

91020

ROUTING OF SHOP DRAWINGS, EQUIPMENT DATA, MATERIAL SAMPLES, OR MANUFACTURER'S CERTIFICATES OF COMPLIANCE FOR APPROVAL

(Used to route ENG Form 4025 with items attached. Not to become a part of the Contractors's record)

1

TO:	USEPA REGION III MAIL CODE 3HW22 841 CHESTNUT BUILDING PHILA., PA. 19107	FROM:	OHM REMEDIAL SERVICES CORPORATION 180 MYRTLE STREET LOCK HAVEN, PA 17745	DATE	SEPTEMBER 10, 1997
	ATTN: GREG CRYSTAL		ATTN: TONY GARCIA		

The attached items listed on ENG Form 4025 are forwarded for approval action

CONTRACT NUMBER	DACW45-93-C-0200	CONTRACTOR	OHM REMEDIAL SERVICES CORPORATION
TRANSMITTAL NUMBERS	202	PROJECT TITLE AND LOCATION	DRAKE CHEMICAL SUPERFUND SITE, LOCK HAVEN, PA.

COMMENTS (Attach additional sheet, if necessary.)

RISK BURN CONDITION NO. 1 UNCHARACTERIZED CHOMATOGRAPHAPHABLE FRACTION REPORT

NO. OF INCL.	TYPED NAME AND TITLE	SIGNATURE
15 TO EPA 5 TO AREA	ANTHONY T. GARCIA QUALITY CONTROL MANAGER	<i>Anthony T. Garcia</i>
TO:	FROM:	DATE

2
COMMENTS (Attach additional sheet, if necessary.)

NO. OF INCL.	TYPED NAME AND TITLE	SIGNATURE
TO:	FROM:	DATE

3
COMMENTS (Attach additional sheet, if necessary.)

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4
The following action codes are given to items listed on ENG Form 4025:

ACTION CODES

- | | |
|---|--|
| A - APPROVED AS SUBMITTED. | D - WILL BE RETURNED BY SEPARATE CORRESPONDENCE. |
| B - APPROVED, EXCEPT AS NOTED ON DRAWINGS.
RESUBMISSION NOT REQUIRED. | E - DISAPPROVED (SEE ATTACHED) |
| C - APPROVED, EXCEPT AS NOTED ON DRAWINGS.
REFER TO ATTACHED SHEET. RESUBMISSION REQUIRED. | F - RECEIPT ACKNOWLEDGED |
| | G - OTHER (specify) |

ACTION CODES TO BE INSERTED IN COLUMN G, SECTION I, ENG FORM 4025 (Attach sheets, when required.)

ITEM NO.
(Taken from ENG Form 4025)

CODE GIVEN

REMARKS

NO. OF INCL.	TYPED NAME AND TITLE	SIGNATURE

**Drake Chemical Superfund Site
Lock Haven, PA**

REPORT ON ESTIMATION OF
UNCHARACTERIZED FRACTION OF
CHROMATOGRAPHABLE ORGANICS FOR
RISK BURN
Condition 1 - Run 3

This data has not been subjected to final QA/QC

The draft data quality cannot be assured for the intended final use
until final QA/QC has been performed

AR315307

**Report on Estimation of Uncharacterized Fraction
of Chromatographable Organics for
Risk Burn Condition 1—Run 3**

For OHM Remediation Service Corp.

OHM Subcontract No. 292521-02

MRI Project No. 3620-50-21

September 5, 1997

MRI REPORT

DRAFT

Report on Estimation of Uncharacterized Fraction of Chromatographable Organics for Risk Burn Condition 1—Run 3

For OHM Remediation Service Corp.
180 Myrtle Street
Lock Haven, Pennsylvania 17745

Attn: Mr. Gary Jones
Technical Manager

OHM Subcontract No. 292521-02

MRI Project No. 3620-50-21

September 5, 1997

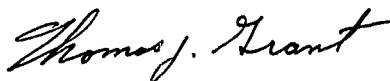
Preface

Midwest Research Institute (MRI) carried out this work, to estimate the uncharacterized fraction of chromatographable organics for Run 3 of Risk Burn Condition 1, as directed by OHM in their letter dated August 11, 1997. This report presents the results of that work.

