SECTION TWO

BENCH-SCALE TREATABILITY STUDY METHODS, PROCEDURES, AND ANALYTICAL PARAMETERS

SECTION TWO
BENCH-SCALE TREATABILITY STUDY METHODS, PROCEDURES AND ANALYTICAL PARAMETERS

MARCOR subcontracted Kiber Environmental Services, Inc. (Kiber) of Atlanta, Georgia to perform the reagent blending and analytical services on estuarine sediments collected from the Newtown Creek test site. Kiber has extensive experience with treatability testing associated with soil stabilization. A copy of their laboratory QA was submitted with MARCOR's technical proposal.

Bench-scale testing was completed in two phases. During the initial phase a total of five (5) mixtures of ACT reagent compounds were added to separate and distinct sample aliquots. See Table 2-1 for mixture listings. The ACT mixture which was determined most effective in the reduction of chemical contaminants in this phase was then used to prepare a 30 day cure batch. The results for the 30 day cure batch will be used to evaluate the overall effectiveness of ACT chemical stabilization at the bench scale.

Summarized in the subsections below are the sample preparation and chemical fixation procedures utilized over the course of the bench-scale study. A copy of the Kiber (1996) report is presented as Appendix B.

2.1 SEDIMENT SAMPLE PREPARATION

Two (2) Five (5)-gallon buckets of "black, oily sludge" sediment test material was forwarded by Brookhaven National Laboratory (BNL) to Kiber under Chain-of-Custody documentation on October 12, 1995. Upon receipt by Kiber, the head space of both containers were monitored for volatile organic compounds using a photoionization detector, then resealed and refrigerated at a temperature of approximately 4°C until later use.

Table 2-1

ACT Reagent Mixtures Used in 7 Day Cure Treatment Study
BREP Bench-Scale Treatability Study
MARCOR Environmental Services, Inc.

ACT Reagent Type	Kiber Sample Number	Reagent Addition (%)	Water Addition (%)
SWT-27T	1397-001	20	25
SWT-27	1397-002	20	25
SWT-25	1397-003	20	24
SWT-23	1397-004	25	28.8
SWT-23	1397-005	12.5	14.4

Prior to commencement of testing activities, chilled untreated sediment was homogenized in a mixing tub. Coarse-grained particles greater than 0.5 inches in diameter were separated and discarded. Upon homogenization of both buckets of test material, the sediment was returned into the original 5-gallon sample containers and placed back into refrigeration (Kiber 1996).

2.2 INITIAL SEVEN (7) DAY CURE CHEMICAL FIXATION TREATMENT PROCEDURES

A total of five (5) ACT reagent mixtures (Table 2-1) were prepared and used as part of the Initial Seven (7) Day Cure Evaluation. A unique ACT reagent design and potable water mix was added to each sediment 200 gram sample aliquot, then placed into a blending chamber. The percent of reagent and water added to each aliquot was based on the initial weight of the untreated aliquot. Each aliquot was then homogenized in the blending chamber for approximately three (3) minutes; mechanical blending occurred at a rate of approximately 40-60 rotations per minute.

Volatile organic compound emissions were monitored every 15 seconds using a photoionization detector (PID) mounted over the treatment chamber. Sample temperature measurements were also recorded throughout the duration of the seven (7) day cure period using a digital temperature probe. The purpose of temperature monitoring was to qualitatively evaluate the rate of reaction generated by the addition of hydrated ACT reagent mixtures. In general, very low levels of volatile organic emissions were observed. The results for PID and temperature monitoring are presented in Table 8 of the Kiber report.

Prepared sample aliquots were allowed to cure for a period of seven (7) days in an oven maintained at a constant 47°C to simulate full-scale field conditions where high heats of hydration are expected in treated stock-piled soil. Based on the results for the seven (7) day cure and other previously completed studies using ACT technology, it is anticipated that the heats of hydration curing temperature during full-scale conditions will be higher than this temperature. It was not feasible to

simulate higher curing temperatures due to rapid heat loss from the small volume of sample (Kiber, 1996).

2.2.1 Seven (7) Day Cure Mechanical Strength Testing

Penetrometer strength and volumetric expansion tests were conducted during and after the seven (7) day cure period to determine the physical properties of the treated samples. These mechanical tests and their results are summarized below, and on Table 9 of the Kiber report.

