



Dispersion by Chemical Reaction of Rocky Mountain Arsenal Basin F Waste Soils

James R. Payne and Giles M. Marion

February 1997

Abstract: Many military installations have soil contamination problems that range from heavy metals to petroleum products. Rocky Mountain Arsenal (RMA) Basin F contains high concentrations of salts, heavy metals, ammonia, urea, and organics. The Dispersion by Chemical Reaction (DCR) process leads to a reduction in the mobility of the organic and inorganic constituents by first removing volatile constituents via steam stripping and volatilization, then trapping the nonvolatile contaminants in a nonmobile phase (microencapsulation), and finally compacting the treated material into large soil bodies (macroencapsulation). This report summarizes the results of the DCR testing of soil-amended Basin F sludge from RMA. The primary focus of this study is on pesticide leachability. The DCR process used to treat the Basin F waste soil produced a dry, homogeneous, soil-like material with desirable physical properties that on compaction achieved the

following remediation goals: reduction of all leachable volatiles to nondetectable levels, confinement of all metals to below RCRA TCLP levels, and a decrease in pesticide leachability to levels approaching RCRA standards. For example, endrin TCLP concentration was reduced from 74 µg/L to 20–28 µg/L (regulatory limit = 20 µg/L). In several cases, reductions in pesticide leachability could be attributed to simple dilution with the calcium oxide (CaO) reagent. However in other cases, microencapsulation and/or macroencapsulation also played a role in reducing pesticide leachability. Additional work is necessary to optimize the amounts of lime-milk, hydrophobic CaO, and benign oil used in the processing of RMA Basin F waste soils. Ideally, the optimum design should achieve the regulatory and client goals, while minimizing materials handling, energy, and reagent inputs.

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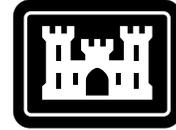
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James R. Payne and Giles M. Marion

February 1997

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OFFICE OF THE CHIEF OF ENGINEERS

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PREFACE

This report was prepared by James R. Payne, Research Chemist, SOUND/**epic** Environmental, Encinitas, California, and Dr. Giles M. Marion, Research Physical Scientist, the Geochemical Science Division, Research and Engineering Directorate, U.S. Army Cold Regions Research and Engineering Laboratory (CRREL), Hanover, New Hampshire.

This work was funded by the Office of the Chief of Engineers through CPAR Project AC910260, *Site Remediation via Dispersion by Chemical Reaction (DCR)*, to evaluate DCR technologies for handling different types of contaminants at DOD facilities. Technical review was provided by Dr. Thomas F. Jenkins and Daniel Leggett of CRREL.

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Dispersion by Chemical Reaction Testing of Rocky Mountain Arsenal Basin F Waste Soils

JAMES R. PAYNE AND GILES M. MARION

INTRODUCTION

Many military installations have soil contamination problems that range from heavy metals to petroleum products. The Rocky Mountain Arsenal (RMA) near Denver, Colorado, was established in 1942 and was the site for the manufacture of chemical incendiary devices and demilitarization of chemical munitions (Woodward-Clyde 1989). An evaporation basin (Basin F) for discharge of waste effluents was constructed in 1956 and used until 1981. In 1988, remediation of Basin F began. Basin F contains high concentrations of salts (high ionic strength), heavy metals, ammonia, urea, and organics, many of which are unidentified (Woodward-Clyde 1989).

Initially developed in Germany by Professor Friedrich Boelsing of the University of Hannover, the Dispersion by Chemical Reaction (DCR) process has been widely utilized to treat successfully more than one million cubic meters of both organic and inorganic wastes in Europe for over 18 years (Boelsing 1988, 1994; Payne et al. 1992). Most recently it has been used in the United States to stabilize 26,100 tons of sludges from petroleum barge-cleaning operations at the NICOR National Louisiana Inc. Impoundment Closure in Belle Chasse, Louisiana, and 233,000 tons of acid tar residuals remaining from lubricating-oil refining activities at the Sand Springs Superfund Site in Sand Springs, Oklahoma. Recently the DCR process was used to remediate asphalt tar and other organic-contaminated soils from Eareckson Air Force Station at Shemya, Alaska (Brar and Marion 1995; Marion et al., in press). Like several other stabilization technologies, the patented DCR process utilizes calcium oxide (CaO) as a reagent; however, by treating normally hydrophilic CaO

with a natural fatty acid or other material, a hydrophobic reagent can be prepared that preferentially adsorbs any organic (or oil/pesticide phase) in a waste liquid or soil/sludge matrix upon initial mixing. Hydrophilic CaO can also be utilized if special rapid-mixing procedures are used to predistribute the waste onto the CaO before hydration. After this predistribution of the organic phase onto the CaO, it undergoes a delayed exothermic hydration to produce calcium hydroxide [Ca(OH)₂] (eq 1), which is fractured into sponge-like submicron-sized particles that are homogeneously charged throughout their internal and external cavities with the organic phase:



Any oils or organics that were previously adsorbed on the CaO particles are now dispersed and bound within the newly formed and highly adsorptive cavities of the hydrophobized Ca(OH)₂ crystals. The 30- to 40-fold increase in specific surface area ensures that no free liquid phase of the microencapsulated waste remains, and the resultant product is a free-flowing solid, which can be compacted into a hydrophobized body of soil with outstanding soil-mechanical properties.

When DCR-treated materials are compacted as subgrade for construction (or in a landfill), the outside surface of the Ca(OH)₂ reacts with CO₂ to generate relatively insoluble CaCO₃ (limestone) (eq 2) while still bound in the original soil matrix.



By this process, the soil body forms its own self-healing CaCO₃ crust, and the organics are removed from the biological environment by a combina-

tion of micro- and macroencapsulation. Because of the low permeabilities and hydrophobic, water-repelling nature of the compacted materials, water cannot easily penetrate the soil body (Pamukcu et al. 1989–90). In Europe, such DCR-treated soil bodies have been compacted (with permeabilities in the range of 10^{-6} to 10^{-8} cm s⁻¹) into unlined landfills, roads, berms around tank farms, and subgrade for industrial sites. Data from adjacent monitoring wells have verified the absence of any significant off-site migration of organic components for over 16 years (Boelsing 1994).

This report summarizes the results of DCR testing of soil-amended Basin F sludge from the Rocky Mountain Arsenal. The report is divided into several sections dealing with waste characterization, selection of a “worst case” sample for further testing, initial DCR treatability testing (scoping experiments) on the worst-case sample, and bench-scale (13 kg) batch preparation of DCR-treated material for independent, third-party laboratory analyses. The results of the independent chemical testing are then presented, followed by overall conclusions and recommendations for further study.

METHODS AND MATERIALS

Initial RMA waste soil characterization

Four separate 15-gal drums containing representative soil samples were transported under full chain of custody to SOUND/epic Environmental in Encinitas, California, on 9 March 1995. Upon initial receipt, each sample was further homogenized and examined to assess handling and physical characteristics (Table 1). An aliquot of each was removed for hexane extraction and gravimetric determinations of oil and grease and total petroleum hydrocarbons (TPH). The extracts were then further characterized by flame ionization detector (FID) gas chromatography (GC) to determine total resolved hydrocarbons and the approximate molecular weight range (distillate cut fraction) of any petroleum-type constituents in the sample. Electron capture detector (ECD) gas chromatography was then used to screen for pesticides.

Figure 1 presents the chromatograms obtained from the FID GC analyses. Of the four samples, the “soil-amended Basin F sludge from above the liner” is clearly the most contaminated, with five

Table 1. Initial characterization of four selected waste/soil samples from the Rocky Mountain Arsenal Basin F area.