Mr. Gary Jones was the technical manager for OHM Remediation Services Corp. and Mr. Paul Gorman was the project leader for MRI. Dr. Vincy Abraham and Mr. Mike Molloy were the task leaders responsible for the completion and evaluation of all the GC/MS data.

Approved for:

MIDWEST RESEARCH INSTITUTE



Thomas J. Grant, Ph.D., P.E.
Director
Applied Engineering

September 5, 1997

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Section 1

Introduction

The purpose of this work was to estimate the percentage of the uncharacterized chromatographable organics in the stack gas during Risk Burn Condition 1 using Run 3 at the OHM Chemical Superfund site. The uncharacterized chromatographable organics is defined as the total chromatographable organics minus target analytes and tentatively identified compounds (TICs).

The work was accomplished by reexamining the full scan GC/MS data for the volatile organics (VOST - Condensate and 4 Trap Pairs) and the semivolatile organics (MM5-SV - Filter/XAD, Rinse and Condensate samples). The following summarizes the assumptions and steps used to determine the uncharacterized fraction:

- All compounds were considered to have an instrument relative response factor of 1.0 based on the average response for the internal standards.
- Area counts for each sample and blank were obtained via careful review of the GC/MS output.
- The sample area counts were corrected for the area counts in the associated blank.
- The blank corrected sample area counts were converted to mass/sample using an appropriate response value (nanograms in sample per million counts in sample aliquot).
- The target analyte mass and TIC mass were subtracted from the total sample mass to obtain the uncharacterized mass.
- The uncharacterized chromatographic volatile organic and semivolatile organic fractions were determined by dividing the uncharacterized mass by the sample mass.

- The total uncharacterized fraction was computed by converting the VOST and MM5-SV total and uncharacterized masses to concentrations and then calculating the uncharacterized percentage.

A summary of results is given in Section 2, and details of the procedures used are given in Section 3.

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Section 2

Summary of Results

The uncharacterized chromatographic fraction for Risk Burn Condition 1 Run 3 was estimated to be 9 percent. Development of this estimate is documented in the next section of this report.

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Section 3

Procedure and Tabulation of Data

The procedure used to determine the uncharacterized fraction is summarized in the following steps:

1. The area counts (total injection, internal standards, surrogates, target analytes and TICs) for each of the samples and blanks were obtained from the VOST and MM5-SV Full Scan GC/MS data. The term "VOST" refers to chromatographable volatile organic compounds and "MM5-SV" refers to chromatographable semivolatile organic compounds.

Samples used:

VOST: Four (4) trap pairs and associated trap pair field blank.
Condensate sample and associated laboratory method blank.

MM5-SV: Filter/XAD sample and associated Filter/XAD field blank.
Front half/Back half rinse sample and associated FH/BH field blank.
Condensate sample and associated condensate field blank.

Data is presented in Tables 1 (VOST) and 5 (MM5-SV).

2. The area counts for the samples (total, internal standards, surrogates, target analytes and TICs) were normalized to the area counts for the blanks, based on the internal standards, such that the response factor for sample and blank are the same and all measurements are placed on an equivalent basis.

The normalization formula used:

$$\text{normalized area count} = \text{area count} \times \frac{\text{Blank internal standards area count (ISC}_b\text{)}}{\text{Sample internal standards area count (ISC}_s\text{)}}$$

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Data is presented in Tables 2 (VOST) and 6 (MM5-SV).

3. The area counts of the sample and blank were determined by subtracting the normalized area counts for internal standards (ISC) and surrogates (SCC) from the normalized total area count.

Data is presented in Tables 2 (VOST) and 6 (MM5-SV).

4. The individual constituents (sample, target analytes & TICs) for the samples were blank corrected by subtracting the blank area counts from the normalized sample area count for each constituent. Where the blank constituent area count was greater than the normalized constituent area count for the sample, the blank corrected area count was treated as zero (e.g., negative area counts for individual constituents were not allowed.)

Data presented in Tables 3 (VOST) and 7 (MM5-SV).

