I. Objective

Six soil core samples were collected in the vicinity of the Wolverine Company in Grand Rapids, Michigan, by the Eastern District Office of the Environmental Protection Agency. The area from which the samples were taken was believed to be contaminated with a variety of organic compounds. The six samples (CRL #'s 79-EP11S01 to S06) arrived at the Central Regional Laboratory on October 25, 1978 along with an accompanying letter requesting that they be analyzed for the following parameters: PCB's, GC-MS organic scan, and the characterization of each sample's organic matrix. This report summarizes the analytical procedures used and the results obtained for the analysis of these samples.

II. Results

Four of the core samples (CRL #79-EP11S01→S04) contained very similar organic matrices. Among the compounds detected in these samples were: polyclorinated biphenyls, substituted benzenes, naphthalene, substituted naphthalenes, several different phthalates and other polynuclear aromatics (e.g. pyrene). Concentrations for these compounds ranged from 70 µg/kg (pyrene in 78-EP11S03) to 60,200 µg/kg (alkyl benzene in 78-EP11S03). The two other samples (78-EP11S05→S06) did not contain the above compounds in detectable quantities. One compound was found to be present in all six samples. Although the exact structure of this compound could not be verified by spectrum match, it is proposed that it is an hydroxy ketone having the empirical formula $C_8H_{16}O_2$ (MW = 144). The organic composition of each sample is summarized in Tables I and II. The organic matrix in samples 79-EP11S01→S04 are very similar to each other and appear to be that of a light fuel oil.

III. Methodology

The six sediment samples were prepared for analysis by initially allowing the samples to air dry overnight at room temperature. The following day each sample was pulverized and sieved (mesh 40/60). Fifty grams of each

sieved sample was transferred to a soxhlet flask and allowed to extract continuously for 16 hours with a 50/50 (V/V) acetone/hexane solution. The organic extract was then concentrated on a steam bath to less than 5 ml using a Kuderna-Danish flask equipped with a 10 ml receiver and snyder column. Each concentrated extract was then analyzed by gas chromatography using a flame ionization detector to characterize each sample's organic matrix. Qualitative and quantitative analyses were performed by gas chromatography/mass spectroscopy and by dual column gas chromatography with electron capture detection.

IV. Quality Control

To insure the Quality of the analysis, a reagent blank and a sample spike were analyzed at the same time. The sample spike consisted of adding several suspected organics to a portion of one of the samples (CRL #79-EP11S04). Analysis of the blank did not show detectable organics and acceptable recoveries were obtained with the spiked sample.

V. Conclusions

By referring to the sampler's map (Figure I) which designates the area where the samples were taken, one can see that the majority of the organic compounds were found in the sediments sampled to the North and Northwest of the Wolverine Solvent Company. The samples taken Northeast (79-EP11S05) and South-Southeast of this company contained only one detectable compound (the hydroxy-ketone $C_8H_{16}O_2$).

VI. Participants

Mr. Robert Glowacky, Chemist
Dr. Sukwha Kim, GC/MS Expert
Dr. Chacko Joseph, Pesticide Team Leader
Dr. Emilio Sturino, Author

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TABLE II

District Office Eastern - Tennessee Tk Sampling Date 10/18 10 78 Lab Arrival Date 25 Oct 78 Analysis Due Date As Soon As Pc

Custody

General Qualitative and Quantitative Organic Analyses

Account No. [illegible] Study Oil Spill - Toxics

Fy-79 Lab Number	Compound 1242 Units =	Conc Mg/kg	Compound 1248 Units =	Conc Mg/kg	Compound 1254 Units =	Conc Mg/kg	Compound 1260 Units =	Conc Mg/kg	Compound Units =	Conc
EP115										
1 01	<K	= 0.04	2.65		<K	= 0.04	<K	= 0.04		
1 02	<K	= 0.04	0.8		0.3					
1 03	<K	= 0.04	1.1		0.4					
1 04	<K	= 0.04	.3		<K	= 0.04				
1 05	K	= 0.04	<K	= .04	K	= 0.04				
1 06	<K	= 0.04	<K	= 0.04	<K	= 0.04				
1										
1										
1										
1										
1										
10										

