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ENGINEERING AND DEVELOPMENT SUPPORT OF GENERAL DECON
TECHNOLOGY FOR THE DARCOM INSTALLATION RESTORATION PROGRAM

Task 8. Identification of Recovered Organics from
Supercritical Fluid Process to Regenerate
Carbon

FINAL REPORT

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Submitted to:

Commander
U.S. Army Toxic and Hazardous Materials Agency
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A. Objective

The purpose of this task was to qualitatively identify the contaminants of Rocky Mountain Arsenal groundwater by GC/MS and compared the results to a GC/MS scan of the supercritical fluid extract of carbon provided by A.D. Little. In addition to the qualitative analysis, a semi-quantitative analysis of the DIMP levels in both groundwater and carbon extract was performed.

B. Qualitative Analysis of RMA Groundwater

A 200 ml sample of the RMA groundwater was extracted with two 5 ml of ethyl ether. The extract was further concentrated to approximately 0.1 ml in a micro "Kuderna Danish" evaporator for GC/MS analysis. The chromatographic conditions were as follows:

Column: 2% Dexsil 300 on Anachrom Q
Oven Temperature: 140 - 200°C @ 15°C/minute
Injector Temperature: 200°C

Mass scanning was from 40 - 550 AMU at 8 samples per 0.1 AMU. An electron multiplier potential of 2,000 volts was employed.

The chromatogram of the groundwater extract gave two main peaks. The first gave a mass spectrum with major ions at 97 and 123 AMU. Comparison of this spectrum to that found for an authentic sample of DIMP established the identity of the peak. The second peak was tentatively identified as 2,6-di(τ -butyl)4-methylphenol by a manual search of the EPA/NIH Mass Spectral Data Base.

C. Analysis of A.D. Little U-Tube

As received from A.D. Little, the U-tube containing the supercritical extract from the carbon was wrapped in aluminum foil. Removal of the foil exposed a U-tube capped with teflon stopper. The tube had broken where the stopper was inserted but there was no evidence of loss of any of the tube's contents. The

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Availability Codes	
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tube contained about 4 - 5 ml of what appeared to be water. The tube contents were extracted by addition of two separate 5 ml portions of ether. The tube was recapped each time and shaken well before pouring out the contents. The combined extracts were separated from the water phase and injected directly without concentration. The U-tube extract showed the presence of only DIMP.

D. Semi-Quantitative Determination of DIMP Content in the RMA
Ground Water and U-Tube

A second 100 ml sample of RMA groundwater was extracted twice with chloroform, the total extract amounting to 2 ml. Analysis was performed on a Hewlett Packard 5880 gas chromatograph equipped with a nitrogen phosphorous detector with the following conditions:

Column:	1.5% OV-17/1.95% OV-210
Temperature:	Oven 130°C
	Injector 300°C
	Detector 320°C
Carrier:	N ₂ @ 28 cc/minute
Auxillary Gas:	H ₂ @ 4 cc/minute
	Air @ 90 cc/minute
Sensitivity:	532 AU/ng (based on a 20.3 ng injection)
Retention Time:	varies with sample size (0.8 - 1.5 minutes)

The RMA groundwater contained 1.55 mg/L of DIMP. The U-tube extract contained 31.47 mg/L of DIMP or a total 0.315 mg of DIMP. The efficiency of the supercritical carbon extraction can be determined if the amount of groundwater run through the carbon is known.

SAMPLE NAME WELL WATER EXTRACT (GC/MS)