5. Response values were developed for each blank sample type. These response values were developed by calculating an instrument response factor and multiplying the instrument response factor by any sample split factor. The instrument response factor was calculated by dividing the mass (nanograms) of internal standards by the area count for the internal standards. This approach is based on treating each analyte group (internal standards, surrogates, target analytes, TICs and uncharacterized constituents) as if they have a relative response factor of 1.0. While this is not strictly true for the target analytes, it is the most technically sound approach to put all constituents on an equivalent basis and it is the accepted standard practice when the response is unknown and must be estimated because it is not measurable. The split factor is based on the volume of sample injected into the instrument versus the total sample volume. Development of the response values is shown in Appendix A.

Data is presented in Tables 1 (VOST) and 5 (MM5-SV).

6. The individual blank corrected constituents (sample, target analytes & TICs) for the samples were then converted to mass (nanograms) in the constituent sample using the response values developed in Step 5.

Data is presented in Tables 4 (VOST) and 8 (MM5-SV).

7. The VOST blank corrected samples were added together to obtain the blank corrected VOST sample mass for the constituents (sample, target analytes, and TICs).

Data is presented in Table 4.

8. The VOST blank corrected uncharacterized mass was determined by subtracting the blank corrected target analyte and TIC mass from the blank corrected sample mass.

Data is presented in Table 4.

9. The blank corrected VOST uncharacterized fraction was determined by dividing the blank corrected uncharacterized mass by the blank corrected sample mass.

Data is presented in Table 4.

10. Steps 7 through 9 were repeated for the MM5-SV samples.

Data for MM5-SV steps 7 through 9 is presented in Table 8.

11. To determine the total (volatile + semivolatile) uncharacterized fraction the blank corrected VOST and MM5-SV sample and uncharacterized fraction masses were converted to concentrations by dividing the mass by the stack gas sample volume.

Data is presented in Table 9.

12. Blank corrected VOST results (sample and uncharacterized concentrations) were added to blank corrected MM5-SV results (sample and uncharacterized concentrations) to obtain total blank corrected results (sample and uncharacterized concentrations).

Data is presented in Table 9.

13. The total uncharacterized fraction was calculated by dividing the blank corrected uncharacterized concentration by the blank corrected sample concentration.

Data is presented in Table 9.

3.1 Volatile Chromatographable Organics

The VOST area counts were obtained from the computer generated total ion current plots for the samples and automatically integrated using Lab Base software. Representative portions of the chromatograms and integrations were manually checked to insure that peaks were correctly integrated. The integrations were based on the total ions; not on selected quantities.

VOST data and uncharacterized fraction calculations are presented in Tables 1 through 4.

The full trap pair is desorbed during the VOST trap pair analysis which means the split factor is 1.0.

Five (5) mL of the 60.7 mL condensate sample was analyzed which results in a split factor of 12.14.

The condensate blank area count was greater than the condensate sample area count, which results in blank corrected values being treated as zero. This was due to the presence of some late eluting peaks in the chromatogram for the blank that were not present in the VOST condensate sample.

3.2 Semivolatile Chromatographable Organics

The MM5-SV area counts were obtained from the computer generated total ion current plots for the samples and automatically integrated using Lab Base software. Representative portions of the chromatograms and integrations were manually checked to insure that

peaks were correctly integrated. The integrations were based on the total ions, not on selected quantitation ions.

MM5-SV data and uncharacterized fraction calculations are presented in Tables 4 through 8.

The filter/XAD, FH/BH rinse and condensate each have the same split factor. In each case the full sample was extracted, the extract was concentrated to a volume of 1 mL (1,000 μ L) and 2 μ L was injected into the GC. This results in a split factor of 500.

3.3 VOST and MM5-SV Combined

The total mass data and uncharacterized mass for VOST (from Table 4) and for MM5-SV (from Table 8) were entered into Table 9 along with the respective volumes of gas sampled by each method, in order to calculate the total sample mass concentration and the mass concentration of the uncharacterized portion, for VOST and MM5 individually. It was necessary to convert the mass units to concentration units because the volume of gas sampled is very different for these two sampling methods. Thus, the mass values cannot simply be added together for the two methods.

The two pair of mass concentration values were added together, as shown in the last two columns of Table 9. The resulting two overall mass concentration values were used to calculate the overall uncharacterized fraction (%) for VOST and MM5-SV combined.

