COMPLETION REPORT

LANDFARMING TECHNOLOGY FOR ON-SITE BIOREMEDIATION OF HYDROCARBON-CONTAMINATED SOILS: LABORATORY AND FIELD-SCALE EVALUATION

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Introduction

Contamination of air, surface water, sediment, groundwater, and soils with toxic and hazardous chemicals is a potential health and social-economic problem among all industrialized nations today. There are over 1,250 abandoned sites with toxic and hazardous chemicals and wastes in the National Priority List, and the number may potentially increase to over 32,000 in 1996. There are millions of underground fuel storage tanks in the United States and a significant number of these tanks are leaking. Despite the regulatory efforts to decrease the release of toxic chemicals to the environment, significant environmental contamination has occurred. At the current rate of Superfund site clean-up and the removal of leaking underground storage tanks, the environmental contamination problems will be with us for a long time.

In Rhode Island along, there are 12 Superfund sites eligible for federal clean-up, 82 hazardous waste sites throughout the state requiring public clean-up, and another 220 hazardous privately owned sites. Most of these sites need clean-up to reduce their long term effects on groundwater pollution. With federal and state mandates to clean up these sites, soil contamination has to be eliminated so that a contaminated site can be restored to its clean state. A restored site can have significant economic value, e.g., the former Kettle Point Petroleum Products Terminal site will be decontaminated and re-developed into a condominium development project. Before the site can be re-developed, soil total petroleum hydrocarbon up to 53,000 ppm and BTEX (benzene-toluene-ethyl benzene-xylene) up to 1,690 ppm need to be reduced to 1 ppm BTEX. This can be accomplished by using various chemical, physical, or biological techniques which emphasize the detoxification and destruction of the contaminants rather than the conventional approach of disposal. The selection of technology alternatives depends on the applicability and economics of the technology for the clean-up of a specific site. Land farming is a category of enhanced onsite bioremediation which reduces the time required for the clean-up of a contaminated site compared to other physical, chemical, and biological alternatives.

Land farming requires the excavation of the contaminated soil and put it on piles. The piles are turned over periodically for mixing and moisture control. Soil pH adjustment and nutrient addition may be required to enhance the biological activity. Depending on the nature of the contaminants and the operational control of the system, the site can be cleaned up in several weeks to months.

Methodology

This project investigate various techniques of land farming for soil decontamination of soils polluted with petroleum products, e.g., fuel oil, gasoline, kerosene, and related products. Land farming is a form of onsite bioremediation that relies on aerobic microbial metabolism for the oxidation of the petroleum products. The factors governing the design, operation, and success of land farming technology include biodegradability of the contaminants, use of indigenous or imported bacterial consortium, control of moisture, pH, nutrient supplements, control of fugitive VOC (volatile organic carbon) emissions, and labor requirement. Usually these factors are evaluated in the laboratory and the applicability of the various design and operational factors are confirmed in the field using pilot-scale or full-scale investigation.

(1) Laboratory-scale Investigation

Contaminated soil samples and clean soil at or near the site (Aqua Tank Farm Site) of the former Naval Air Station at Quonset Point, Rhode Island, were obtained. Laboratory-scale optimization of conditions for land farming can be accomplished initially using static microcosms. The information obtained from these microcosms was then used to developed optimal conditions for field-scale implementation of the various land farming techniques.

The indigenous micoflora of the contaminated soil was used to accomplish the aerobic biodegradation of the hydrocarbons. The site was contaminated by jet fuel JP-5 and aviation fuel. After the soil nitrogen and phosphate analysis, it was determined that the soil was poor and nutrients had to be added. Nutrients in the form of a fertilizer with N to P ratio of approximately 5 times were added to give an organic carbon/N/P ratio of about 100/15/3. Most samples did not require pH adjustment as the measured pH was within a range of 5.8 to 6.8. Outside of this range, lime was added to the soil sample(s) to adjust the pH to near neutral. The following non-ionic detergents were added to groups of soil samples: Dawn, holding 25% by weight of surfactant, Sunlight, holding 15% surfactant, Tide, holding 25% of surfactant, and BioSolve, holding 15% of surfactant. The amount of surfactant in the soil was controlled to 1.0, 1.5, 2.0, 2.5, and 3.0% of soil weight. All soil samples were moisture adjusted to about 60% of the soil water holding capacity to start the experiment. Mercury chloride was added to the negative control sample in an amount of 1,500 mg/kg of dry contaminated soil. Positive control samples consisted of uncontaminated soil adjacent to the site subject to the same treatment as contaminated soils. The samples were put into sealed glass serum vials as static microcosms and incubated at room temperature. Rates of microbial respiration were determined by measuring changes in carbon dioxide concentration in the head space of the vials as a function of time. The laboratory-scale study lasted 5 weeks and was terminated when the CO_2 generation rate diminished and stabilized.