Waste sample	Below the liner, sample no. 41238	Soil-amended Basin F sludge from above the liner, sample no. 24216	Below the liner, sample no. 24093	Below the liner, sample no. 41249
Waste description	Lt. brown, dry, and slightly plastic and sticky soil. No crystals present.	Med. brown soil/silt. Very dry and friable; not plastic. Yellow-green crystals present.	Wet, saturated gray clay/silt sludge. Surface and interstitial water, light tan/brown.	Lt. tan/brown clayey soil. Compressible. No crystals present.
Odor	Mild, sweet	Rotten (like old gym socks or shoes) and trace of ammonia.	Light petroleum hydrocarbon-like smell	Moderate “pesticide” or organic smell.
Peak OVA in drum headspace (ppm)	430	N.A.	100	400
Stable OVA in drum headspace (ppm)	35	N.A.	2	15
Moisture content (%)	15.8	11.7	27.2	16.3
Gravimetric oil and grease (mg/kg dry wt.)	365	10,700	4,110	634
Total resolved hydrocarbons (FID GC) (mg/kg dry wt.)	726	2,420	82	943
Total resolved pesticides (ECD GC) (mg/kg dry wt.)	1,010	3,270	110	1,280

N.A. = Not analyzed

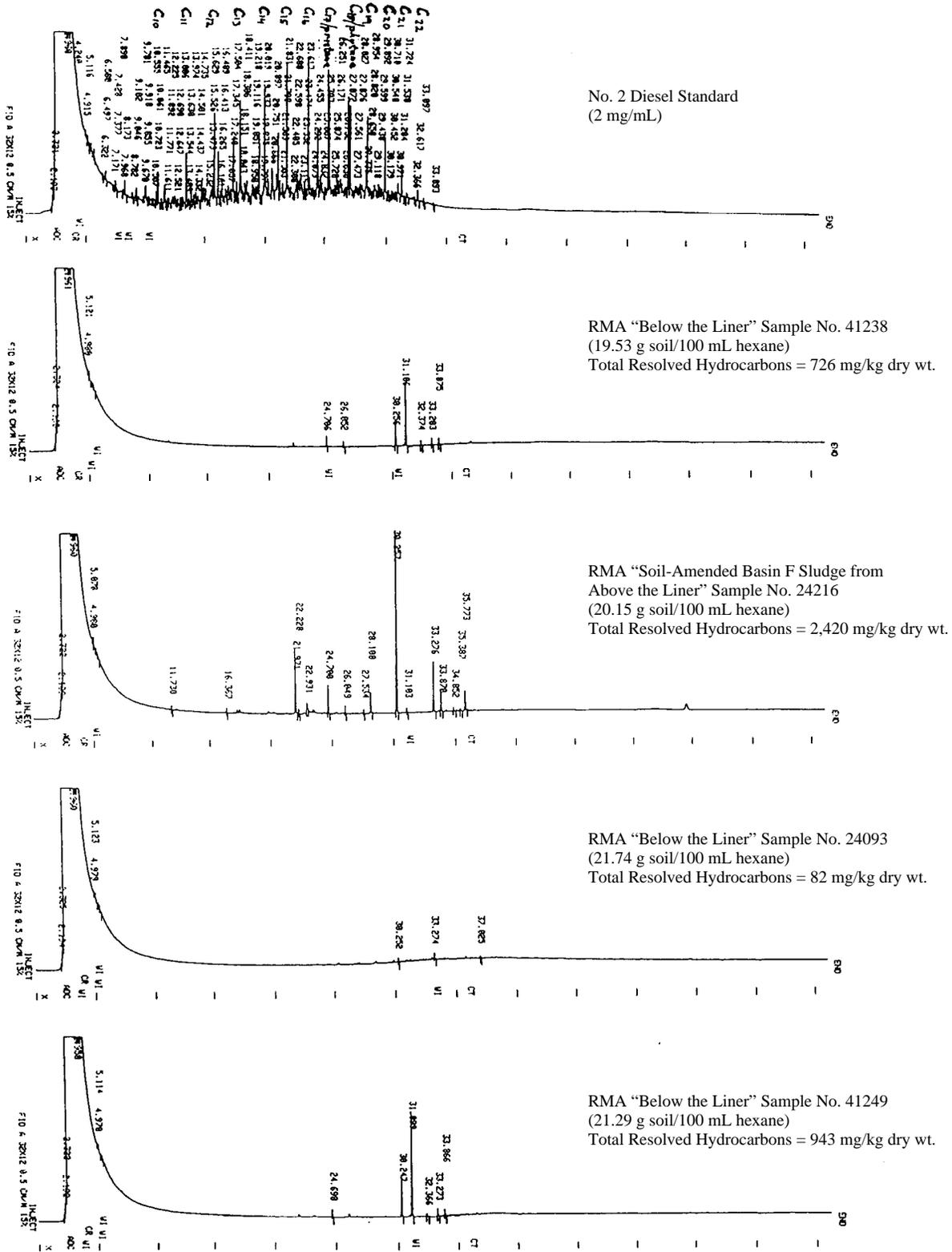
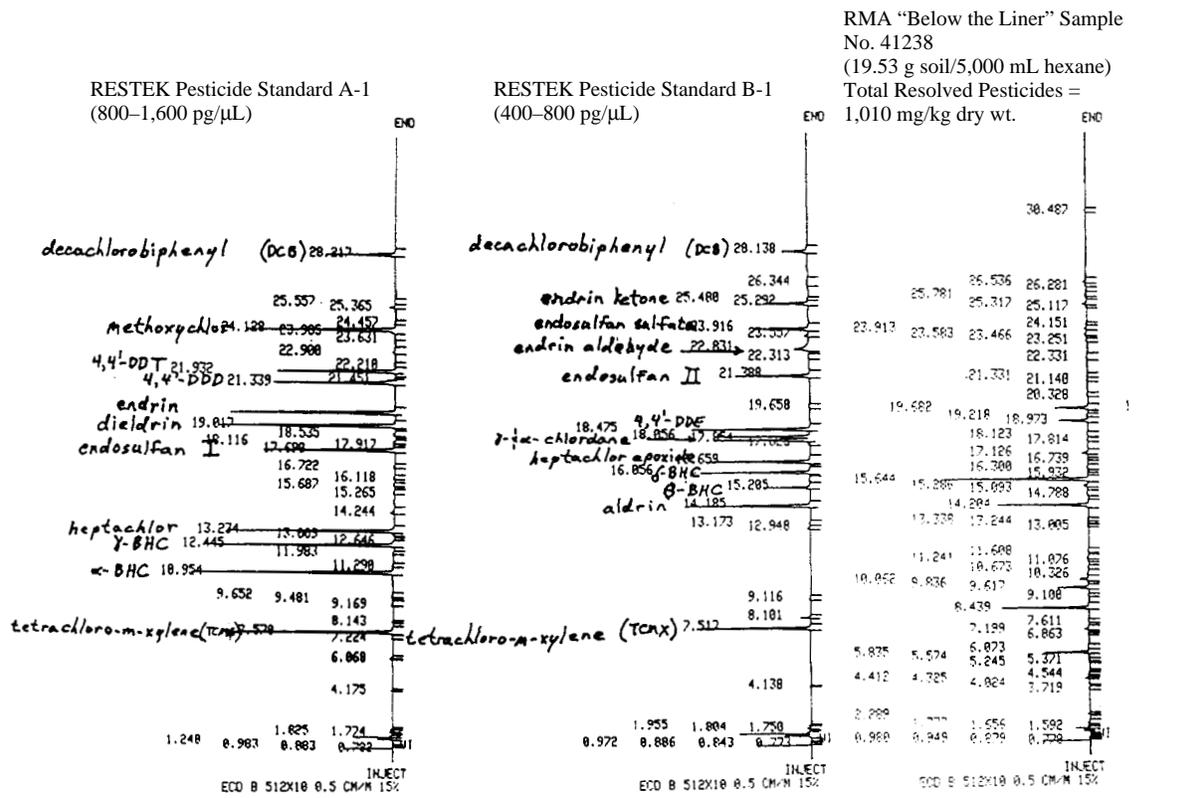


Figure 1. Flame ionization detector (FID) capillary gas chromatographic (GC) profiles from a No. 2 Diesel Standard containing n-alkanes from n-C₁₀ to n-C₂₂ and hexane extracts of RMA soil and sludge samples specially prepared to allow direct comparisons of total resolved hydrocarbons among the samples (i.e., a constant nominal weight [20 g] of each sample was extracted with a constant volume [100 mL] of solvent). All chromatograms were obtained under identical instrument conditions (1.0 μL injected; 30 m, OV-1 narrow bore column; 50°C [4 min] then 7°C/min to 275°C [30 min]; GC attenuation 32 × 12).



RMA "Soil-Amended Basin F Sludge from Above the Liner" Sample No. 24216 (20.15 g soil/5,000 mL hexane) Total Resolved Pesticides = 3,270 mg/kg dry wt.

RMA "Below the Liner" Sample No. 24093 (21.74 g soil/5,000 mL hexane) Total Resolved Pesticides = 110 mg/kg dry wt.

RMA "Below the Liner" Sample No. 41249 (21.29 g soil/5,000 mL hexane) Total Resolved Pesticides = 1,280 mg/kg dry wt.

