452 did not contain any observable creosote material. All other samples contained observable amounts of creosote. The results of these analyses are presented in Table 5 in the body of the report.

Methodology:

A. Moisture

About 10 grams of thoroughly mixed sample was weighed in a tared 50 ml beaker and dried overnight in an oven at 105°C. The water loss was determined by reweighing the cooled and desiccated beakers. Calculations:

Wt. of water loss x 100 = % moisture Wt. of sample wet

B. Methylene Chloride Extractables

Waters - From 1,000 ml to 3.5 liters of the water samples were extracted twice with 100 ml of methylene chloride. The extracts were combined and evaporated to dryness in tared beakers on a warm hot plate under a gentle stream of carbon-filtered air. Each beaker was reweighed and the residue determined.

C. Creosote Compounds

Creosote is a complex mixture of compounds, some of which can be detected on a flame ionization gas chromatograph. These compounds give a distinctive pattern of peaks. This pattern was used to identify the presence of creosote.

The methylene chloride extracts were dissolved in 10 ml of acetone and an aliquot was injected on a Hewlett-Packard 5700A gas chromatograph equipped with an automatic sampler. Chromatograms were compared with a "standard" creosote sample that was purchased locally. The standard creosote sample was labelled as containing 60% creosote by weight. The sample was mixed with a light refined petroleum-based oil.

G.C. Conditions

Instrument: Hewlett Packard Model 5700

equipped with a dual flame ionization detector and

automatic sampler

Column: 2mm ID X 3.3 M stainless steel

6% OV-101 on 60/80 GCQ

Instrument Conditions: Carrier Gas - Helium - 25 ml/min flow Oven temp: 80°C-230°C programmed at 8°C/min

with hold at 230°C for 16 min. Injector: 250°C

Detector: 250°C