(2) Field-scale Investigation

A repeated effort to carry out a field-scale study on the same site from which soil samples were obtained for the laboratory study failed because the agencies that had authority over the site including Army Corps of Engineers, State Property Office, State Airport Authority, and the State Department of Economic Development could not agreed on the term(s) of allowing such study to be conducted. Consequently a commercial site was selected for the field study. The site has soils contaminated with jet fuel and other fuels including heating oil. Two windrow soil piles each approximately 18-ft by 4-ft at the base and 3-ft high were constructed. One soil pile served as the control while the other pile received nutrients and moisture control. Gypsum block probes connected to a conductivity meter were used for field moisture monitoring. A third pile, 12-ft by 12-ft and 1-ft high simulating area farming was constructed. No soil turn-over was attempted for this pile. This pile also was divided into two halves, with one-half for control and the other half with additions of nutrients, and moisture control. No surfactant was added to all piles. Spatial and temporal samples were taken once a week from all piles taken at designated locations to determine the total petroleum hydrocarbon degradation. Total petroleum hydrocarbon of soil samples were analyzed according to the EPA Method 8015A using carbon disulfide as the extraction solvent. A Shimadzu gas chromatograph, 14A series, with a flame ionization Detector, a CR501 Chromatopac integrator, an AOC-17 auto injector, and a 30m x 0.45 ID DB-TPH capillary column were used for TPH analysis.

Results

In the laboratory-scale study, the CO_2 generation was measured weekly in each of the microcosms. Four samples of contaminated soil were taken from the site for the laboratory study, designated as 1A, 1B, 2A, and 3A. The gas generation expressed in terms of mg of CO_2 per g of soil for each microcosm was recorded with result of the entire experiment shown in Appendix A.

The gas generation for each microcosm was compared with a similar microcosm with different surfactant and different surfactant concentration and with the control. The positive control microcosm, i.e., uncontaminated soil with the same moisture , pH, and nutrient control but without detergent showed strong bacteria action. Within the range of surfactants use in the experiment, from 1.0 to 3.0% of the soil weight, it was found that the surfactant inhibited the bacterial action.

For the microcosms with contaminated soils, the surfactant in general enhanced the biodegradation of the hydrocarbon. However Tide appeared to have too much disinfectant in the detergent such that the bacterial action was significantly inhibited. For the other three detergents, surfactant 1.0 to 1.5% appeared to be the optimal concentration for enhancing hydrocarbon biodegradation in soil. At higher concentration, the detergent

inhibited the bacterial action. Among these three detergents, Sunlight, Dawn, and BioSolve, BioSolve appeared to be slightly better.

Because of the much delay in securing a site for the field study, only 50 days were available for data collection after the soil piles were constructed. The windrow soil piles were turned over by hand regularly every two weeks. The one pile with moisture control needed water addition only once and the moisture after water addition was found actually slightly over corrected. The TPH removal for the control pile was from 3,130 ppm to 290 ppm in 49 treatment day or 90.7 percent while the removal for the moisture control pile was from 3,200 ppm to 325 ppm or 89.8 percent. The moisture content of 6.5% of soil weight plus or minus 0.6% did not seem to have any effect on the rate of the removal.

The third soil pile simulating an area method of farming showed a slower removal rate. In 45 treatment days (4 days less than the windrow soil piles), the TPH removal was from 2980 ppm down to 340 ppm or 88.6%. No moisture was added to either side of the pile. It is difficult to draw a conclusion which landfarming method, windrow soil piling or area soil piling, was better for a study of a short duration. However the result did indicate significant removal in either case. It appears entirely possible that the site can be cleaned up in a duration of several months.

APPENDIX A

Carbon Dioxide Generation from Soil Microcosms

QPD PROJECT SAMPLE CO2 PRODUCTION - WEEKLY READINGS AND TOTALS (mg/g)