Pc. [illegible] 11-15-78

MONS 002627

TABLE I

Concentrations of Organics Found in Core Sediment Samples Taken in Grand Rapids, Michigan (units = ug/kg)

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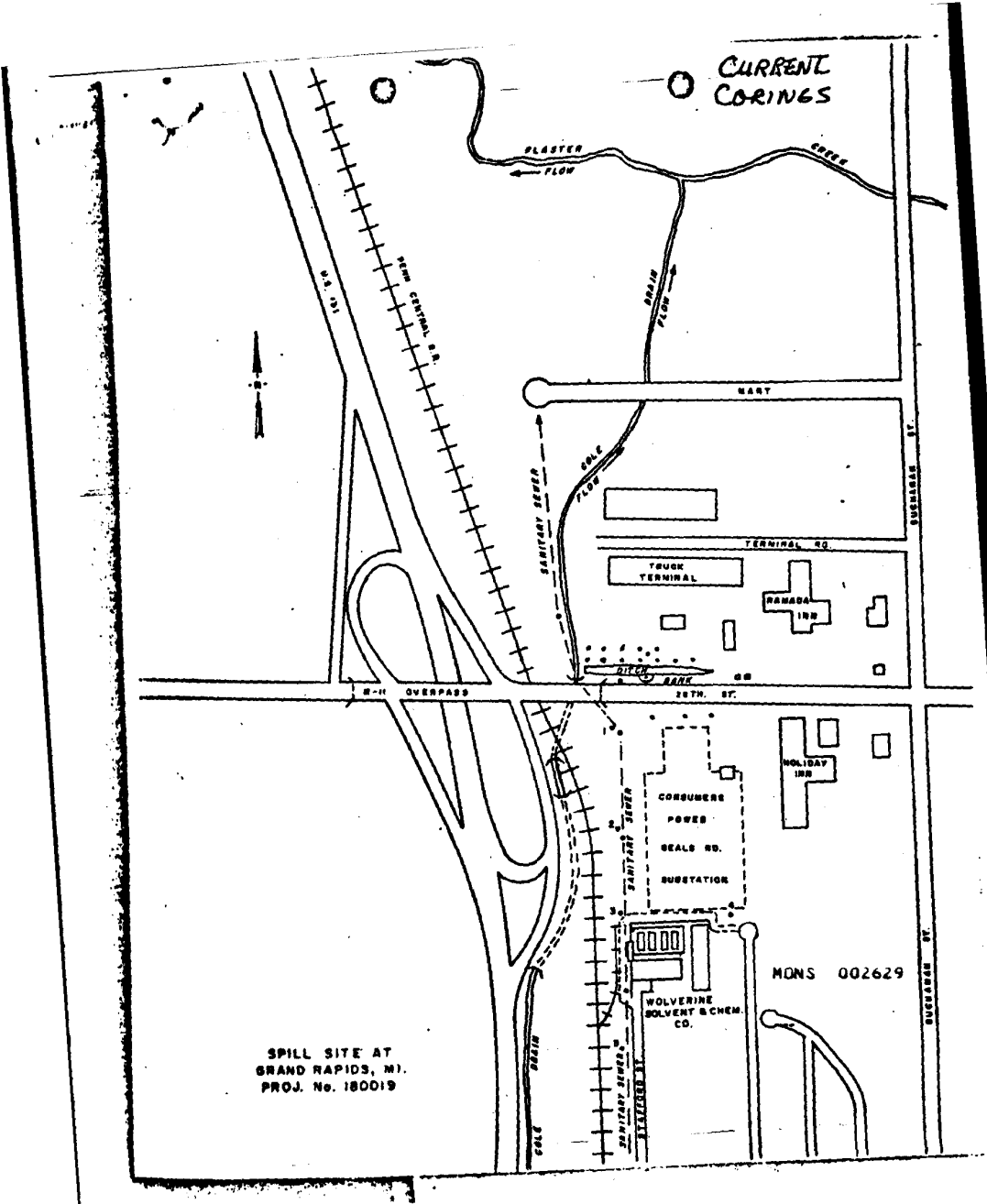
Compound	#4th of 78-EP11S01	#1 78-EP11S02	#2 78-EP11S03	#3 78-EP11S04	#4 78-EP11S05	#5 78-EP11S06
Xylene or Ethyl Benzene	116	< 50	2,400	< 50	< 50	< 50
Propyl Benzene Trimethyl Benzene or Methyl Ethyl Benzene	94	230	23,084	200	< 50	< 50
Butyl Benzene Propylmethyl Benzene Tetramethyl Benzene or Diethyl Benzene	6,600	990	60,200	1,250	< 50	< 50
Pentamethyl Benzene Diethyl Methyl Benzene Propyl Ethyl Benzene Butyl Methyl Benzene Pentyl Benzene	4480	690	19,300	170	< 50	< 50
Naphthalene	5570	670	18,000	1,200	< 50	< 50
Methyl Naphthalene(s)	3900	590	17,200	790	< 50	< 50
Dimethyl Naphthalene(s)	7,500	680	16,600	430	< 50	< 50
Anthracene or Phenanthrene	310	120	282	160	< 50	< 50
Fluoranthene	70	< 50	100	70	< 50	< 50
Pyrene	< 50	< 50	70	< 50	< 50	< 50
Butyl Phthalate	5,200	1,370	4,250	570	< 50	< 50
Bis (2 Ethylhexyl) Phthalate	24,000	15,700	15,800	2,360	< 50	< 50
Phthalate *	18,400	9,800	16,000	2,400	< 50	< 50
$C_8H_{16}O_2$ ** (Hydroxy Ketone)	36,200	10,400	24,100	3,550	2,140	2,300