Table 1. Volatile Chromatographic Organic Uncharacterized Fraction Input Data^a

Ion Current	VOST Traps						VOST Condensate ^b		Comments
	Pair 1	Pair 2	Pair 3	Pair 4	Field Blank Pair	Sample	Method Blank		
Total Injection	TC	205	184	201	194	64.6	37.4	53.4	
Internal Standards	ISC	19.0	17.3	17.4	17.3	25.8	16.7	18.0	
Surrogate Compounds	SCC	27.1	18.4	19.9	18.3	23.5	9.0	10.5	
Target Analyte	TAC	129	128	143	138	5.6	9.7	12.8	
TICs	TICC	13.5	9.8	10.3	10.0	0.2	NA	NA	TICs for condensate were not included in the Risk Burn report.
Response Value ^c (ng/10 ⁶ counts)		-----	-----	-----	-----	29.1	-----	506	

^a Ion currents shown are area counts, in millions.

^b For VOST condensate, the ion current are counts are for 5 ml aliquot.

^c Response values are used to calculate mass from area counts as shown in Table 4. Development of the response factors is presented in Appendix A.

Table 2. Volatile Chromatographic Organic Uncharacterized Fraction^a Data Normalized to Internal Standards Ion Current Area Counts for Blanks

Ion Current	Normalized VOST Traps					Normalized VOST Condensate		Comments
	Pair 1	Pair 2	Pair 3	Pair 4	Field Blank Pair ^b	Sample	Method Blank ^b	
Total Injection	TCN 278	274	298	289	64.6	40.3	53.4	TCN=TC*(ISC ₆ /ISC _s)
Internal Standards	ISCN 25.8	25.8	25.8	25.8	25.8	18.0	18.0	ISCN=ISC*(ISC ₆ /ISC _s)
Surrogate Compounds	SCCN 36.8	27.4	29.5	27.3	23.5	9.7	10.5	SCCN=SCC*(ISC ₆ /ISC _s)
Sample	SCN 216	221	243	236	15.3	12.6	24.9	SCN=TCN-ISCN-SCCN
Target Analyte	TACN 175	191	212	206	5.6	10.5	12.8	TACN=TAC*(ISC ₆ /ISC _s)
TICs	TICCN 18.3	14.6	15.3	14.9	0.2	NA	NA	TICCN=TICC*(ISC ₆ /ISC _s)

^a Ion currents shown are area counts, in millions. Values shown for VOST condensate are for 5 ml aliquot

^b Normalizing on blank, therefore, blank is not changed from what is shown in Table 1.

Note: Subscript s means sample and subscript b means blank.

Table 3. Volatile Chromatographic Organic Uncharacterized Fraction Blank Corrected Data^a

Ion Current ^a	VOST Traps					VOST Condensate Sample	Comments Reference Table 2
	Pair 1	Pair 2	Pair 3	Pair 4	Trap Subtotal		
Sample	200	206	227	221	854	0	SCNbc=SCN _s -SCN _b
Target Analytes	170	185	206	200	761	0	TACNbc=TACN _s -TACN _b
TICs	18.1	14.4	15.1	14.7	62.3	0	TICCNbc=TICCN _s -TICCN _b

^a Ion currents shown are area counts, in millions.

Notes:

Subscript s means sample and subscript b means blank.

When blank correction results in a negative number than it is treated as 0.

Table 5. Semivolatile Chromatographic Organic Uncharacterized Fraction Input Data^a

Ion Current ^a	Filter/XAD		FH/BH Rinse		Condensate		Comments
	Sample	Field Blank	Sample	Field Blank	Sample	Field Blank	
Total	697	754	397	579	411	289	
Internal Standards	129	142	95	106	92.4	97.1	
Surrogate Compounds	269	344	169	209	153	159	
Target Analyte	33.5	15.4	4.2	5.4	39.1	0.8	
TICs	76.9	74.2	60.1	125	64.8	8.8	
Response Value ^b (ng/10 ⁶ counts)	----	845	----	1130	----	1240	

^a Ion currents shown are area counts, in millions, for 2µl injection.

^b Response values are used to calculate mass from area counts as shown in Table 8. Development of the response factors is presented in Appendix A.