Sample	Description	WT. (g)	Headspace (ml)	WEEK #1	TOTAL	WEEK #2	TOTAL	WEEK #3	TOTAL	WEEK #4	TOTAL	WEEK #5	TOTAL
	3A - Control												
1		4.78	17.90	*133.36*		2.63		2.12		2.52		0.23	
2		4.56	18.00	*150.69*		2.75		2.24		2.90		0.38	
3		4.03	18.23	4.16	**4.16**	2.93	**7.10**	2.65	**9.75**	1.89	**11.64**	0.19	**11.83**
	3A - Neg Control												
4		4.22	18.15	0.16	**0.16**	0.72	**0.87**	0.89	**1.76**	1.70	**3.46**	0.30	**3.76**
5		4.61	17.97	0.06	**0.06**	0.33	**0.38**	0.59	**0.97**	0.71	**1.68**	0.23	**1.91**
6		5.39	17.63	0.93	**0.93**	2.04	**2.97**	1.98	**4.95**	2.82	**7.77**	0.57	**8.34**
	1A - Control												
7		5.16	17.72	2.48	**2.48**	1.52	**4.00**	1.55	**5.55**	1.03	**6.58**	0.21	**6.79**
8		5.18	17.71	2.91	**2.91**	2.23	**5.13**	1.90	**7.03**	2.64	**9.67**	0.44	**10.11**
9		4.44	18.03	3.49	**3.49**	2.01	**5.49**	2.07	**7.56**	1.00	**8.56**	0.13	**8.69**
	1A - Neg Control												
10		5.23	17.68	0.09	**0.09**	0.04	**0.12**	0.03	**0.15**	0.00	**0.15**	0.00	**0.15**
11		5.59	17.52	0.07	**0.07**	0.03	**0.10**	0.02	**0.12**	0.00	**0.12**	0.00	**0.13**
12		5.84	17.41	0.21	**0.21**	0.44	**0.65**	0.45	**1.10**	0.56	**1.66**	0.11	**1.77**
	1B - Control												
13		4.53	18.11	3.25	**3.25**	1.78	**5.03**	1.81	**6.84**	1.96	**8.79**	0.16	**8.95**
14		5.2	17.83	2.97	**2.97**	1.63	**4.60**	1.73	**6.32**	1.99	**8.31**	0.41	**8.73**
15		4.87	17.97	3.42	**3.42**	2.33	**5.76**	2.09	**7.84**	1.13	**8.97**	0.14	**9.11**
	1B - Neg Control												
16		4.91	17.95	0.05	**0.05**	0.02	**0.07**	0.02	**0.08**	0.00	**0.08**	0.00	**0.09**
17		5.14	17.85	0.01	**0.01**	0.00	**0.01**	0.00	**0.01**	0.00	**0.01**	0.01	**0.02**
18		5.09	17.87	0.05	**0.05**	0.02	**0.08**	0.02	**0.09**	0.00	**0.09**	0.00	**0.10**
	2A - Control												
19		4.33	18.15	2.75	**2.75**	1.88	**4.63**	1.55	**6.18**	1.39	**7.57**	0.21	**7.78**
20		5.4	17.69	2.77	**2.77**	2.61	**5.38**	2.01	**7.39**	2.33	**9.72**	0.47	**10.20**
21		5.29	17.74	2.95	**2.95**	1.94	**4.89**	1.84	**6.73**	2.40	**9.13**	0.51	**9.64**
	2A - Neg Control												
22		5.68	17.57	0.03	**0.03**	0.02	**0.05**	0.02	**0.07**	0.00	**0.07**	0.00	**0.07**
23		5.34	17.72	0.01	**0.01**	0.00	**0.01**	0.00	**0.01**	0.00	**0.01**	0.00	**0.01**
24		5.72	17.55	0.03	**0.03**	0.02	**0.05**	0.02	**0.07**	0.00	**0.07**	0.00	**0.07**