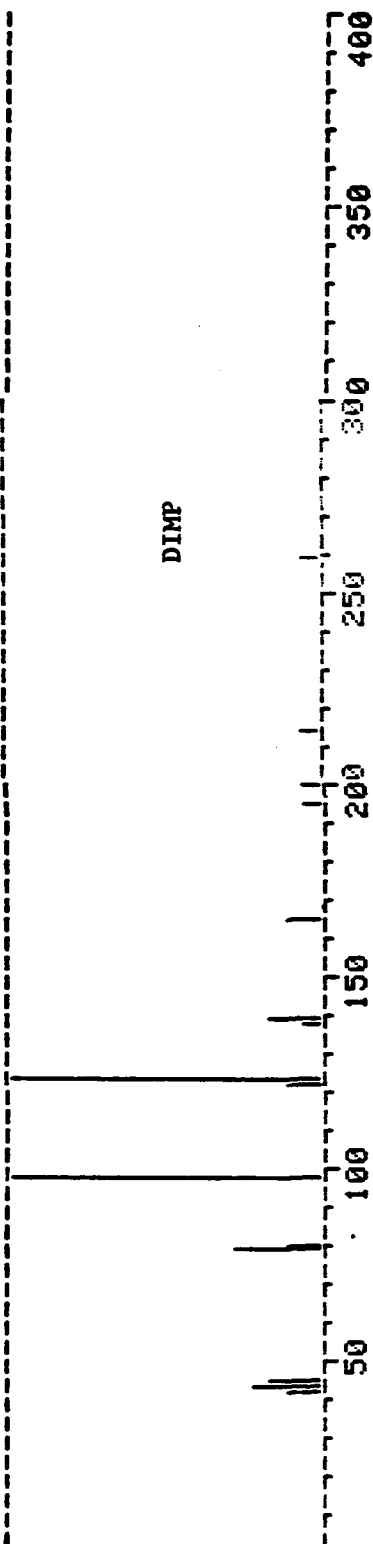
OPERATOR

TOTAL ABUNDANCE FROM 40 TO 450 amu
Full Scale= 9920
ION 269.0
Full Scale= 150

DMP
2,6-di(1-butyl)4-methylphenol

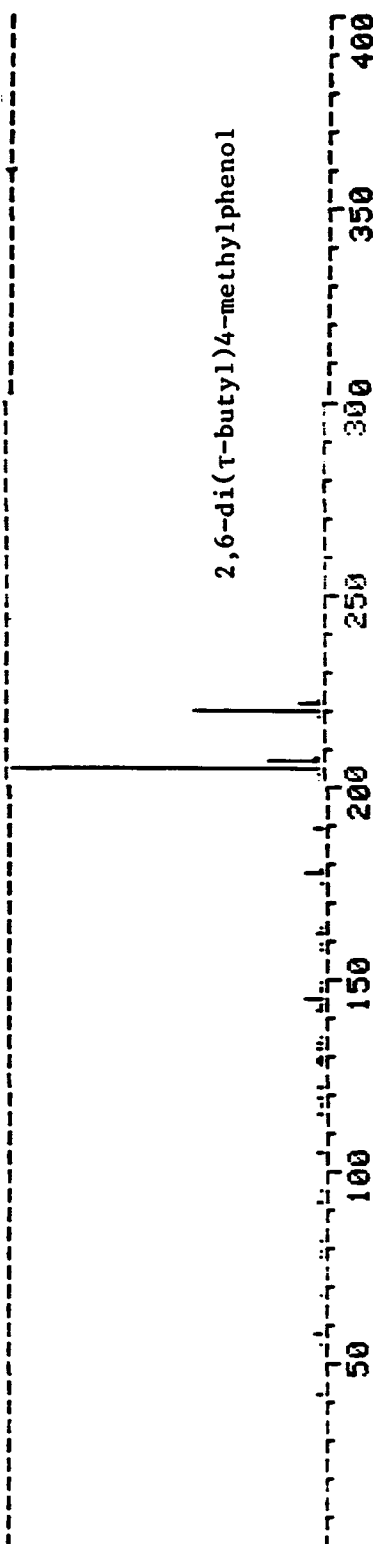
GROUNDWATER EXTRACT - MASS SPECTRA

** Spectrum # 84 ** Sample # 1 Retention Time = 0.4 minutes
 Scanned from 40 to 450 amu Number of Peaks Detected = 15
 File type = linear
 Base Peak = 96.95