* - Quantitated Using the Response of a (2-Ethylhexyl) Phthalate Standard
** - Quantitated Using the Response of a T Butyl Benzene Standard


CURRENT CORINGS
PLASTER CREEK
FLOW
BRAIN FLOW
U.S. 131
PENN CENTRAL R.R.
MART
SANITARY SEWER
GALE FLOW
TERMINAL RD.
DIVERSEY ST.
TRUCK TERMINAL
RAMADA INN
DITCH
BANK
28TH. ST.
M-11 OVERPASS
HOLIDAY INN
CONSUMERS POWER
SEALS RD.
SUBSTATION
WOLVERINE SOLVENT & CHEM. CO.
MONS 002629
SPILL SITE AT
GRAND RAPIDS, MI.
PROJ. No. 180019

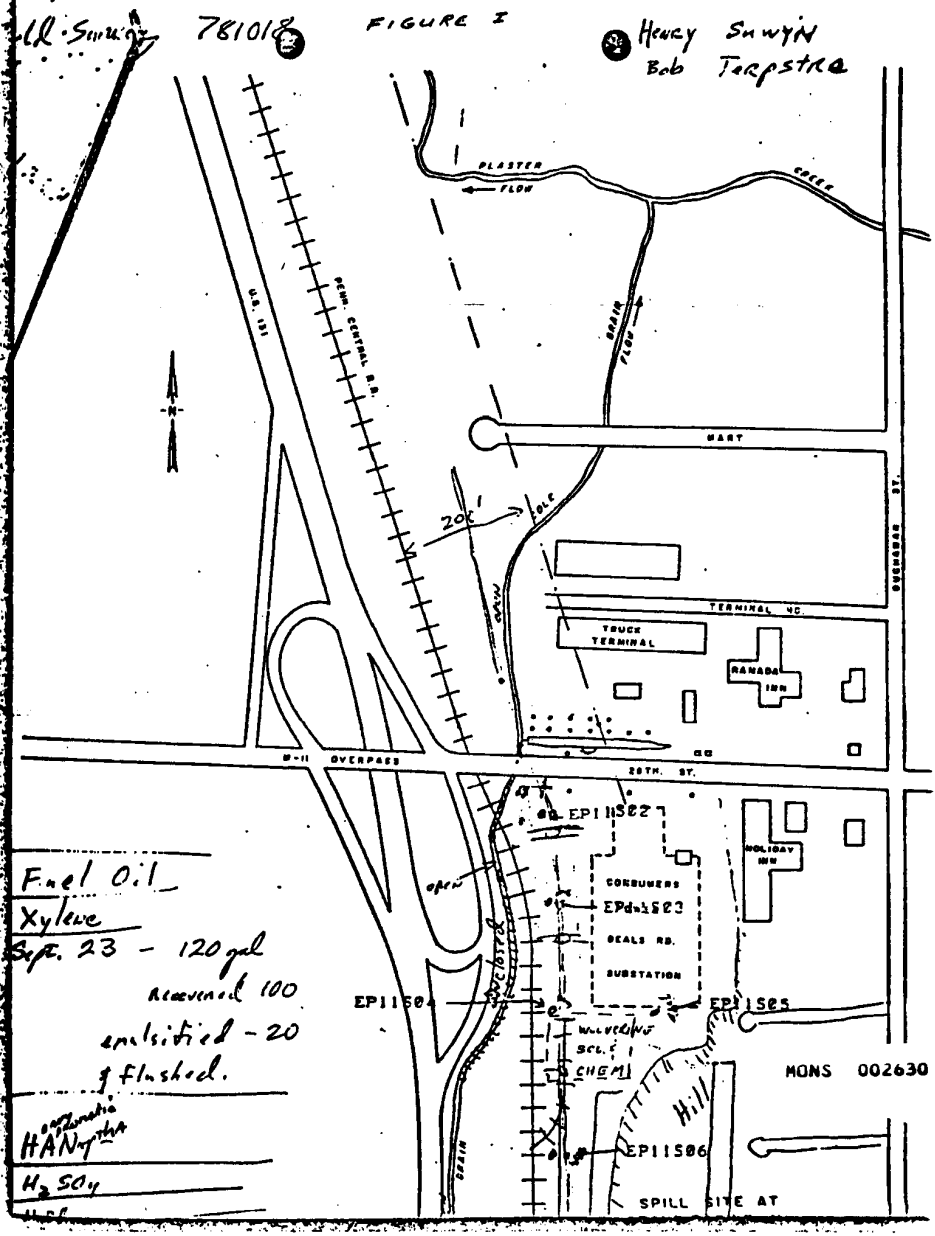


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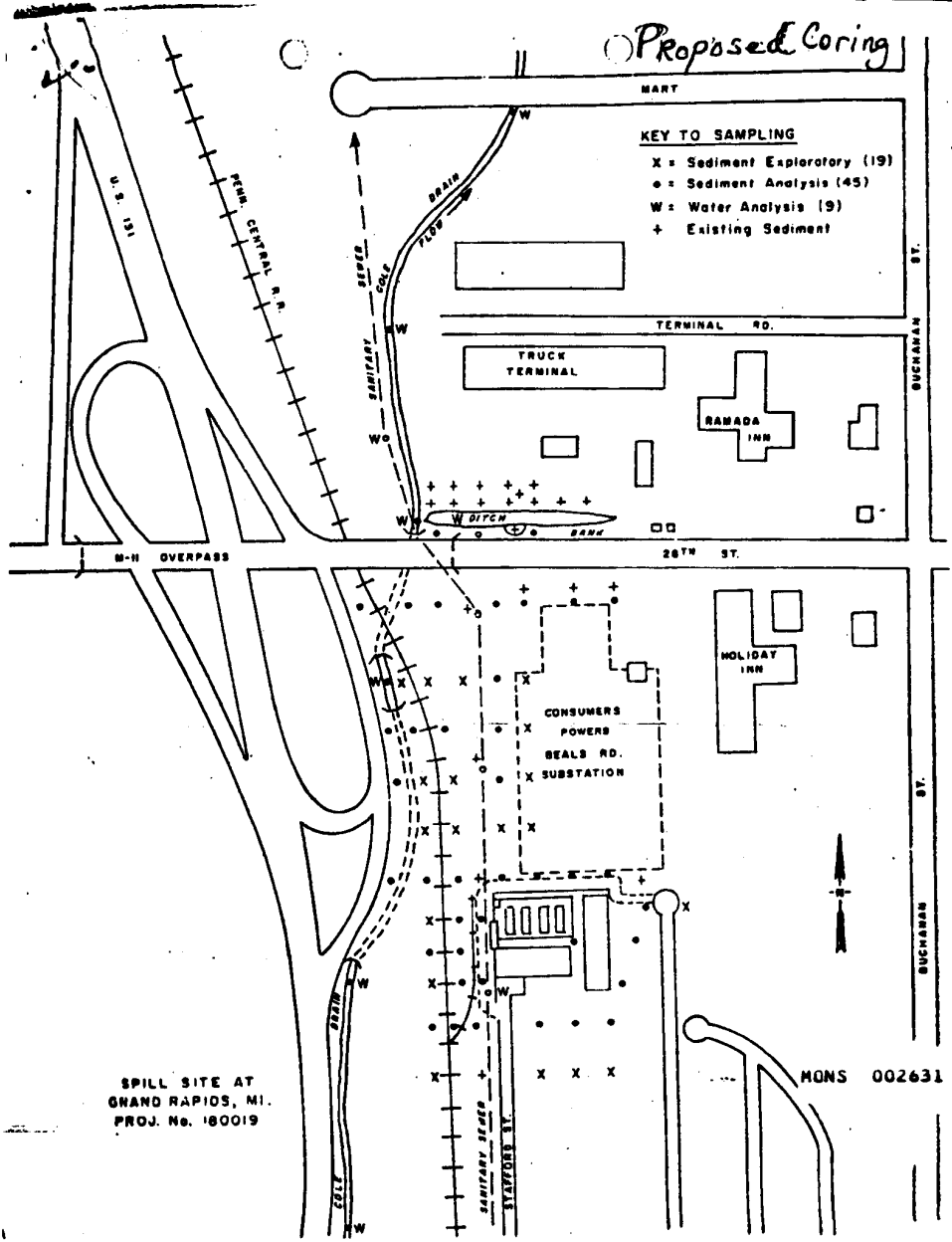
FIGURE I
Henry Suwyn
Bob Terpstra
PLASTER
FLOW
CREEK
U.S. 131
PENN. CENTRAL R.R.
GRAIN
FLOW
MART
DUGHAMAN ST.
TERMINAL RD.
TRUCK TERMINAL
RANADA INN
M-11 OVERPASS
28TH. ST.
EP11502
HOLIDAY INN
CONSUMERS
EPdwk503
SEALS RD.
SUBSTATION
open
closed
EP11504
EP11505
WOLVERINE
SCL.
CHEM
Hill
EP11506
GRAIN
SPILL SITE AT
MONS 002630
Fuel Oil
Xylene
Sept. 23 - 120 gal
Recovered 100
emulsified - 20
& flushed.
HANaptha
H_2SO_4


Proposed Coring
KEY TO SAMPLING
X = Sediment Exploratory (19)
• = Sediment Analysis (45)
W = Water Analysis (9)
+ Existing Sediment
MART
TERMINAL RD.
TRUCK TERMINAL
RAMADA INN
HOLIDAY INN
CONSUMERS POWERS SEALS RD. SUBSTATION
U.S. 131
PENN. CENTRAL R.R.
SEWER
COLE
DRAIN
FLOW
SANITARY
W DITCH
BANK
M-11 OVERPASS
28TH ST.
BUCHANAN ST.
STAFFORD ST.
SPILL SITE AT GRAND RAPIDS, MI.
PROJ. No. 180019
MONS 002631