– 1st week's reading hurts chance to calculate total CO_2 for samples #1 and #2

Sample	Description	WT. (g)	Headspace (ml)	WEEK #1	TOTAL	WEEK #2	TOTAL	WEEK #3	TOTAL	WEEK #4	TOTAL	WEEK #5	TOTAL
	Sunlight												
	1A												
25	3	4.31	17.92	1.06	**1.06**	1.56	**2.63**	1.94	**4.57**	0.81	**5.37**	0.14	**5.51**
26	3	4.68	17.74	1.09	**1.09**	2.12	**3.21**	1.85	**5.05**	2.06	**7.11**	0.34	**7.45**
27	2.5	4.57	17.82	0.98	**0.98**	1.71	**2.69**	1.97	**4.66**	1.33	**5.99**	0.28	**6.27**
28	2.5	5.39	17.43	1.60	**1.60**	1.78	**3.38**	1.59	**4.96**	1.08	**6.04**	0.45	**6.49**
29	2	4.57	17.85	2.03	**2.03**	1.51	**3.54**	1.93	**5.47**	0.85	**6.32**	0.09	**6.41**
30	2	5.31	17.51	1.63	**1.63**	1.90	**3.52**	1.83	**5.35**	2.11	**7.47**	0.47	**7.93**
31	1.5	4.84	17.76	1.85	**1.85**	1.51	**3.36**	1.78	**5.14**	2.57	**7.70**	0.45	**8.15**
32	1.5	4.98	17.70	1.85	**1.85**	2.12	**3.97**	1.99	**5.96**	2.63	**8.60**	0.47	**9.07**
33	1	4.89	17.77	2.09	**2.09**	2.07	**4.16**	1.91	**6.07**	2.57	**8.64**	0.31	**8.95**
34	1	4.96	17.74	1.90	**1.90**	1.79	**3.69**	1.73	**5.41**	1.88	**7.29**	0.45	**7.74**
	1B												
35	3	4.16	18.10	1.99	**1.99**	1.91	**3.90**	2.23	**6.13**	1.01	**7.13**	0.20	**7.34**
36	3	4.69	17.85	1.94	**1.94**	1.43	**3.37**	2.06	**5.43**	2.48	**7.91**	0.46	**8.38**
37	2.5	4.99	17.75	1.23	**1.23**	1.04	**2.27**	1.64	**3.91**	1.64	**5.55**	0.18	**5.73**
38	2.5	4.99	17.75	1.97	**1.97**	1.31	**3.29**	1.91	**5.20**	1.98	**7.18**	0.16	**7.34**
39	2	4.95	17.80	1.96	**1.96**	1.34	**3.30**	1.85	**5.15**	1.33	**6.48**	0.23	**6.71**
40	2	4.97	17.79	2.00	**2.00**	1.40	**3.41**	2.00	**5.41**	2.72	**8.12**	0.20	**8.32**
41	1.5	4.97	17.82	1.98	**1.98**	1.38	**3.36**	1.72	**5.07**	1.66	**6.74**	0.16	**6.89**
42	1.5	5	17.81	2.02	**2.02**	2.03	**4.05**	2.00	**6.04**	1.86	**7.90**	0.13	**8.03**
43	1	5.07	17.81	2.13	**2.13**	1.23	**3.35**	1.75	**5.10**	0.64	**5.74**	0.44	**6.19**