2.2.1.1 Penetrometer Strength Testing

Penetrometer strength tests were conducted during the 1st, 2nd, 3rd and 7th days of the seven (7) day cure period. Penetrometer strength tests were conducted using a Humboldt Pocket Penetrometer. Penetrometer strength tests varied between 0.5 tons/ft^2 for sample mixture 1397-005 (ACT mixture SWT-23) to greater than 4.5 tons/ft^2 (mixtures 1297-001 through 1397-004) (Kiber, 1996).

2.2.1.2 Volumetric Expansion Testing

Volumetric expansion testing was performed on all sample aliquots prior to, and at the conclusion of the seven (7) day cure period. All soil aliquots were first weighed, then placed into a cylindrical mold whereby an initial, untreated sample volume was determined. The aliquots were next removed from containment and blended with the predescribed ACT formulations before being placed back into the molds for curing purposes. At the conclusion of the 7 day cure period, each aliquot of treated material was measured for expansion or contraction. The percent volumetric expansion or contraction was determined using the following equation:

$$\text{Percent Volumetric Expansion/Shrinkage} = \left[\frac{(\text{Final Volume} - \text{Initial Volume})}{\text{Initial Volume}}\right] * 100$$

Results for volumetric expansion are presented on Table 9 of the Kiber report. Review of these data report that volumetric expansion between treated samples varied between -1 and 27%. Greatest expansion was noted in the 1397-004 (25% SWT-23 mixture) and 1397-003 (20% SWT-25 mixture) where volumetric expansions of 27% and 22% were observed, respectively. A percent volume shrinkage of -1% was noted in the 1397-005 (12.5% SWT-23 mixture) sample.

2.2.2 Seven (7) Day Cure Chemical Performance Testing

At the conclusion of the 7 day cure period, each treated sediment aliquot was analyzed for chemical analytical compounds. The results for chemical analytical and mechanical strength testing were evaluated against untreated sample results for samples collected from Newtown Creek Sediment to determine the most effective design mix of ACT reagent compounds to be used in the 30 day cure. Analytical parameter groups include PAHs, Pesticides, Herbicides, PCBs, TAL Metals and Material pH. A summary of test methods for both untreated and seven (7) day cure parameters is presented below. The results of analysis is presented in Section 5.3 and Tables 10-14 of the Kiber report. A review of analytical results indicated that ACT reagent design mixture SWT-25 (20% mixture) was most effective in reducing contaminant levels in site sediment samples after the seven (7) day cure period.

2.3 30 DAY CURE CHEMICAL FIXATION TREATMENT PROCEDURES

Methods and procedures employed for the 30 day cure batch included the completion of a glovebox volatilization study and the analysis of additional geotechnical and chemical analytical samples. The ACT reagent design mixture chosen for the 30 day cure batch was SWT-25. At the request of MARCOR, Kiber prepared the sample with the addition of a 25% mixture of the ACT compound. The purpose for increasing the amount of ACT (+5%) was to enhance treatment characteristics.

2.3.1 Glovebox Volatilization Monitoring

Glovebox volatilization monitoring was conducted to evaluate the potential for volatile compound emissions which could occur in the field during the completion of pilot and full-scale treatment operations. To check for variation in glovebox test results, this study was performed in duplicate.

Prior to initiation of the mixing, all required equipment and materials were placed into the glove-box. To minimize the potential for pretest contamination, the sample aliquots, reagent mixtures and water were sealed in air-tight containers until commencement of mixing operations.

A breathing quality air supply was connected to the influent side of the glovebox. The containment chamber was then purged for approximately 12 minutes. Three (3) carbon sampling cartridges were connected in series in addition to an air evacuation sample pump to the effluent sample port of the glovebox. A slight positive pressure was maintained within the glove box by inducing an air inflow rate of 4 liters per minute (Lpm) throughout the duration of the 30 day cure period; air was evacuated from the glovebox at a rate of 3 Lpm. The purpose for maintaining positive pressure was to "flush" volatile organic compounds across the air sample cartridges.