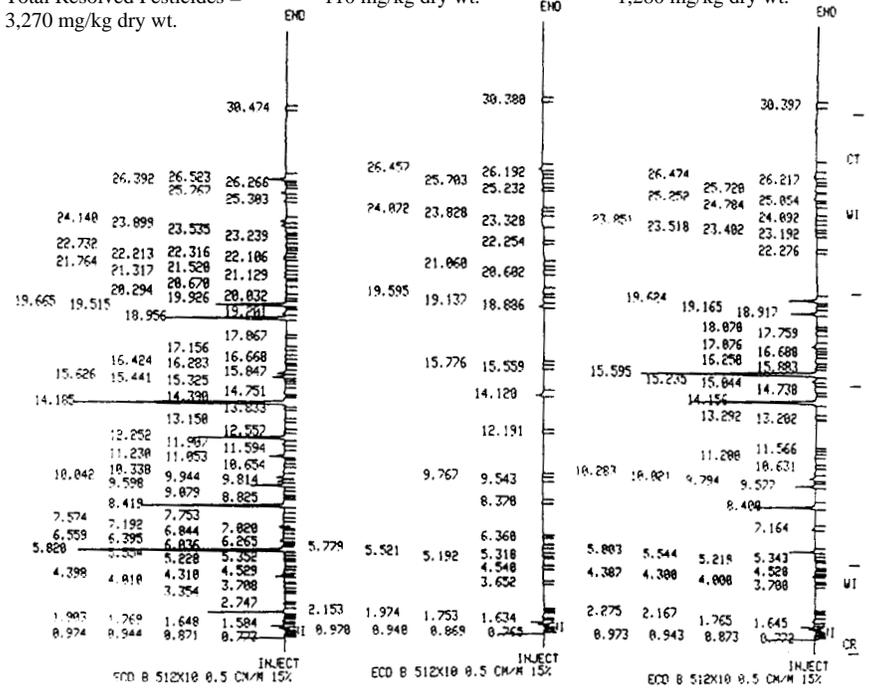


Figure 2. Electron capture detector (ECD) capillary gas chromatographic (GC) profiles from two RESTEK Pesticide Standard solutions and hexane extracts of RMA soil and sludge samples specially prepared to allow direct qualitative and quantitative comparisons of pesticide burdens among the samples (i.e., a constant nominal weight [20 g] of each sample was extracted/diluted with a constant volume [5000 mL] of solvent. All chromatograms were obtained under identical instrument conditions (1.0 μL injected; 30 m, DB-1701 megabore column; 140°C [0.5 min] then 5°C/min to 275°C [7.5 min]; GC attenuation 512 × 10).

major peaks in the Kovats Index 1500 to 2300 range (between n-C₁₅ and n-C₂₃ in the diesel standard; von Kovats 1958). The exact identification of these constituents is not known, but they could be aldrin, dieldrin, and endrin based on the exceedingly high concentrations of these materials in the sample (see the ECD GC discussion below). The FID GC profiles for two of the “below the liner” samples (41238 and 41249) are very similar to each other, but different (both quantitatively and qualitatively) from the soil-amended sludge (sample 24216). The third below-the-liner sample (24093) was a wet, saturated gray clay/silt sludge, and although it had a light petroleum hydrocarbon smell, the FID GC showed only two very small resolved peaks with retention times similar to the larger constituents in sample 24216. Clearly there is no evidence of significant petroleum hydrocarbon-type contamination (i.e., diesel or other distillate product) in any of the samples.

Figure 2 presents the chromatograms obtained on two RESTEK Co. pesticide standards and the hexane extracts from the four RMA soil samples. Each of the four RMA sample chromatograms was obtained under identical instrument conditions (attenuation 512 × 10) on the same “nominal concentration” of sample extract. That is, a constant nominal weight (20 g) of each sample was extracted/diluted with a constant volume (5,000 mL) of solvent so that the relative concentrations of individual pesticides would be readily discernible from the peak size. Here again, the soil-amended Basin F sludge from above the liner is clearly the most contaminated, with 3,270 mg/kg dry weight total pesticides resolved. Comparison of retention times with those from individual constituents in the two RESTEK pesticide standards allows aldrin, dieldrin, and endrin to be identified in three out of the four RMA samples. As with the FID GC results discussed above, two of the below-the-liner samples (41238 and 24093) are qualitatively very similar with one unique component [retention time (RT) 15.6] that is not present in the above-the-liner soil-amended sludge (sample 24216). The other peaks in these two samples are common with sample 24216 (although at much lower levels). At these “nominal” extract concentrations, there are only traces of aldrin and essentially no other resolved compounds in the wet, saturated gray clay/silt sludge from below the liner (sample 24093).

To allow better comparison of the common constituents in all of the RMA samples, the extracts were concentrated so that the peaks from each of

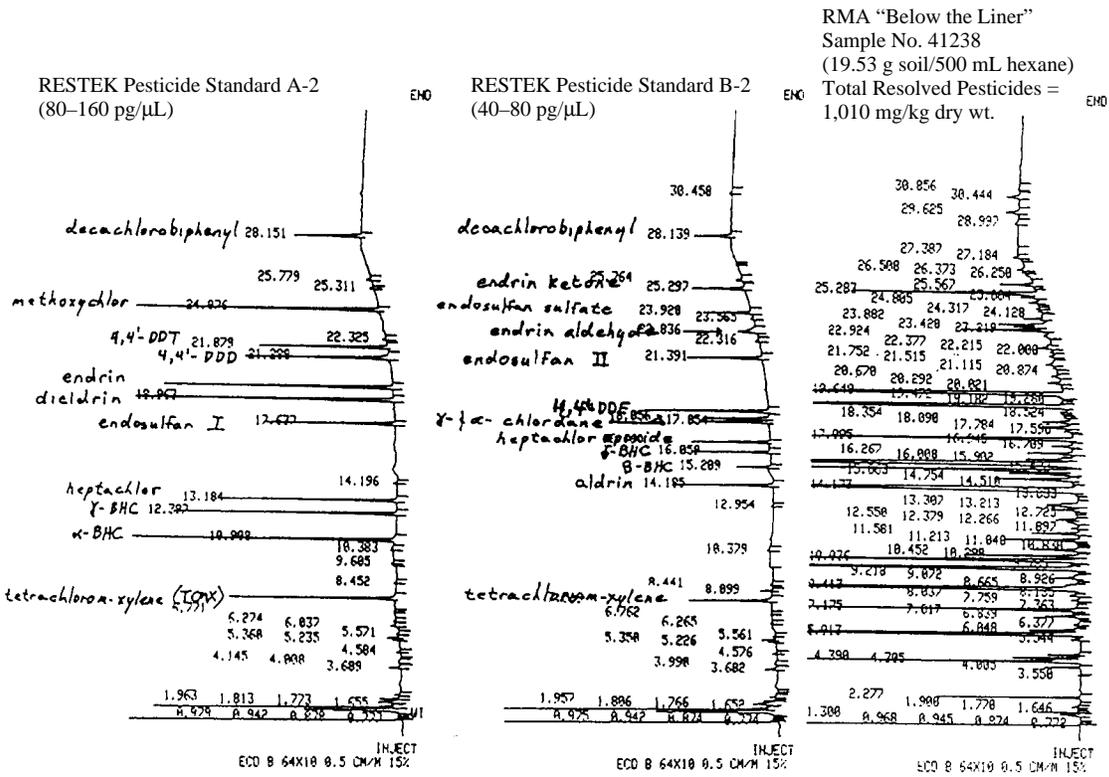
the major constituents would be truncated at 100% full scale [at an increased instrument sensitivity (attenuation 64 × 10)], thereby facilitating detection of the smaller peaks. The results of these analyses are shown in Figure 3 along with the two RESTEK Co. pesticide standards for reference. In this case, aldrin, dieldrin, and endrin can now also be observed in the wet, saturated gray clay/silt sludge from below the liner (sample 24093), along with numerous other constituents common to all four samples. Unfortunately, given the lack of adequate standards, it is impossible to identify most of these unknown compounds. They are probably chlorinated organics; given their sensitivity to the electron capture detector, however, they do not correspond to any of the components in the standards available at the time of these analyses. Compound identification by GC/MS might be possible, but it too would be difficult due to the low (pg) quantities involved.

These analyses show the general qualitative similarity among the samples, and based on these chromatographic observations and the data in Table 1, it was not difficult to select the soil-amended Basin F sludge as the worst-case material for DCR treatability testing. In addition, it was known that the majority of the contamination originated in the Basin F liquid (Table 2), and for this reason as well, sample 24216 was selected. In addition to significant levels of pesticides, the Basin F liquid (later mixed with clean soil to yield the soil-amended Basin F sludge) contained very high concentrations of inorganic salts [e.g., NaCl, NH₄Cl, and (NH₄)₂SO₄] as well as 14% urea (Table 2).