44	1	5.03	17.83	1.96	**1.96**	1.33	**3.29**	1.88	**5.16**	1.51	**6.67**	0.12	**6.80**
	2A												
45	3	4.65	17.83	0.81	**0.81**	1.41	**2.22**	1.42	**3.63**	0.57	**4.20**	0.36	**4.56**
46	3	4.5	17.90	0.13	**0.13**	0.42	**0.55**	0.54	**1.09**	0.86	**1.95**	0.16	**2.11**
47	2.5	4.98	17.70	1.05	**1.05**	1.51	**2.55**	1.65	**4.21**	2.11	**6.32**	0.27	**6.59**
48	2.5	5.06	17.74	0.52	**0.52**	1.11	**1.63**	1.16	**2.79**	1.83	**4.62**	0.31	**4.93**
49	2	4.9	17.80	0.53	**0.53**	0.99	**1.51**	0.94	**2.46**	1.30	**3.76**	0.21	**3.97**
50	2	4.84	17.71	0.87	**0.87**	1.10	**1.97**	1.00	**2.97**	1.38	**4.35**	0.20	**4.55**
51	1.5	5.04	17.78	0.79	**0.79**	1.25	**2.04**	1.19	**3.23**	0.92	**4.15**	0.24	**4.39**
52	1.5	4.96	17.78	0.99	**0.99**	1.31	**2.30**	1.20	**3.50**	0.63	**4.14**	0.25	**4.38**
53	1	4.96	17.81	1.07	**1.07**	1.34	**2.41**	0.98	**3.39**	1.08	**4.47**	0.16	**4.63**
54	1	4.82	17.88	1.32	**1.32**	1.67	**2.99**	1.45	**4.44**	0.75	**5.19**	0.22	**5.40**
	3A												
55	3	4.94	17.63	1.00	**1.00**	1.45	**2.45**	1.87	**4.32**	0.92	**5.25**	0.27	**5.52**
56	3	4.43	17.88	0.83	**0.83**	2.01	**2.83**	2.08	**4.92**	2.54	**7.46**	0.33	**7.79**
57	2.5	5	17.64	1.48	**1.48**	2.00	**3.49**	1.74	**5.22**	2.04	**7.26**	0.28	**7.55**
58	2.5	4.88	17.69	1.13	**1.13**	2.04	**3.17**	1.88	**5.05**	2.43	**7.48**	0.30	**7.78**
59	2	4.86	17.74	1.82	**1.82**	1.34	**3.16**	1.70	**4.86**	2.56	**7.42**	0.33	**7.75**
60	2	4.81	17.76	1.56	**1.56**	1.99	**3.55**	1.62	**5.17**	2.16	**7.33**	0.36	**7.69**
61	1.5	5.02	17.69	2.03	**2.03**	1.45	**3.48**	1.71	**5.18**	2.02	**7.20**	0.44	**7.64**
62	1.5	4.84	17.78	2.18	**2.18**	1.99	**4.17**	1.80	**5.97**	1.92	**7.89**	0.49	**8.38**
63	1	4.75	17.85	2.24	**2.24**	1.55	**3.79**	1.99	**5.79**	0.72	**6.51**	0.40	**6.91**
64	1	4.83	17.81	2.14	**2.14**	1.52	**3.66**	1.82	**5.47**	1.92	**7.39**	0.39	**7.78**