Table 6. Semivolatile Chromatographic Organic Uncharacterized Fraction^a Data Normalized to Internal Standards Ion Current Area Counts for Blanks

Ion Current	Filter/XAD		FH/BH Rinse		Condensate		Comments Reference Table 5
	Sample	Field Blank ^b	Sample	Field Blank	Sample	Field Blank	
Total	767	754	443	579	432	289	TCN=TC*(ISC _b /ISC _s)
Internal Standards	142	142	106	106	97.1	97.1	ISCN=ISC*(ISC _b /ISC _s)
Surrogate Compounds	296	344	189	209	161	159	SCCN=SCC*(ISC _b /ISC _s)
Sample	329	268	148	264	174	33	SCN=TCN-ISCN-SCCN
Target Analyte	36.9	15.4	4.7	5.4	41.1	0.8	TACN=TAC*(ISC _b /ISC _s)
TICs	84.6	74.2	67.1	125	68.1	8.8	TICCN=TICC*(ISC _b /ISC _s)

^a Ion currents shown are area counts, in millions, for 2µl injection versus total sample volume of 1 ml.

^b Normalizing on blank, therefore, blank is not changed from that shown in table 5.

Note: Subscript s means sample and subscript b means blank.

Table 7. Semivolatfile Chromatographic Organic Uncharacterized Fraction Blank Corrected Data^a

Ion Current ^a	Filter/XAD	FH/BH Rinse	Condensate	Comments Reference Table 6
Sample	61	0	141	SCNbc=SCN _s -SCN _b
Target Analytes	21.5	0	40.3	TACNbc=TACN _s -TACN _b
TICs	10.4	0	59.3	TICCNbc=TICCN _s -TICCN _b

^a Ion currents shown are area counts, in millions, for 2µl injection versus total sample volume of 1 ml.

Notes:

Subscript s means sample and subscript b means blank.

When blank correction results in a negative number then it is treated as 0.

**Table 8. Semivolatile Chromatographic Organic Uncharacterized Fraction
Conversion of Blank Corrected Area Counts to Mass Units**

	Filter/XAD			FH/BH Rinse			Condensate			Total Mass (ng)
	Ion Current ^a	Response ^b (ng/10 ⁶ counts)	Trap Subtotal Mass (ng)	Ion Current ^a	Response ^b (ng/10 ⁶ counts)	FH/BH Subtotal Mass (ng)	Ion Current ^a	Response ^b (ng/10 ⁶ counts)	Condensate Subtotal Mass (ng)	
Sample	61	845	51,500	0	1,130	0	141	1,240	175,000	227,000
Target Analytes	21.5	845	18,200	0	1,130	0	40.3	1,240	50,000	68,200
TICs	10.4	845	8,790	0	1,130	0	59.3	1,240	73,500	82,300
Uncharacterized Mass (ng)										76,500
Uncharacterized Mass Fraction										34%

^a Blank corrected ion currents are from Table 7, and are area counts, in millions, for 2µl injection.

^b Response values from Table 5 (Also see Appendix A).

Table 9. Total Uncharacterized Fraction of Chromatographic Organics

	VOST Samples Blank Corrected		SV Samples Blank Corrected		Total Blank Corrected	
	Sample	Uncharacterized Fraction	Sample	Uncharacterized Fraction	Sample	Uncharacterized Fraction
Blank Corrected Mass (ng)	24,900 ^a	890 ^a	227,000 ^b	76,500 ^b	381,800	32,600
Sample Gas Volume (dscm) ^c	0.07815	0.07815	3.613	3.613	381,800	32,600
Concentration (ng/dscm)	319,000	11,400	62,800	21,200	381,800	32,600
Overall Uncharacterized Fraction						9%

^a VOST sample mass results are from Table 4.

^b SV sample mass results are from Table 8.

^c Sample volumes are from Final Report on Risk Burn No. 1:

Table 3.3-3 VOST Sample Volume = 78.15L.

Table 3.4-4 SV Sample Volume = 3.613 dscm.

Appendix

Calculations of Response Values

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Appendix

Calculations of Response Values

Response values were determined for the blanks associated with VOST traps and VOST condensate, and for the Filt/XAD, FH/BH Rinse, and condensate for the MM5-SV samples. These response values were calculated in order to convert ion current area counts to mass units (i.e., ng). The blanks were used for determination of response values because the samples were normalized on the blanks.