Table 2. Descriptive chemical characterization of RMA Basin F liquid* (highest historical data).

<i>Constituent</i>	<i>mg/L</i>
Water	620,000
Major metals	
Sodium (Na)	65,000
Copper (Cu)	5,860
Potassium (K)	2,900
Major inorganics	
Chloride (Cl ⁻)	159,000
Ammonia (NH ₃)	60,900
Sulfate (SO ₄ ⁼)	27,000
Total phosphorus	16,200
Nitrate (NO ₃ ⁻)	1,300
Additional components	
Urea	143,000
Dimethylmethylphosphonate	2,000

* From R.F. Weston document 1212WG.APA (12/17/90)



RMA "Soil-Amended Basin F Sludge from Above the Liner" Sample No. 24216 (20.15 g soil/2,000 mL hexane) Total Resolved Pesticides = 3,270 mg/kg dry wt.

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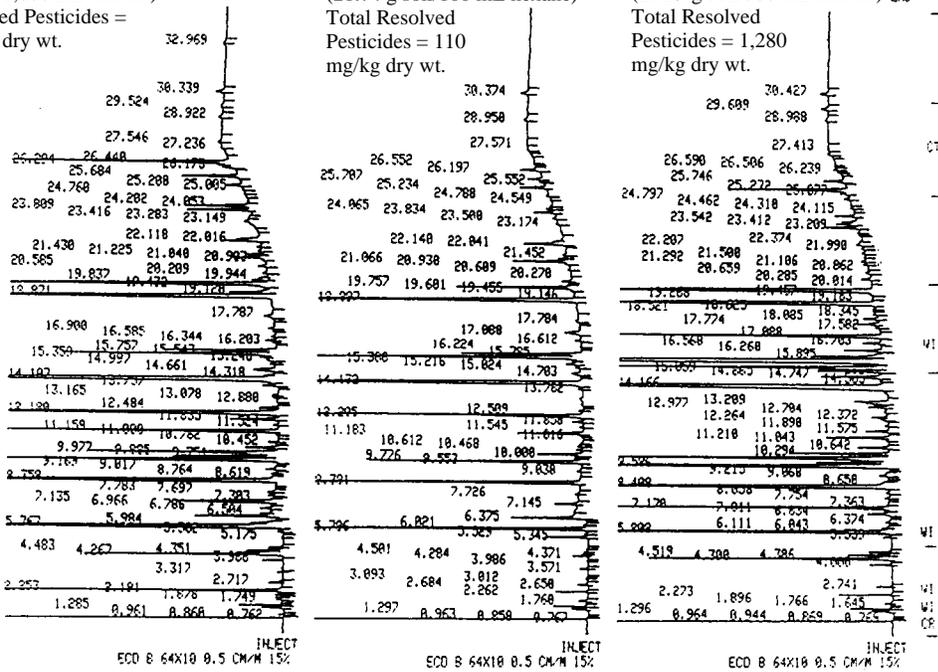


Figure 3. ECD GC profiles from two RESTEK Pesticide Standard solutions and hexane extracts of RMA soil and sludge samples volume-adjusted to yield >100% full-scale chromatograms of each sample to highlight qualitative similarity among the samples (i.e., a constant nominal weight [20 g] of each sample was extracted/diluted with different volumes of solvent to allow detection of lower-concentration components while major constituents all exceed 100% full scale). All chromatograms were obtained under identical instrument conditions (1.0 μL injected; 30 m, DB-1701 megabore column; 140°C [0.5 min] then 5°C/min to 275°C [7.5 min]; GC attenuation 64 × 10).

DCR treatability testing

Following these initial sample characterizations, 100-g aliquots of soil-amended Basin F sludge from above the liner (sample 24216) were removed for treatment with various quantities of natural and hydrophobized CaO. For these scoping studies, the samples were mixed by hand in a laboratory fume hood. No attempt was made to quantify any vapor emissions, because the third-party laboratory GC/MS analyses scheduled for the samples would allow quantification of volatile constituent removal.

After several iterations, it was determined that the high levels of sulfate (and possibly urea) in the waste (Table 2) were interfering with the CaO hydration inherent to the DCR process. As a result, uneconomically high levels of CaO reagent were required to obtain an acceptable product. To circumvent this problem, additional tests were undertaken to see if the waste could be pretreated with lime milk [hydrated $\text{Ca}(\text{OH})_2$] to tie up the sulfate (for example, as gypsum, $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and also remove the urea. These tests were very successful, and it was found that addition of 30% lime milk slurry in H_2O [$\text{Ca}(\text{OH})_2$ at approximately 10–13% of contaminated soil weight] was sufficient to tie up all the sulfate as gypsum. Then with the subsequent addition of dry CaO for the DCR reaction, it proceeded without delay and at economically feasible reagent addition levels. The lime milk pretreatment also proved to be an excellent way to remove ammonia by freeing up any ammonium originally present as ammonium sulfate and catalyzing the hydrolysis of urea to CO_2 and ammonia. In field applications, this approach could be used to release and reclaim ammonia through a specially designed emissions control capture system on the transportable treatment unit (TTU) prior to DCR processing.

The DCR process (unlike other pozzolan-based stabilization approaches) achieves its greatest technological advantage with wastes containing very high (>10–30%) levels of organics (e.g., diesel oil residuals and waste oils) because such materials facilitate transfer of the contaminants of concern onto the hydrophobic CaO reagent before the dispersion by chemical reaction takes place. From the data presented in the previous section, it was apparent that there would not be sufficient free organic (oily) phase in the sludge/soil matrix for optimum DCR processing. Based on the Basin F waste pile leachate data provided by GeoTrans*

it was believed that DCR treatment of the stockpiled soil could eliminate most, if not all, of the organic constituents that are currently leaching into the water being collected in the primary and secondary sumps at the site. However, stabilization of the higher-molecular-weight chlorinated pesticides might be improved by elevated concentrations of liquid-phase organics. Therefore, DCR treatability tests were undertaken with and without the addition of a benign food oil additive [Archer Daniels Midland (ADM) Regular Soy Oil Soap Stock]. It was determined in a wettability study that addition of the soy oil significantly improved the water repellency of the resultant product, so its addition was implemented as a standard procedure for all further tests. Under the limited time constraints of this project, however, no attempt was made to optimize the quantity (10% of soil weight) or type of food oil additive, and this remains a study area that should be pursued.

Independent laboratory testing

Following the initial scoping studies completed in the first week after the sample was received, 0.7 kg-scale batch preparations were undertaken to generate sufficient DCR-treated material for independent, third-party laboratory testing. Under the extremely short time-frame required to obtain initial laboratory results by 1 April 1995, little or no additional reagent optimization testing was possible, and the initial DCR product prepared for outside laboratory analyses was generated from 55% CaO addition to lime-milk pretreated soil plus 10% ADM soy oil. The total CaO reagent requirement (including that used to generate the lime milk slurry) for this sample was 64.7%. This material and a sample of the raw untreated waste soil (sample 24216) were packaged for shipment and transferred under chain of custody to the U.S. Army Corps of Engineers Environmental Laboratory in Hubbardston, Massachusetts, on 20 March 1995.

The following analyses were requested with a one-week turnaround time for the results:

FID GC (Diesel Range Total Petroleum Hydrocarbon Screening, EPA Method 8015M)

ECD GC (EPA Method 8081 Pesticide and PCB Analyses)

GC/MS (EPA Method 8270 Total Waste Analyses)

GC/MS (EPA Method 8260 Total Volatile Organic Analyses)

* K. Swingle FAX to J. Payne, 12/29/94.

TCLP (EPA Method 1311 Extraction for Volatiles [8240] and Semivolatiles [8270])

TCLP (EPA Method 1311 Extraction for Metals, ICP EPA Method 6010)

TCLP (EPA Method 1311 Extraction for Pesticides, EPA Method 8081).

After the sample was shipped, several additional larger-scale (1.5 kg) scoping experiments were completed with SOUND/*epic*'s high-intensity Eirich mixer. Through these later determinations it was found that, at the larger batch size and with more aggressive mixing, an acceptable product could be obtained with only a 23% CaO addition to lime-milk pretreated soil (total CaO addition, including preparation of lime milk was 27.4%).

Because the 27.4% CaO addition is more representative of what might be utilized in field applications, samples of this DCR-treated material were used to determine the following geophysical properties:

Optimum Moisture (ASTM 1557—Std. Proctor Density Measurement)

Compressive Strength (ASTM 2166—Use extruded Proctor Test samples)

Permeability (ASTM D 5084—Use extruded Proctor Test samples).