Sample	Description	WT. (g)	Headspace (ml)	WEEK #1	TOTAL	WEEK #2	TOTAL	WEEK #3	TOTAL	WEEK #4	TOTAL	WEEK #5	TOTAL
	DAWN												
	1A												
65	3	4.82	17.49	1.20	**1.20**	1.91	**3.12**	1.71	**4.82**	1.83	**6.65**	0.36	**7.01**
66	3	5.11	17.34	1.33	**1.33**	1.82	**3.15**	1.77	**4.93**	2.26	**7.19**	0.38	**7.57**
67	2.5	4.99	17.46	1.62	**1.62**	1.32	**2.95**	1.63	**4.58**	1.25	**5.83**	0.38	**6.21**
68	2.5	4.91	17.50	1.63	**1.63**	1.35	**2.99**	1.84	**4.82**	2.62	**7.45**	0.51	**7.95**
69	2	5.12	17.47	1.52	**1.52**	1.83	**3.35**	1.83	**5.18**	2.66	**7.84**	0.49	**8.33**
70	2	4.98	17.53	1.59	**1.59**	1.56	**3.15**	1.68	**4.84**	1.11	**5.94**	0.39	**6.33**
71	1.5	4.92	17.63	1.74	**1.74**	1.33	**3.07**	1.89	**4.96**	2.10	**7.06**	0.44	**7.49**
72	1.5	4.96	17.61	1.66	**1.66**	1.91	**3.57**	1.69	**5.26**	2.21	**7.47**	0.47	**7.94**
73	1	4.9	17.70	1.73	**1.73**	1.37	**3.10**	1.96	**5.06**	2.24	**7.31**	0.47	**7.77**
74	1	4.91	17.70	1.84	**1.84**	1.95	**3.79**	1.98	**5.77**	0.93	**6.69**	0.46	**7.16**
	1B												
75	3	5.53	17.26	0.36	**0.36**	0.77	**1.12**	1.36	**2.48**	0.50	**2.99**	0.04	**3.03**
76	3	4.92	17.56	0.55	**0.55**	1.41	**1.96**	1.62	**3.58**	1.84	**5.42**	0.34	**5.76**
77	2.5	4.97	17.60	0.92	**0.92**	1.74	**2.66**	1.67	**4.33**	0.84	**5.17**	0.41	**5.58**
78	2.5	4.96	17.60	1.53	**1.53**	1.31	**2.83**	1.61	**4.44**	2.74	**7.19**	0.51	**7.69**
79	2	4.98	17.66	1.66	**1.66**	0.87	**2.54**	1.66	**4.19**	0.71	**4.91**	0.48	**5.39**
80	2	5.03	17.64	1.66	**1.66**	1.42	**3.08**	1.81	**4.89**	2.66	**7.55**	0.51	**8.06**
81	1.5	4.87	17.77	1.75	**1.75**	1.40	**3.15**	1.89	**5.04**	0.60	**5.64**	0.39	**6.03**
82	1.5	5.27	17.59	1.56	**1.56**	1.25	**2.81**	1.71	**4.51**	0.92	**5.43**	0.40	**5.84**
83	1	4.73	17.90	2.51	**2.51**	2.49	**5.00**	2.05	**7.05**	1.56	**8.61**	0.45	**9.06**
84	1	5	17.78	1.74	**1.74**	2.04	**3.77**	1.88	**5.66**	1.52	**7.17**	0.34	**7.51**
	2A												
85	3	4.38	17.78	0.19	**0.19**	0.61	**0.80**	1.24	**2.03**	1.40	**3.43**	0.20	**3.62**
86	3	4.38	17.78	0.07	**0.07**	0.60	**0.67**	1.08	**1.75**	0.40	**2.15**	0.02	**2.17**
87	2.5	4.85	17.61	0.72	**0.72**	1.72	**2.44**	1.81	**4.25**	0.55	**4.80**	0.38	**5.19**
88	2.5	5.08	17.50	1.15	**1.15**	1.35	**2.50**	1.59	**4.10**	0.56	**4.65**	0.27	**4.92**
89	2	4.82	17.69	1.74	**1.74**	1.30	**3.04**	1.78	**4.82**	0.00	**4.82**	0.00	**4.82**
90	2	4.85	17.67	1.74	**1.74**	1.72	**3.46**	1.80	**5.26**	2.25	**7.51**	0.35	**7.86**
91	1.5	4.95	17.69	1.78	**1.78**	2.09	**3.87**	1.75	**5.62**	2.19	**7.81**	0.34	**8.15**
92	1.5	4.81	17.75	1.89	**1.89**	1.24	**3.12**	1.81	**4.93**	2.31	**7.24**	0.42	**7.66**
93	1	4.89	17.78	1.95	**1.95**	1.66	**3.62**	1.98	**5.59**	2.53	**8.12**	0.43	**8.56**
94	1	4.99	17.74	1.97	**1.97**	2.09	**4.06**	1.85	**5.92**	2.30	**8.22**	0.41	**8.63**
	3A												
95	3	4.52	17.66	0.58	**0.58**	1.47	**2.05**	1.74	**3.79**	1.11	**4.90**	0.35	**5.25**
96	3	4.56	17.64	0.47	**0.47**	1.45	**1.91**	1.59	**3.50**	2.23	**5.73**	0.42	**6.15**
97	2.5	4.86	17.55	1.22	**1.22**	2.00	**3.22**	1.89	**5.11**	2.56	**7.67**	0.44	**8.11**
98	2.5	5.02	17.47	0.99	**0.99**	1.58	**2.57**	1.81	**4.37**	2.03	**6.40**	0.35	**6.75**
99	2	4.99	17.55	1.94	**1.94**	1.98	**3.92**	1.93	**5.85**	2.61	**8.46**	0.48	**8.94**
100	2	4.95	17.57	1.84	**1.84**	1.91	**3.75**	1.87	**5.62**	2.49	**8.11**	0.47	**8.58**
101	1.5	4.97	17.62	1.83	**1.83**	0.00	**1.83**	1.77	**3.60**	0.97	**4.57**	0.44	**5.01**
102	1.5	4.9	17.65	1.90	**1.90**	1.44	**3.34**	1.84	**5.18**	0.98	**6.15**	0.28	**6.43**
103	1	4.86	17.74	2.01	**2.01**	2.05	**4.07**	1.82	**5.89**	2.55	**8.44**	0.42	**8.86**
104	1	4.99	17.68	2.00	**2.00**	2.23	**4.23**	1.69	**5.93**	1.91	**7.83**	0.39	**8.22**