ACT reagent and water was blended to the aliquot placed into each glovebox for three (3) minutes at a rate of approximately 40 to 60 revolutions per minute. Upon completion of the mixing process, the treated soil was placed into unsealed sample jars for an additional 120 minutes. PID and glovebox temperature readings were recorded at regular intervals over the 2 hour post mixing period.

Glovebox testing air sample cartridges were analyzed for total volatile organic compounds according to EPA Method 8260. For verification purposes, Kiber was also instructed to prepare and perform an additional glovebox test. The three (3) resultant air sample cartridges were forwarded to BNL under Chain-of-Custody documentation on December 14, 1995.

Qualitative PID results report very low volatile organic compound emissions (0.6 ppm maximum) within the final mixture and duplicate gloveboxes over the course of the study. These data suggest that emissions of volatile organic compounds may be minimal during Pilot and Full-Scale operations.

Glovebox temperature monitoring data reported maximum temperatures of 25.0 and 25.6°C, for the final mixture and duplicate samples, respectively. The pretreatment temperature was 18.9°C.

Results for Kiber air sample cartridge results report that methylene chloride (a common laboratory contaminant) and Trichloroethene were the only volatile organic compounds (VOCs) present in all three (3) samples submitted for analysis. The highest concentration for trichloroethene in the final mixture sample was 1.9 µg in the "C" air sample cartridge located farthest from the effluent sample port. The highest trichloroethene concentration (2.8 µg) in the duplicate sample was collected from the middle "B" sample cartridge. Other VOCs detected in at least one cartridge include Bromodichloromethane, Chloroethane, 2-Hexanone, 4-Methyl-2-pentanone, 1,1,1-Trichloroethene and Vinyl Acetate (Kiber, 1996).

At the conclusion of glovebox testing, the treated samples (sample 1397-003 and the duplicate) were permitted to cure in an oven maintained at 47°C for a period of 30 days.

2.3.2 30 Day Cure Mechanical Strength Testing

Penetrometer Strength Tests were conducted on day 1,2,3,7,14 and 30 of the curing period. Volumetric Expansion of treated samples was also determined at the completion of the test period (day 30 of test). The results for mechanical strength testing (Table 17 of Kiber report) indicate that the final mixture sample reached a penetrometer test strength of greater than 4.5 tons/ft^2 after 14 days of curing. Volumetric expansion for the same sample after 30 days was 3% (Kiber, 1996). Other physical parameter testing included Bulk Density, Unconfined Compressive Strength and Permeability.

2.3.3 30 Day Cure Chemical Analysis

At the conclusion of the curing period, the treated sediment aliquot was analyzed for chemical constituents and waste characteristic parameters. Sample analyte groups include VOCs, PAHs, Semivolatiles (SVOCs) Pesticides, Herbicides, Dioxins/Furans, PCBs, TAL Metals, RCRA Metals, Reactive Cyanide, Reactive Sulfide, Ignitability, Material pH, and Moisture Content. Toxic Leaching Procedure (TCLP) analysis was also conducted for all above referenced compound (excluding cyanide and sulfides) groups. A discussion of the analytical program is presented below.

2.4 ANALYTICAL PROGRAM

Chemical and physical parameter testing was conducted at three discrete periods throughout the Bench-Scale Treatability Study. Base-line (untreated sediment) sampling (Section 3.1) was conducted prior to ACT chemical fixation treatment. Treatment performance sampling was completed after an initial seven (7) day cure period and upon conclusion of the 30 day cure period. A summary of sample parameters and respective test methods are presented below. A discussion of results for base-line and 30 day cure sampling is presented in Section 3 of this document.

2.4.1 Untreated Sediment Analysis

Chemical compound and physical parameter base-line analysis was completed prior to ACT chemical fixation of untreated Newtown Creek sediment samples. The purpose for untreated sediment chemical compound analysis was to determine the untreated characteristics of representative New York/New Jersey Harbor sediments, and to provide a frame of reference to gauge the effectiveness of ACT as a potential remedial alternative.