DIMP

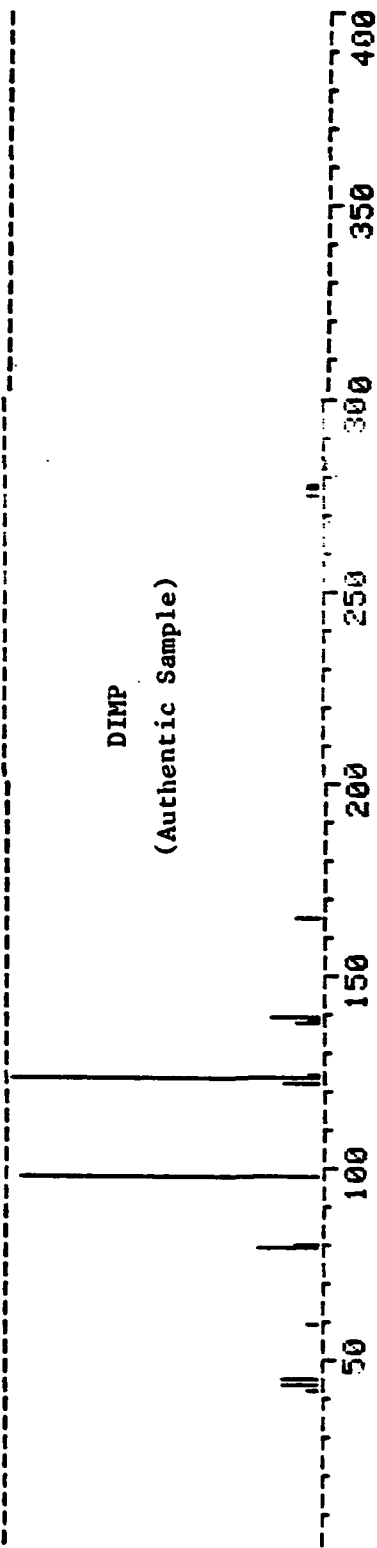
** Spectrum # 85 ** Sample # 1 Retention Time = 3.1 minutes
 Scanned from 40 to 450 amu Number of Peaks Detected = 83
 File type = linear
 Base Peak = 205.20



2,6-di(t-butyl)4-methylphenol

DIMP - Mass Spectra

** Spectrum # 28 ** Sample # 1 Retention Time = 0.7 minutes
Scanned from 40 to 550 amu Number of Peaks Detected = 17
File type = linear
Base Peak = 123.00



Chromatograph of Groundwater Extract

RT: DET 2 TEMP=300°C SETPT=300°C LIMIT=0.31

0.31

EXP 5886A MANUAL INJECTION @ 14:30 AUG 7, 1981
AREA %

RT	AREA	TYPE	WIDTH	HEIGHT	BASELINE	AREA %
0.00						
0.00						
0.00						
0.31	510.85	BP	0.024	334.27	33.48	0.646
0.31	79542.80	PS	*0.20 *	4453.97	28.75	99.354

TOTAL AREA = 79053.70
MULTIPLIER = 1

Chromatograph of U-Tube Extract

RT: DET 2 TEMP=300°C SETPT=300°C LIMIT=405°C
 0.48 1.18

MANUAL INJECTION @ 14:17 AUG 7, 1981

RT	AREA	TYPE	WIDTH	HEIGHT	BASELINE	AREA %
0.00						
0.00						
0.00						
0.30	135.15	VP	0.012	175.84	5.47	0.381
0.48	2876.82	PH	*-----*	376.91	0.94	8.115
1.18	32439.80	HH	-----*	646.03	0.94	91.504

TOTAL AREA = 35451.80
 MULTIPLIER = 1

Chromatograph of DIMP 77 ppm (SARM)

DIMP ANALYSIS OV17-UV21 COLUMN AT 130.1

RT: DET 2 TEMP=300°C SEIPT=300°C LIMIT=405°C

0.61

80947.10 ANNUAL INJECTION @ 13:41 AUG 7, 1981

0.00

RT	AREA	TYPE	WIDTH	HEIGHT	BASELINE	AREA %
0.00						
0.00						
0.00						
0.61	80947.10	PH	*0.36 *	3519.01	0.61	100.000

BASELINE @ START RUN = 35.65

THRESHOLD @ START RUN = 1

PEAK WIDTH @ START RUN = 0.08

TOTAL AREA = 80947.10

MULTIPLIER = 1