Sample	Description	WT. (g)	Headspace (ml)	WEEK #1	TOTAL	WEEK #2	TOTAL	WEEK #3	TOTAL	WEEK #4	TOTAL	WEEK #5	TOTAL
	TIDE												
	1A												
105	3	5.13	17.42	0.10	**0.10**	0.07	**0.16**	0.05	**0.21**	0.04	**0.25**	0.01	**0.27**
106	3	5.29	17.34	0.09	**0.09**	0.06	**0.15**	0.04	**0.19**	0.04	**0.23**	0.01	**0.24**
107	2.5	4.98	17.55	0.10	**0.10**	0.06	**0.16**	0.05	**0.21**	0.05	**0.26**	0.02	**0.28**
108	2.5	5.01	17.53	0.09	**0.09**	0.05	**0.14**	0.06	**0.20**	0.09	**0.28**	0.03	**0.31**
109	2	4.92	17.62	0.08	**0.08**	0.05	**0.13**	0.05	**0.18**	0.05	**0.23**	0.01	**0.24**
110	2	4.97	17.60	0.10	**0.10**	0.06	**0.16**	0.05	**0.21**	0.05	**0.26**	0.01	**0.27**
111	1.5	5.02	17.63	0.13	**0.13**	0.30	**0.43**	0.97	**1.40**	0.53	**1.93**	0.34	**2.27**
112	1.5	4.99	17.64	0.74	**0.74**	0.70	**1.43**	1.50	**2.93**	0.66	**3.59**	0.36	**3.95**
113	1	4.97	17.70	1.74	**1.74**	0.98	**2.71**	1.37	**4.08**	1.71	**5.80**	0.38	**6.18**
114	1	4.88	17.74	1.68	**1.68**	0.27	**1.95**	0.25	**2.20**	0.00	**2.20**	0.00	**2.20**
	1B												
115	3	5	17.61	0.06	**0.06**	0.03	**0.09**	0.03	**0.12**	0.02	**0.14**	0.01	**0.15**
116	3	5.58	17.33	0.06	**0.06**	0.04	**0.09**	0.03	**0.12**	0.02	**0.15**	0.01	**0.16**
117	2.5	5.01	17.66	0.07	**0.07**	0.05	**0.12**	0.04	**0.17**	0.03	**0.20**	0.01	**0.21**
118	2.5	5.01	17.66	0.07	**0.07**	0.04	**0.11**	0.04	**0.15**	0.03	**0.17**	0.01	**0.18**
119	2	5	17.71	0.08	**0.08**	0.09	**0.17**	0.26	**0.43**	0.87	**1.30**	0.43	**1.73**
120	2	4.95	17.73	0.19	**0.19**	0.82	**1.01**	1.50	**2.51**	0.30	**2.81**	0.21	**3.02**
121	1.5	4.95	17.78	1.10	**1.10**	2.09	**3.19**	1.84	**5.03**	2.73	**7.76**	0.48	**8.24**
122	1.5	4.96	17.78	0.06	**0.06**	0.04	**0.10**	0.03	**0.14**	0.05	**0.19**	0.01	**0.20**
123	1	5.01	17.81	1.10	**1.10**	1.06	**2.16**	1.35	**3.51**	1.38	**4.89**	0.13	**5.02**
124	1	5	17.81	1.13	**1.13**	0.97	**2.10**	1.02	**3.13**	0.57	**3.70**	0.24	**3.94**
	2A												
125	3	4.64	17.74	0.04	**0.04**	0.04	**0.08**	0.03	**0.11**	0.03	**0.14**	0.01	**0.15**
126	3	5.15	17.49	0.04	**0.04**	0.04	**0.08**	0.04	**0.12**	0.04	**0.16**	0.01	**0.16**
127	2.5	4.78	17.72	0.04	**0.04**	0.04	**0.08**	0.04	**0.12**	0.04	**0.16**	0.01	**0.17**
128	2.5	5.1	17.56	0.05	**0.05**	0.04	**0.09**	0.04	**0.13**	0.04	**0.17**	0.01	**0.18**
129	2	4.69	17.81	0.05	**0.05**	0.04	**0.09**	0.04	**0.12**	0.04	**0.16**	0.01	**0.17**
130	2	4.87	17.72	0.04	**0.04**	0.04	**0.08**	0.03	**0.12**	0.04	**0.15**	0.01	**0.16**
131	1.5	5.01	17.71	0.05	**0.05**	0.04	**0.10**	0.04	**0.14**	0.04	**0.18**	0.01	**0.19**
132	1.5	4.92	17.75	0.04	**0.04**	0.04	**0.08**	0.04	**0.12**	0.04	**0.16**	0.01	**0.17**
133	1	4.79	17.86	0.09	**0.09**	0.09	**0.18**	0.09	**0.26**	0.14	**0.40**	0.05	**0.45**
134	1	5.03	17.75	0.06	**0.06**	0.05	**0.10**	0.04	**0.14**	0.04	**0.18**	0.01	**0.19**
	3A												
135	3	5.08	17.46	0.09	**0.09**	0.07	**0.16**	0.07	**0.23**	0.07	**0.30**	0.02	**0.31**
136	3	4.71	17.65	0.04	**0.04**	0.04	**0.08**	0.04	**0.11**	0.04	**0.15**	0.01	**0.16**
137	2.5	4.91	17.60	0.05	**0.05**	0.04	**0.09**	0.04	**0.13**	0.04	**0.17**	0.01	**0.19**
138	2.5	4.83	17.64	0.04	**0.04**	0.04	**0.08**	0.04	**0.12**	0.04	**0.16**	0.01	**0.17**
139	2	4.87	17.66	0.07	**0.07**	0.12	**0.19**	0.15	**0.34**	0.22	**0.56**	0.07	**0.63**
140	2	4.89	17.66	0.05	**0.05**	0.05	**0.10**	0.05	**0.15**	0.05	**0.20**	0.01	**0.21**
141	1.5	4.85	17.72	0.35	**0.35**	1.34	**1.69**	1.57	**3.26**	2.35	**5.61**	0.40	**6.01**
142	1.5	5.07	17.62	0.13	**0.13**	0.05	**0.19**	0.06	**0.25**	0.10	**0.35**	0.02	**0.37**
143	1	4.71	17.84	0.14	**0.14**	0.79	**0.92**	1.72	**2.65**	2.47	**5.11**	0.54	**5.66**
144	1	4.96	17.72	0.31	**0.31**	1.22	**1.54**	1.72	**3.26**	2.34	**5.60**	0.41	**6.01**