On 23 March 1995 approximately 13.6 kg of the 27.4% DCR-treated soil was delivered to Southern California Soil and Testing in San Diego, California. This material was used for Proctor maximum density/optimum moisture content determinations, and one of the compacted 4-in.-diam. cylinders from these tests was then used for determination of unconfined compressive strength. One of the other Proctor cylinders was prepared for permeability analysis and shipped via Federal Express under full chain of custody to Wallace-Kuhl & Associates in Sacramento, California.

A third Proctor cylinder was allowed to cure and was set aside for additional chemical analyses to assess the effects of lower DCR reagent utilization and sample compaction on pesticide sequestering and leachability. This third compacted cylinder sample was shipped via Federal Express under chain of custody to the U.S. Army Corps of Engineers Environmental Laboratory in Hubbardston, Massachusetts, on 10 April 1995. The same suite of analyses used for the raw untreated soil sample and loose (uncompacted) 64.7%

DCR-treated material was requested, with the proviso that the cylinder be crushed to the minimum extent necessary to pass through the 9.5 mm sieve (specified in the TCLP test) before leaching.

RESULTS AND DISCUSSION

64.7% DCR-treated product

Two basic analytical approaches are currently utilized by the U.S. EPA to evaluate reduction in contaminant mobility and total constituent concentrations. These are total waste analyses (organic solvent extraction followed by GC or GC/MS analyses) and leachability, usually by the Toxic Characteristic Leaching Procedure (TCLP). Table 3 presents the results of the SOUND/*epic* and Corps of Engineers Environmental Laboratory analyses of RMA sample 24216 before and after DCR treatment with 64.7% CaO. For these analyses, the raw soil and treated product were first analyzed for total volatile constituents using standard U.S. EPA 8260 methods, and then additional samples were extracted with an appropriate extraction solvent (e.g., methylene chloride) for semivolatile constituent analyses using U.S. EPA 3550/8270 procedures.

Total waste analyses

The volatile lower molecular weight alkylated and chlorinated benzenes in the RMA soil sample are essentially eliminated during the DCR process (Table 3). This is due to volatilization and/or steam stripping during the exothermic hydration of calcium oxide to calcium hydroxide, and in field implementation these constituents can be captured using transportable treatment units (TTUs) equipped with modular emissions control systems. Their removal is important in that they constitute the more water-soluble constituents, which can contribute to leachate or groundwater contamination if not otherwise controlled.

With regard to the pesticides and semivolatile organics in the RMA soils, it should be emphasized again that the DCR process is not a destructive technology. Any liquid (or solubilized) organic wastes treated by the DCR process are homogeneously dispersed into very fine (submicron-sized), hydrophobic particles. Since the pesticides present in the RMA soil samples are for the most part solids under ambient-temperature conditions, the ADM soy oils were added in an effort to partially "solubilize" them and facilitate

Table 3. Results of SOUND/epic and independent laboratory analyses* of RMA Basin F soil sample no. 24216 before and after DCR treatment with 64.7% CaO.

Waste constituent		Raw waste	DCR-treated	TCLP	TCLP
		before treatment	product	raw waste before treatment	DCR-treated product
Moisture content ¹ (%)		11.7	10.1		
Gravimetric oil and grease ¹ (mg/kg dry wt.)		10,700	12,900		
Total resolved hydrocarbons ¹ — FID GC (mg/kg dry wt.)		2,420	2,400		
Total resolved pesticides ¹ — ECD GC (mg/kg dry wt.)		3,270	2,150		
Individual constituents		(ng/g dry wt.)	(ng/g dry wt.)	(µg/L)	(µg/L)
Volatiles— GC/MS EPA 8260	Toluene	11	< 4.0	< 1.9	< 1.9
	Ethylbenzene	4.1 J	< 2.9	< 1.4	< 1.4
	m,p-Xylene	24	< 4.2	< 2.0	< 2.0
	o-Xylene	19	< 3.2	< 1.5	< 1.5
	n-Propylbenzene	3.9 J	< 2.9	< 1.4	< 1.4
	1,3,5-Trimethylbenzene	19	< 3.2	< 1.5	< 1.5
	1,2,4-Trimethylbenzene	28	< 2.9	< 1.4	< 1.4
	sec-Butylbenzene	2.3 J	< 2.9	< 1.0	< 1.0
	1,3-Dichlorobenzene	5.0 J	< 2.9	< 1.4	< 1.4
	1,4-Dichlorobenzene	19	< 3.2	0.47	< 0.081
	n-Butylbenzene	3.6 J	< 3.2	< 1.5	< 1.5
	1,2-Dichlorobenzene	11	< 3.4	< 1.6	< 1.6
	Pesticides and PCBs— ECD GC EPA 8081	Total PCBs	< 13,000	< 12,000	
Alpha-BHC		2,400	2,300		
Beta-BHC		< 136	540		
Aldrin		390,000	270,000	52	30
Dieldrin		260,000	180,000	90	51
Endrin		140,000	100,000	74	44
Endosulfan II		< 165	1,700		
Endrin aldehyde		20,000	7,900		
Endrin ketone		3,200	1,700		
Semivolatiles— GC/MS EPA 8270	N-Nitroso-di-n-propylamine	1,000	82 J		
	1,2,4-Trichlorobenzene	210	120	< 1.9	< 1.9
	Naphthalene	38	30	< 2.7	< 2.7
	Hexachlorobutadiene	6,900	630	3.4 J	< 1.8
	2-Methylnaphthalene	130	41 J		
	Dibenzofuran	34 J	< 11		
	Fluorene	70	32 J		
	Phenanthrene	110	92		
	Anthracene	88	<21		
	Fluoranthene	75	<16		
Chrysene	35 J	26 J			

* Analyses denoted by superscript¹ completed by SOUND/epic, all others by U.S. Army Corps of Engineers Environmental Laboratory, Hubbardston, Massachusetts.
J=Estimated value; less than the practical quantitation limit.

transfer to the CaO before hydration. Therefore, the slight increase in the gravimetric oil and grease value (Table 3) is no doubt due to the addition of the ADM soy oil. This increase is partially offset by oil sequestration/adsorption (even against organic solvent extraction) into the voids, cracks, and fissures of the newly

formed Ca(OH)₂ generated during the DCR process. More importantly, there is a significant decrease in the total resolved pesticides as measured by ECD GC at SOUND/epic, and this is also reflected in the individual pesticides reported by the Corps of Engineers laboratory. These reductions in total and individual pesti-

cide concentrations are believed to be due primarily to simple dilution from DCR reagent addition. Similar reductions are noted for most of the semivolatile constituents, with an order of magnitude decrease in hexachlorobutadiene (presumably from volatilization).

Leachability

The TCLP test is carried out using an acidic leaching solution to mimic an acidic landfill environment. Table 3 presents the results obtained on the RMA 24216 soil sample before and after DCR treatment. These are worst-case data, in that all TCLP testing on treated waste material was performed on the powdered DCR product in a noncompacted state. Leachable constituent analyses completed in Europe using the standard DEV S4 method demonstrated that compaction of the DCR-treated material reduces the leachable organic constituents by yet another order of magnitude compared with the highly dispersed, powdered DCR product (Gerschler 1984).

The only volatile constituent detected in the TCLP leachate of the raw soil was the aromatic 1,4-dichlorobenzene at 0.47 ppb. There was also a trace of the semivolatile hexachlorobutadiene at 3.4 ppb. Both of these constituents were below the reported detection limits in the DCR-treated product. There was a 40–43% reduction in leachable aldrin, dieldrin, and endrin (Table 3), which was probably due largely to dilution by the 64.7% CaO addition. While these decreases are significant, the reduction for endrin to 44 ppb did not satisfy the RCRA limit of 20 ppb, so additional testing was undertaken with the compacted 27.4% DCR-treated product (see below). There are no reported limits for aldrin and dieldrin.

For TCLP-extractable metals, there were significant reductions in leachability for several metals (most notably As, Cd, Cu, and Hg) (Table 4). In all cases, however, metals passed RCRA limits before treatment.