For VOST traps, the response value was calculated by dividing the total mass of the three internal standards spiked onto the field blank pair (750 ng) by the internal standards area counts, as shown in Table A-1.

For the VOST condensate, the total mass of internal standards was the same (750 ng), but it was spiked into a 5 mL amount of the method blank and 5 mL of sample. The response value was therefore determined by dividing the mass (750 ng) by the area counts for the internal standards spiked into 5 mL, and then multiplying by the split factor of 12.14. This split factor was the actual total sample volume of 60.7 mL divided by the 5 mL volume analyzed.

The MM5-SV samples consisted of individual extracts of the filter/XAD, FH/BH rinses and condensate. The final volume for each extract was 1 mL (1,000 μ L) of which 2 μ L was analyzed. Each 2 μ L portion contained 40 ng of each of six internal standards, for a total of 240 ng. The response values were calculated for each fraction of the blank train, as shown in Table A-2. Since 2 μ L of the total 1 mL extract was analyzed, the response value was determined using the split factor of $1000 \mu\text{L}/2 \mu\text{L}$ or 500.

The response values, which include the split factor, allow calculation of the mass of compounds (i.e., ng) in the entire sample, based on the area counts for that portion of the sample actually analyzed (i.e., 5 mL of VOST condensate or 2 μ L of MM5-SV extract). However, this assumes that the response for the target analytes, TICs or unknowns is the same as the response for the internal standards. This could also be expressed as assuming a relative response average factor (RRF) equal to 1.0 for all targets, TICs and unknowns.

Table A-1. Response Values for VOST Traps and VOST Condensate

VOST Traps

IS counts for field blank pair - 25.8×10^6
Amount of IS spiked onto each trap pair - 750 ng

$$\text{Response Value} = \frac{750 \text{ ng}}{25.8 \times 10^6 \text{ counts}} =$$

$$29.1 \frac{\text{ng}}{10^6 \text{ counts}}$$

VOST Condensate

IS counts for 5 mL of method blank = 18.0×10^6
Amount of IS spiked into 5 mL = 750 ng

$$\text{Split factor} = \frac{60.7 \text{ mL sample}}{5.0 \text{ mL analyzed}} = 12.14$$

$$\text{Response Value} = \frac{750 \text{ ng}}{18.0 \times 10^6 \text{ counts}} \times 12.14$$

$$506 \frac{\text{ng}}{10^6 \text{ counts}}$$

Table A-2. Response Values for MM5-SV Blank Train Samples

Filt/XAD

IS counts for 2 μL of extract = 142×10^6
 Amount of IS in 2 μL = 240 ng

$$\text{Split factor} = \frac{1000 \mu\text{L sample}}{2 \mu\text{L analyzed}} = 500$$

$$\text{Response} = \frac{240 \text{ ng}}{142 \times 10^6 \text{ counts}} \times 500$$

$$845 \frac{\text{ng}}{10^6 \text{ counts}}$$

FH/BH

IS counts for 2 μL of extract = 106×10^6
 Amount of IS in 2 μL = 240 ng

$$\text{Split factor} = \frac{1000 \mu\text{L sample}}{2 \mu\text{L analyzed}} = 500$$

$$\text{Response} = \frac{240 \text{ ng}}{106 \times 10^6 \text{ counts}} \times 500$$

$$1,130 \frac{\text{ng}}{10^6 \text{ counts}}$$

Condensate

IS counts for 2 μL of extract = 97.1×10^6
 Amount of IS in 2 μL = 240 ng

$$\text{Split factor} = \frac{1000 \mu\text{L sample}}{2 \mu\text{L analyzed}} = 500$$

$$\text{Response} = \frac{240 \text{ ng}}{97.1 \times 10^6 \text{ counts}} \times 500$$

$$1,240 \frac{\text{ng}}{10^6 \text{ counts}}$$

Test Report for Trial Burns No. 1 and No. 2 on the Drake Chemical Superfund Site's Mobile On-Site Hazardous Waste Incinerator

Volume 1—Technical Report

For OHM Remediation Services Corp.

MRI Project No. 9620-13/23

September 12, 1997