Aliquots of homogenized, untreated sediment were submitted for analysis sampling by Kiber; a split sample of homogenized test material was also analyzed by BNL for comparison purposes. A

summary of untreated sediment analytical parameters are identified in Table 2-2. All analyses were conducted by Kiber with the following exceptions:

* Total Dioxins/Furans (Triangle Laboratories of Durham, North Carolina).
* Total Herbicides (Analytical Services, Inc. of Norcross, Georgia).
* Total Organic Carbon (Analytical Services, Inc. of Norcross, Georgia)

All BNL analysis was conducted by Triangle Laboratories.

Geotechnical testing was also completed by Kiber to provide initial, base-line information on the physical and handling properties of untreated sediment. Test analyses and corresponding methods are presented below in Table 2-3. A summary of untreated sediment analytical results is presented in Section 3.1.

2.4.2 Initial Seven (7) Day Cure Chemical Compound and Physical Parameter Analyses

At the conclusion of the seven (7) day cure period, one (1) sample was collected from each of the five (5) treated aliquots. Results for chemical sampling was used in conjunction with the results for mechanical strength testing to determine that ACT reagent compound SWT-25 (20% mixture) was the best candidate reagent type to be used for the 30 day cure. Chemical groups and physical parameters included for analyses are identified below in Table 2-4. A summary of 7 day cure sediment sample results is presented in Section 3.3 and Tables 10-14 of the Kiber (1996) report (Appendix B).

2.4.3 30 Day Cure Chemical Compound and Physical Parameter Sampling

At the conclusion of the 30 day cure period, aliquots of SWT-25 (25% mixture) were analyzed by Kiber, except as noted above in Section 2.4.1, and by BNL for the chemical groups and physical

DRAFT

Table 2-2

Untreated Sediment Sample Parameters
BREP Bench-Scale Treatability Study
MARCOR Environmental Services, Inc.

Compound Group	Analytical Method
Total Volatiles	EPA Method 8260
Total Polycyclic Aromatic Hydrocarbons	EPA Method 3550/8270A
Total Pesticides	EPA Method 3550/8080
Total Polychlorinated Biphenyls	EPA Method 3550/8080
Total Herbicides	EPA Method 8150
Total Dioxins/Furans	EPA Method M8280
Total TAL Metals	EPA Method 3051/6010A/7471
Total Organic Carbon	EPA Method 9060
Material pH	EPA Method 9045

Table 2-3

Untreated Sediment Sample Geotechnical Analysis Parameters
BREP Bench-Scale Treatability Study
MARCOR Environmental Services, Inc.

Geophysical Parameter	Analytical Method
Moisture Content	ASTM D 2216
Bulk Unit Weight/Specific Gravity	ASTM D 5057

Table 2-4

DRAFT

7 Day Cure Treatability Study Sample Parameters
BREP Bench-Scale Treatability Study
MARCOR Environmental Services, Inc.

Compound Group	Analytical Method
Total PAHs	EPA Method 3550/8270A
Total PCBs/Pesticides	EPA Method 3550/8080
Total Herbicides	EPA Method 8150
Total TAL Metals	EPA Method 3051/6010A/7471
Material pH	EPA Method 9045

parameters identified below in Table 2-5. A summary of 30 day cure sediment sample results is presented in Section 3.2.

Table 2-5

30 Day Cure Sample Parameters
BREP Bench-Scale Treatability Study
MARCOR Environmental Services, Inc.

Compound Group	Analytical Method
Total Volatiles	EPA Method 8260
TCLP Volatiles	EPA Method 1311/8260
Total PAHs	EPA Method 3550/8270A
TCLP Semivolatiles	EPA Method 1311/3510/8270A
Total PCBs/Pesticides	EPA Method 3550/8080
TCLP Pesticides	EPA Method 1311/3510/8080
Total Herbicides	EPA Method 8150
TCLP Herbicides	EPA Method 1311/8150
Total Dioxins/Furans	EPA Method 8290
Total TAL Metals	EPA Method 3051/6010A/7471
TCLP RCRA Metals	EPA Method 1311/3015/6010A/7470
Reactive Cyanide	EPA Method 7.3.3
Reactive Sulfide	EPA Method 7.3.4
Ignitability	EPA Method 1020A
Material pH	EPA Method 9045
Bulk Density	ASTM D 2937
Moisture Content	ASTM D 2216
onfined Compressive Stre	ASTM D 2166
Permeability	ASTM D 5084