27.4% DCR-treated product

Total waste analyses

As was the case with the higher reagent addition, all of the volatile constituents present in the raw untreated soil were removed by the DCR process (Table 5). As a result,

volatiles will no longer be a contributing factor in leachate or potential groundwater contamination. In this low-reagent case (Table 5), the gravimetric oil and grease concentration increased more than was the case for the high-reagent addition (Table 3). This is particularly true with the loose 27.4% product before compaction and reflects the fact that less CaO was added, so there would be less sequestering of the soy oil against organic solvent extraction. With subsequent compaction, however, it is more difficult to extract the oil and grease with organic solvents, and a concomitant decrease in gravimetric oil and grease content is observed. As noted with the higher DCR reagent additions, significant reductions in total resolved pesticides by ECD GC are observed, and this reduction is further improved with compaction. No individual pesticide data are available for the loose 27.4% DCR-treated product, but with the compacted product, individual constituent concentrations are reduced, as expected, to an intermediate value between those observed in the raw soil and the 64.7% DCR-treated material. The reductions of aldrin, dieldrin, and endrin can be accounted for by a simple dilution with reagent CaO. The reduction of total resolved pesticides in the compacted sample, however, cannot be due entirely to dilution and implies some sequestration against solvent extraction due to either microencapsulation or macroencapsulation.

The effects of sample compaction are also reflected by decreases in the complexity of the ECD gas chromatograms obtained on the raw, DCR-treated loose, and DCR-treated compacted

Table 4. TCLP metals analyses of RMA Basin F soil sample no. 24216 before and after DCR treatment with 64.7% CaO.

<i>Waste constituent</i>	<i>TCLP concentrations from raw waste before treatment (µg/mL)</i>	<i>TCLP concentrations from DCR-treated product (µg/mL)</i>	<i>RCRA limits (µg/mL)</i>
Silver (Ag)	< 0.0089	< 0.0089	5.0
Arsenic (As)	0.13 J	< 0.075	5.0
Barium (Ba)	0.11	0.11	100.0
Cadmium (Cd)	0.017	< 0.0021	1.0
Chromium (Cr)	0.022	0.053	5.0
Copper (Cu)	36.0	1.6	n.a.
Mercury (Hg)	0.0009	0.0003	0.2
Lead (Pb)	< 0.84	< 0.84	5.0
Selenium (Se)	< 0.26	< 0.26	1.0

Analyses completed by U.S. Army Corps of Engineers Environmental Laboratory, Hubbardston, Massachusetts.

J = Estimated value; less than the practical quantitation limit.

n.a. = Not applicable

Table 5. Results of SOUND/epic and independent laboratory analyses* of RMA Basin F soil sample no. 24216 before and after DCR treatment with 27.4% CaO.

<i>Waste constituent</i>		<i>Raw waste</i>	<i>DCR-treated</i>	<i>DCR-treated</i>	<i>TCLP</i>	
		<i>before</i>	<i>product</i>	<i>product</i>	<i>DCR-treated</i>	
		<i>treatment</i>	<i>(loose)</i>	<i>(compact)</i>	<i>product</i>	
					<i>(compact)</i>	
Moisture content ¹ (%)		11.7	13.7	16.0		
Gravimetric oil and grease ¹ (mg/kg dry wt.)		10,700	41,800	15,200		
Total resolved hydrocarbons ¹ — FID GC (mg/kg dry wt.)		2,420	2,400	N.A.		
Total resolved pesticides ¹ — ECD GC (mg/kg dry wt.)		3,270	2,110	1,100		
<i>Individual constituents</i>		<i>(ng/g dry wt.)</i>	<i>(ng/g dry wt.)</i>	<i>(ng/g dry wt.)</i>	<i>(µg/L)</i>	
Volatiles— GC/MS EPA 8260	Toluene	11	N.A.	< 16	< 1.9	
	Ethylbenzene	4.1 J	N.A.	< 11	< 1.4	
	m,p-Xylene	24	N.A.	< 16	< 2.0	
	o-Xylene	19	N.A.	< 12	< 1.5	
	n-Propylbenzene	3.9 J	N.A.	< 11	< 1.4	
	1,3,5-Trimethylbenzene	19	N.A.	< 12	< 1.5	
	1,2,4-Trimethylbenzene	28	N.A.	< 11	< 1.4	
	sec-Butylbenzene	2.3 J	N.A.	< 11	< 1.4	
	1,3-Dichlorobenzene	5.0 J	N.A.	< 11	< 1.4	
	1,4-Dichlorobenzene	19	N.A.	< 12	< 1.5	
	n-Butylbenzene	3.6 J	N.A.	< 12	< 1.5	
	1,2-Dichlorobenzene	11	N.A.	< 13	< 1.6	
	Pesticides and PCBs— ECD GC EPA 8081	Total PCBs	< 13,000	N.A.	< 1400	Rep. 1
Alpha-BHC		2,400	N.A.	< 1350		
Beta-BHC		< 136	N.A.	< 1304		
Aldrin		390,000	N.A.	350,000	4.0	4.8
Dieldrin		260,000	N.A.	220,000	31.0	42.0
Endrin		140,000	N.A.	120,000	20.0	28.0
Endosulfan II		< 165	N.A.	9,300		
Endrin aldehyde		20,000	N.A.	8,600		
Endrin ketone		3,200	N.A.	11,000		
Semivolatiles— GC/MS EPA 8270	N-Nitroso-di-n-propylamine	1,000	N.A.	< 130		
	1,2,4-Trichlorobenzene	210	N.A.	< 180		
	Naphthalene	38	N.A.	63 J		
	Hexachlorobutadiene	6,900	N.A.	2800	0.65	
	2-Methylnaphthalene	130	N.A.	100 J		
	Dibenzofuran	34 J	N.A.	< 53		
	Fluorene	70	N.A.	< 77		
	Phenanthrene	110	N.A.	130 J		
	Anthracene	88	N.A.	< 100		
	Fluoranthene	75	N.A.	< 77		
Chrysene	35 J	N.A.	< 63			

* Analyses denoted by superscript¹ completed by SOUND/epic, all others by U.S. Army Corps of Engineers Environmental Laboratory, Hubbardston, Massachusetts.

J = Estimated value; less than the practical quantitation limit.

N.A. = Not analyzed

samples (Fig. 4). Quite clearly there is a dramatic reduction in the complexity and total levels of pesticides after DCR treatment and sample compaction, and this is also reflected by the data in Table 6, which summarizes the results from selected constituent analyses in the raw soil, 64.7%

DCR reagent addition (loose), and 27.4% DCR reagent addition (with and without compaction).

Figure 5 presents the FID GC profiles obtained on the RMA soil sample before and after DCR treatment. Because 10% ADM soy oil was added to aid in sequestering the pesticides during the

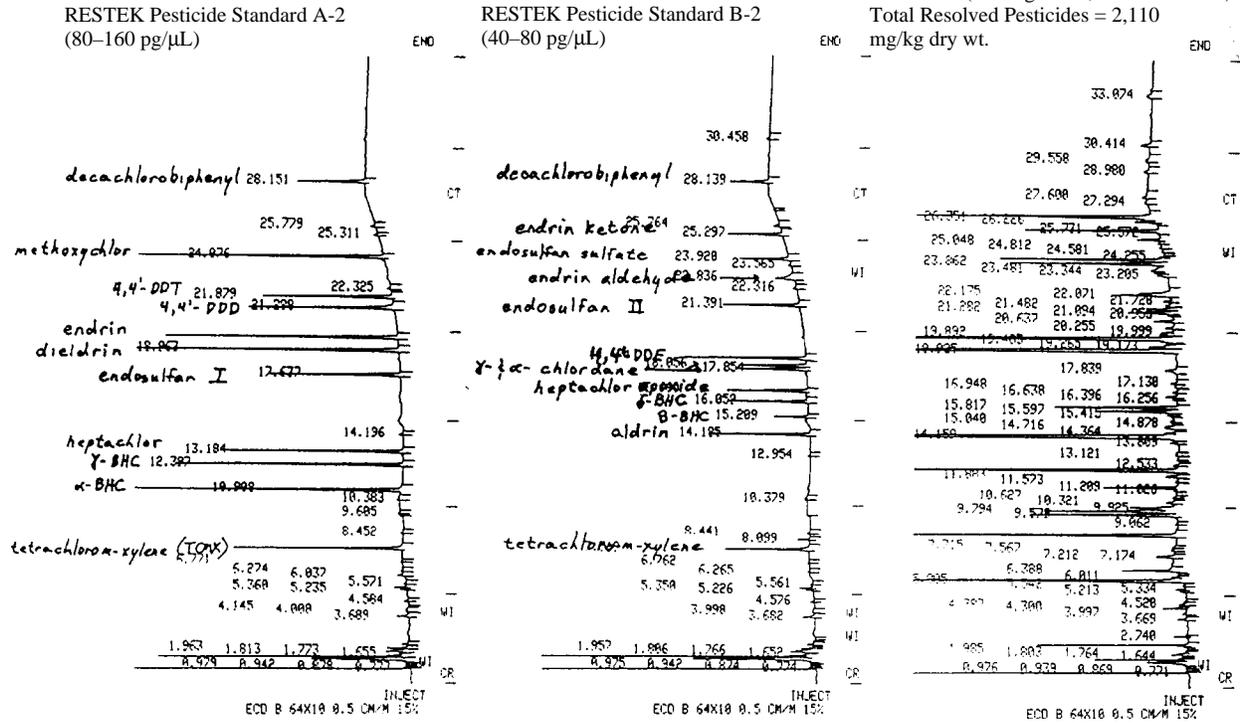
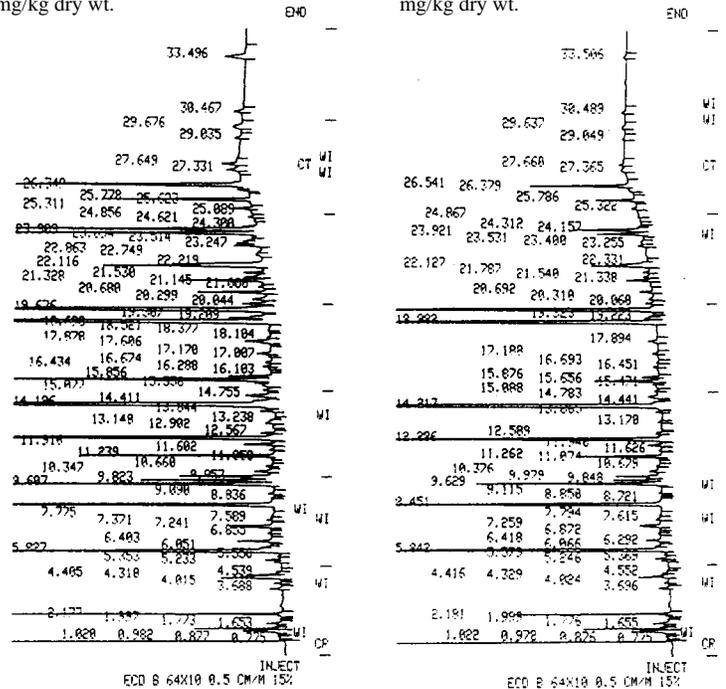