Sample	Description	WT. (g)	Headspace (ml)	WEEK #1	TOTAL	WEEK #2	TOTAL	WEEK #3	TOTAL	WEEK #4	TOTAL	WEEK #5	TOTAL
	Biosolve												
	1A												
145	3	5.97	17.00	1.10	**1.10**	0.98	**2.08**	1.31	**3.39**	2.01	**5.40**	0.45	**5.85**
146	3	4.89	17.55	1.46	**1.46**	1.44	**2.90**	1.52	**4.42**	1.94	**6.36**	0.50	**6.86**
147	2.5	4.89	17.59	1.43	**1.43**	1.16	**2.58**	1.56	**4.14**	2.00	**6.15**	0.45	**6.60**
148	2.5	4.9	17.59	1.36	**1.36**	1.14	**2.50**	1.69	**4.18**	2.48	**6.67**	0.43	**7.09**
149	2	4.87	17.65	1.49	**1.49**	1.59	**3.08**	1.87	**4.94**	2.49	**7.44**	0.52	**7.95**
150	2	4.93	17.62	1.43	**1.43**	1.89	**3.33**	1.81	**5.14**	2.51	**7.65**	0.54	**8.19**
151	1.5	4.9	17.69	1.65	**1.65**	1.09	**2.74**	1.84	**4.58**	2.08	**6.66**	0.45	**7.10**
152	1.5	4.88	17.70	1.59	**1.59**	2.15	**3.74**	2.08	**5.82**	2.57	**8.39**	0.56	**8.95**
153	1	4.83	17.77	1.97	**1.97**	1.84	**3.81**	2.14	**5.95**	2.13	**8.08**	0.44	**8.52**
154	1	4.91	17.73	1.61	**1.61**	2.03	**3.65**	1.88	**5.53**	2.44	**7.97**	0.49	**8.46**
	1B												
155	3	4.92	17.65	0.08	**0.08**	1.25	**1.33**	1.62	**2.95**	2.02	**4.97**	0.44	**5.42**
156	3	4.75	17.74	0.27	**0.27**	2.01	**2.28**	1.95	**4.23**	2.62	**6.85**	0.53	**7.38**
157	2.5	4.99	17.67	1.35	**1.35**	1.07	**2.42**	1.34	**3.76**	2.47	**6.23**	0.42	**6.65**
158	2.5	4.9	17.71	1.15	**1.15**	2.07	**3.22**	1.76	**4.97**	2.54	**7.52**	0.56	**8.08**
159	2	4.98	17.72	1.42	**1.42**	2.01	**3.43**	1.99	**5.43**	2.59	**8.02**	0.58	**8.59**
160	2	5.04	17.70	1.38	**1.38**	1.30	**2.68**	1.84	**4.52**	2.44	**6.97**	0.50	**7.47**
161	1.5	4.97	17.78	1.64	**1.64**	1.99	**3.63**	1.90	**5.53**	2.58	**8.11**	0.55	**8.66**
162	1.5	5.02	17.75	1.58	**1.58**	1.52	**3.10**	1.82	**4.92**	2.57	**7.49**	0.54	**8.03**
163	1	4.83	17.89	1.82	**1.82**	0.94	**2.76**	1.78	**4.54**	1.87	**6.41**	0.34	**6.76**
164	1	4.8	17.90	1.86	**1.86**	1.35	**3.21**	1.96	**5.17**	2.59	**7.76**	0.54	**8.30**
	2A												
165	3	5.01	17.56	0.04	**0.04**	0.70	**0.73**	1.55	**2.28**	2.34	**4.63**	0.48	**5.11**
166	3	4.73	17.70	0.15	**0.15**	1.88	**2.03**	1.78	**3.81**	2.59	**6.40**	0.53	**6.93**
167	2.5	4.71	17.75	0.63	**0.63**	1.04	**1.67**	1.86	**3.54**	2.21	**5.75**	0.32	**6.07**
168	2.5	4.88	17.67	0.27	**0.27**	1.16	**1.43**	1.87	**3.30**	2.53	**5.83**	0.53	**6.36**
169	2	4.59	17.86	1.71	**1.71**	1.35	**3.06**	1.75	**4.82**	2.13	**6.95**	0.44	**7.39**
170	2	4.83	17.74	1.65	**1.65**	1.95	**3.60**	1.86	**5.47**	2.56	**8.02**	0.58	**8.60**
171	1.5	4.86	17.78	1.96	**1.96**	2.05	**4.01**	1.97	**5.98**	2.56	**8.55**	0.55	**9.09**
172	1.5	4.62	17.89	1.91	**1.91**	1.79	**3.71**	2.15	**5.86**	2.65	**8.51**	0.54	**9.05**
173	1	4.83	17.84	1.78	**1.78**	1.50	**3.28**	1.86	**5.14**	2.72	**7.86**	0.57	**8.43**
174	1	4.97	17.78	1.94	**1.94**	2.26	**4.21**	1.95	**6.16**	2.31	**8.47**	0.47	**8.94**
	3A												
175	3	4.64	17.69	0.06	**0.06**	1.67	**1.73**	1.71	**3.44**	2.06	**5.50**	0.49	**5.99**
176	3	5.04	17.49	0.07	**0.07**	1.06	**1.13**	1.54	**2.66**	1.89	**4.55**	0.39	**4.94**
177	2.5	4.95	17.58	1.41	**1.41**	1.39	**2.80**	1.73	**4.53**	2.54	**7.07**	0.49	**7.56**
178	2.5	4.93	17.59	1.48	**1.48**	1.87	**3.35**	1.69	**5.04**	2.05	**7.09**	0.41	**7.50**
179	2	4.91	17.65	1.59	**1.59**	1.28	**2.87**	1.60	**4.47**	2.48	**6.95**	0.53	**7.48**
180	2	4.96	17.63	1.50	**1.50**	1.30	**2.80**	1.52	**4.31**	2.51	**6.82**	0.54	**7.36**
181	1.5	4.9	17.70	1.68	**1.68**	1.92	**3.60**	2.03	**5.63**	2.62	**8.26**	0.57	**8.83**
182	1.5	4.94	17.68	1.60	**1.60**	1.84	**3.45**	1.68	**5.13**	2.33	**7.45**	0.52	**7.97**
183	1	5.01	17.70	1.84	**1.84**	1.80	**3.64**	2.04	**5.69**	2.44	**8.13**	0.43	**8.56**
184	1	4.92	17.74	1.80	**1.80**	2.33	**4.13**	2.16	**6.30**	2.73	**9.02**	0.59	**9.61**