Figure 4. ECD GC profiles from two RESTEK Pesticide Standard solutions and hexane extracts of RMA Soil-Amended Basin F sludge from above the liner (sample no. 24216) before and after stabilization with the DCR process. 27.4% DCR Loose designates 27.4% CaO addition and solvent extraction of the finely dispersed, dry powder obtained immediately after the DCR process. 27.4% DCR Compact designates solvent extraction of the sample following the same percentage DCR reagent addition and compaction for Proctor maximum density and permeability determinations. Identical soil sample and extraction volumes were used in all three RMA samples to facilitate qualitative comparisons, and all chromatograms were obtained under identical instrument conditions (1.0 μ L injected; 30 m, DB-1701 megabore column; 140°C [0.5 min] then 5°C/min to 275°C [7.5 min]; GC attenuation 512 \times 10).

27.4% DCR Loose RMA "Soil-Amended Basin F Sludge from Above the Liner" Sample No. 24216 (17.62 g soil/2,000 mL hexane) Total Resolved Pesticides = 2,110 mg/kg dry wt.

27.4% DCR Compact RMA "Soil-Amended Basin F Sludge from Above the Liner" Sample No. 24216 (17.79 g soil/2,000 mL hexane) Total Resolved Pesticides = 1,100 mg/kg dry wt.



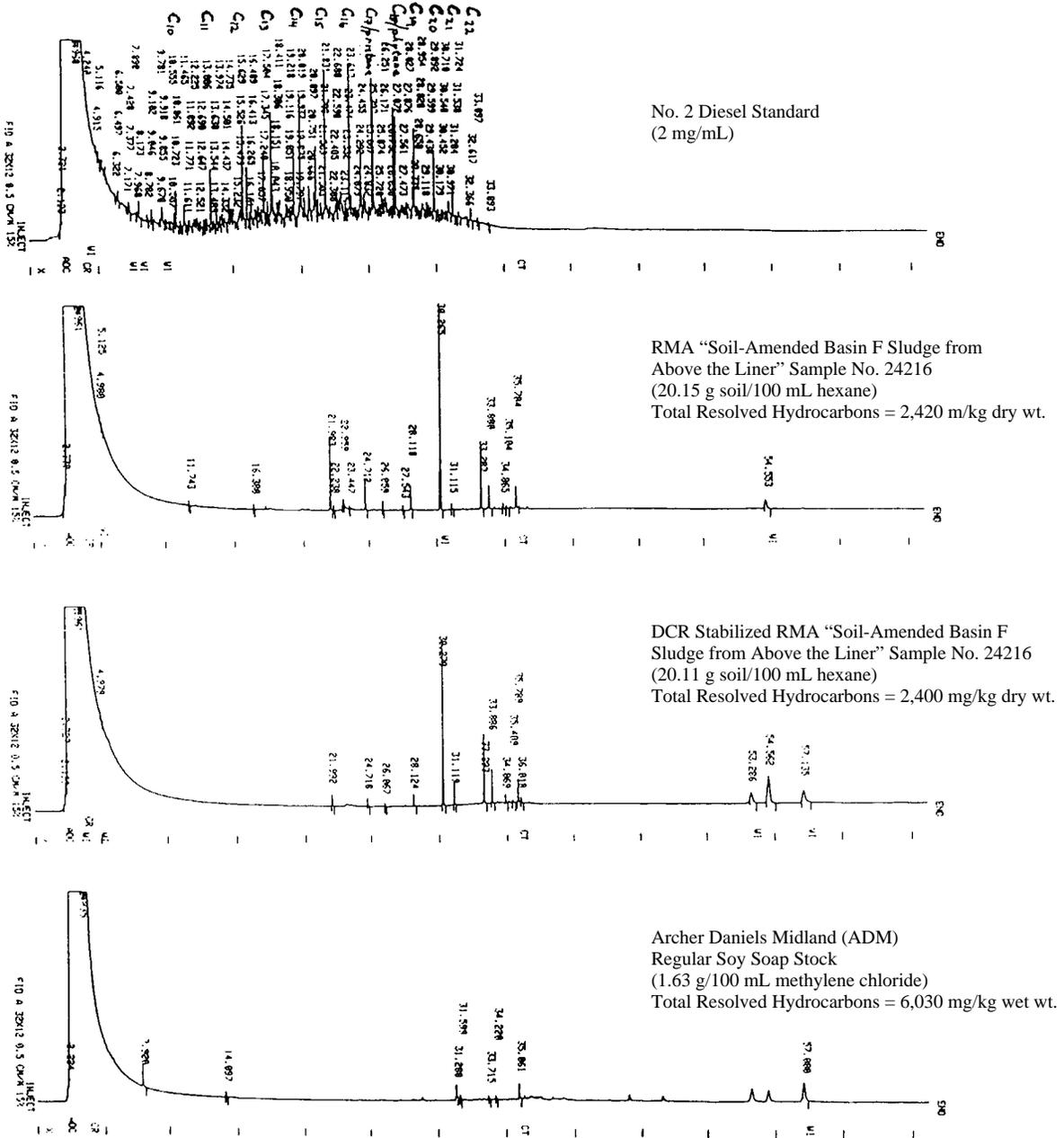


Figure 5. FID GC profiles from a No. 2 Diesel Standard containing *n*-alkanes from *n*-C₁₀ to *n*-C₂₂ and hexane extracts of RMA Soil-Amended Basin F sludge from above the liner (sample no. 24216) before and after stabilization with the DCR process. To facilitate comparison of total resolved hydrocarbons between the RMA samples, a constant nominal weight (20 g) of sample was extracted with a constant volume (100 mL) of solvent. Because ADM regular soy soap stock was used to aid in sequestering the pesticides during the DCR process, a separate chromatogram of the soap stock diluted in methylene chloride is also presented for reference. All chromatograms were obtained under identical instrument conditions (1.0 μL injected; 30 m, OV-1 narrow bore column; 50°C [4 min] then 7°C/min to 275°C [30 min]; GC attenuation 32 × 12).

Table 6. Summary of selected constituent analyses of RMA Basin F soil sample no. 24216 at different levels of DCR reagent addition with and without compaction.

<i>Waste constituent</i>	<i>Raw waste before treatment</i>	<i>64.7% DCR reagent addition (loose)</i>	<i>27.4% DCR reagent addition (loose)</i>	<i>27.4% DCR reagent addition (compacted)</i>
Gravimetric oil and grease ¹ (mg/kg dry wt.)	10,700	12,900	41,800	15,200
Total resolved pesticides ¹ —ECD GC (mg/kg dry wt.)	3,270	2,150	2,110	1,110
Aldrin (ng/g dry wt.)	390,000	270,000	N.A.	350,000
Dieldrin (ng/g dry wt.)	260,000	180,000	N.A.	220,000
Endrin (ng/g dry wt.)	140,000	100,000	N.A.	120,000
1,2,4-trichlorobenzene (ng/g dry wt.)	210	120	N.A.	< 16
Hexachlorobutadiene (ng/g dry wt.)	6,900	630	N.A.	750

* Analyses denoted by superscript¹ completed by SOUND/**epic**, all others by U.S. Army Corps of Engineers Environmental Laboratory, Hubbardston, Massachusetts.
N.A. = Not analyzed

DCR process, a separate chromatogram of the soy oil is also presented. Clearly, the DCR process does not liberate or add any undesirable aromatic or petroleum hydrocarbon constituents to the soil. The only change in the chromatogram of the DCR-treated sample is the addition of three higher molecular weight peaks (retention times 53.286, 54.562, and 57.135 min) from biodegradable free fatty acids or triglycerides in the soy oil.

Leachability

As was the case with the 64.7% DCR-treated sample, no volatile constituents were identified in the leachate of the 27.4 % DCR-treated soil (Table 5). The TCLP leachable pesticide data show an even

more significant improvement with DCR-treated sample compaction. Significant reductions in leachable aldrin, dieldrin, and endrin were observed after the initial DCR treatment at 64.7% reagent addition (Table 7). When the reagent addition was dropped to 27.4% and the sample was compacted, even further reductions in leachable pesticides were observed. In contrast to the 64.7% reagent addition, these reductions at the low reagent addition cannot be attributed solely to reagent dilution of the soil. Because of the interest in pesticide leachability, these later tests were run in duplicate, and in one case the leachable endrin was measured right at the RCRA limit of 20 ppb. In the other replicate, endrin was just slightly higher at 28 ppb.

Table 7. Summary of TCLP leachable constituent analyses on RMA Basin F soil sample no. 24216 before and after DCR treatment.

<i>Waste constituent</i>	<i>Raw waste before treatment (µg/L)</i>	<i>64.7% DCR reagent (loose) (µg/L)</i>	<i>27.4 % DCR reagent (compact) (µg/L)</i>		<i>RCRA regulatory limit (µg/L)</i>	<i>CA STLC regulatory limit (µg/L)</i>
			<i>Rep. 1</i>	<i>Rep. 2</i>		
Aldrin	52	30	4.0	4.8	n.a.	140
Dieldrin	90	51	31	42	n.a.	800
Endrin	74	44	20	28	20	20
1,2,4-trichlorobenzene	<1.9	<1.9	N.A.		n.a.	n.a.
Hexachlorobutadiene	3.4 J	<1.8	N.A.		500	n.a.

CA STLC = State of California Soluble Threshold Limit Concentration

J = Estimated value; less than the practical quantitation limit.

n.a. = Not applicable

N.A. = Not analyzed

Table 8. Geophysical testing of DCR-treated RMA Basin F soil sample no. 24216.

<i>Sample</i>	<i>Maximum density (Mg/m³)</i>	<i>Optimum moisture content (%)</i>	<i>Unconfined compressive strength (MPa)</i>	<i>Hydraulic conductivity (permeability) (cm/s)</i>
Raw material (soil-amended sludge)	N.A.	N.A.	Very low	N.A.
Soil-amended sludge plus 27.4% DCR reagents	1.65	14.3	1.31	1.6 × 10 ⁻⁷

Analyses completed by Southern California Soil & Testing and Wallace-Kuhl & Associates.
N.A. = Not analyzed.

Geophysical properties testing

While the heterogeneous raw untreated starting material was a dry and friable soil/silt mixture with a very low unconfined compressive strength (UCS), the DCR-treated product exhibited a very impressive UCS of 1.31 MPa (Table 8). A maximum density of 1.65 Mg m⁻³ was obtained at 14.3% moisture content, and the permeability (hydraulic conductivity) was very low at 1.6E-7 cm s⁻¹ and in the range of that required for clay cap materials. Once compacted into a hydrophobized soil body (as in an onsite Class I landfill) additional isolation of the contaminants would be derived from a combination of micro- and macroencapsulation. Because of the low permeabilities and hydrophobic, water-repelling nature of the compacted material, water could not easily penetrate the soil body, and with in situ carbonation (formation of limestone, CaCO₃), the permanence and water-repellency of the material would improve with time.

CONCLUSIONS

Based on the results observed to date and presented in this report, it is clear that the DCR process can be used to treat the Basin F waste soil nonthermally to produce a dry, homogeneous, soil-like material with desirable physical properties that on compaction can achieve the following treatment goals: reduction of all leachable volatiles to nondetectable levels, confinement of all metals to below RCRA TCLP levels, and a decrease in pesticide leachability to levels approaching RCRA standards. For example, endrin TCLP concentration was reduced from 74 µg/L to 20–28 µg/L (regulatory limit = 20 µg/L). In several cases, reductions in pesticide leachability could be attrib-

uted to simple dilution with the CaO reagent. In other cases, however, microencapsulation and/or macroencapsulation also played a role in reducing pesticide leachability.

DCR is different from cement- or pozzolan-based approaches in that it does not rely solely on formation of a monolithic structure of hydrated silicates and carbonates to incorporate and/or macroencapsulate the hazardous constituents. The DCR process first traps the mobile organic constituents into nonmobile, hydrophobic, pulverized solids literally at a submicron level (microencapsulation). Then, as with other cement- or pozzolan-based approaches, it is possible to take advantage of macroencapsulation in large, compact soil bodies when appropriate. These hydrophobic compacted soil bodies are surrounded by a self-healing CaCO₃ crust that increases in thickness and stability with time. Unfortunately, the accelerated nature of this treatability work (to date) has not allowed time to achieve any benefit of additional microencapsulation due to this gradual carbonation process. Such in situ carbonation might serve to improve TCLP results as well as reduce permeability and leachability in a groundwater environment.

These studies demonstrate the value of additional work to optimize the DCR mix design. Such optimization would include determinations of: 1) the lime-milk concentrations to optimize the release of ammonia to the emissions control system; 2) the amount of benign oil to adequately suppress odor, improve permeability, and aid in solubilization/transfer of the pesticides onto the CaO during the pre-DCR mixing phase; 3) the degree of hydrophobicity necessary for this particular waste; and 4) the amount of reagent necessary to provide homogeneity and complete volatile removal.

Of the samples collected, we selected the worst-case soil in terms of difficulty for the DCR technology. For an accurate assessment of reagent quantities required for more typical wastes, a more representative sample should be assembled. It can be reasonably assumed that for a more representative sample, reagent quantities will be reduced below the 27% additions achieved in these studies and that TCLP results would be improved. Ideally, the optimum design should achieve the regulatory and client goals, while minimizing materials handling, energy, and reagent inputs.

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13. ABSTRACT (<i>Maximum 200 words</i>) Many military installations have soil contamination problems that range from heavy metals to petroleum products. Rocky Mountain Arsenal (RMA) Basin F contains high concentrations of salts, heavy metals, ammonia, urea, and organics. The Dispersion by Chemical Reaction (DCR) process leads to a reduction in the mobility of the organic and inorganic constituents by first removing volatile constituents via steam stripping and volatilization, then trapping the nonvolatile contaminants in a nonmobile phase (microencapsulation), and finally compacting the treated material into large soil bodies (macroencapsulation). This report summarizes the results of the DCR testing of soil-amended Basin F sludge from RMA. The primary focus of this study is on pesticide leachability. The DCR process used to treat the Basin F waste soil produced a dry, homogeneous, soil-like material with desirable physical properties that on compaction achieved the following remediation goals: reduction of all leachable volatiles to nondetectable levels, confinement of all metals to below RCRA TCLP levels, and a decrease in pesticide leachability to levels approaching RCRA standards. For example, endrin TCLP concentration was reduced from 74 µg/L to 20–28 µg/L (regulatory limit = 20 µg/L). In several cases, reductions in pesticide leachability could be attributed to simple dilution with the calcium oxide (CaO) reagent. However in other cases, microencapsulation and/or macroencapsulation also played a role in reducing pesticide leachability. Additional work is necessary to optimize the amounts of lime-milk, hydrophobic CaO, and benign oil used in the processing of RMA Basin F waste soils. Ideally, the optimum design should achieve the regulatory and client goals, while minimizing materials handling, energy, and reagent inputs.					
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