

TEXT SEARCHABLE DOCUMENT

Data Evaluation Report of Surface Water Monitoring Study

PMRA Submission Number {.....}

EPA MRID Number 46733903

Test material: Fipronil

IUPAC name: 5-amino-1-(2,6-dichloro-α,α,α-trifluoro-p-tolyl)-4-trifluoromethylsulfinylpyrazole-3carbonitrile

CAS name: 5-amino-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-[(trifluoromethyl)sulfinyl]-1Hpyrazole-3-carbonitrile

Primary Reviewer: James Hetrick, Ph.D. EPA

Signature: Date:

Date:

Secondary Reviewer: Thuy Nguyen EPA

mer (1: Hethick 4/30/08 Signature

CITATION: Wyatt, Daryl R. 2005. Fipronil Esturaine Monitoring Study following an Application of Chipco Topchoice® to a Golf Course at Gulf Breeze, Florida. Sponsored by BayerCrop Science, RTP, NC. Performed by Bayer Crop Science, Stillwell, KS and AgVise Laboratories, Northward,

ND. MRID 46733903.



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EXECUTIVE SUMMARY:

The fipronil water monitoring study (MRID 46733903) provides acceptable data on the runoff potential of fipronil and its degradation products (MB46136, MB46513, and MB 46950) and its impact into estuarine surface water in Gulf Breeze, FL. This study was submitted to fulfill a condition of registration regarding runoff concerns of fipronil residues from broadcast use of fipronil for control of fire ants. The registrant did not provide any concurrent biological monitoring of the aquatic environment to assess the impact of fipronil and its degradation products on aquatic invertebrates.

The runoff monitoring study was conducted on the 14th fairway of the East Course at Tiger Point Golf and Country Club in Gulf Breeze, FL. The catchment area of the site is approximately 1.5 acres. The slope of the site ranges from 3 to 5%. The site was selected because it drains into a brackish water estuary, which is hydraulically connected to the Santa Rosa Sound. The estuary is approximately 0.24 acre surface area with a maximum depth of 10 feet. Water samples were taken at three locations in the estuary where runoff is expected to influence the water quality. In 2003, duplicate grab samples of water were taken at pre-fipronil treatment, day 0, 1, 2, ~10 days, and after each natural rainfall event from 3 to 100 days post treatment, and at least 20 day intervals from 100 to 180 days post-treatment. In 2004, water samples were taken using an autosampler as well as grab samples. Autosamplers were designed to take water samples at the edge of the estuary (@ a 1 feet depth below the water surface). The peak concentration of fipronil residues was 0.510 µg/L for fipronil, 0.007 µg/L for MB46513, 0.013 µg/L for MB45950, and 0.026 µg/L for MB46136. MB45950 and MB46136 were detected in sediment samples. The peak concentration of fipronil residues in sediment samples was 0.059 µg/kg for MB45950 and 0.043 µg/kg for MB 46136.

I. MATERIALS AND METHODS

GUIDELINE FOLLOWED: The SETAC-Europe: Procedures for Assessing the Environmental Fate and Ecotoxicity of Pesticides (March 1995; pp. 1, 34) is not applicable.

COMPLIANCE:

This study was conducted in compliance with USEPA FIFRA Good Laboratory Practices (40 CFR Part 160), which are consistent with the OECD Principles of GLP (p. 3). Signed and dated GLP, Data Confidentiality, Quality Assurance, and Certificate of Authenticity statements were provided (pp. 2-3, 5-6).

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A. MATERIALS:

The study objective is to assess the runoff potential of fipronil and its degradation products into estuarine aquatic environments from turf use of Chipco Topchoice® for control of fire ants.

1. Study Description

The runoff monitoring study was conducted on the 14th fairway of the East Course at Tiger Point Golf and Country Club in Gulf Breeze, FL (**Figures B.1, and B.2, pp 63-64**). The catchment area of the site is approximately 1.5 acres. The slope of the site ranges from 3 to 5%. The site was selected because it drains into a brackish water estuary, which is hydraulically connected to the Santa Rosa Sound. The estuary is approximately 0.24 acre surface area with a maximum depth of 10 feet.

The soils in the catchment area are classified as a Leon sand (sandy, siliceous, thermic Aeric Haplaquods). Soil physicochemical properties for surface samples (0-6 inches) are shown in **Table E.1, pp 87**. The soil is classified in the A/D hydrologic group. The D soil classification indicates high runoff potential during periods with a high water table.

Water samples were taken at three locations (Points A, B, C) in the estuary where runoff is expected to influence the water quality (Reference Map). Estuary and irrigation water characterizations are shown in **Table E.2.**, **pp 88**.

In 2003, duplicate grab samples of water were taken at pre-fipronil treatment, day 0, 1, 2, \sim 10 days, and after each natural rainfall event from 3 to 100 days post treatment, and at least 20 day intervals from 100 to 180 days post-treatment.

In 2004, water samples were taken using an autosampler as well as grab samples. Autosamplers were designed to take water samples at the edge of the estuary (@ a 1 feet depth below the water surface) at sampling points A, B, and C. Duplicate samples were taken from the start of the runoff event and then every 4 hours until 44 hours after the runoff event. Initiation of sampling was triggered using a 600 weir equipped with transducer to monitor runoff water. (Reviewer Note: There was no attempt to measure runoff volumes from the test site). Manual control of the autosamplers was generally performed between 36 to 44 hours after the runoff event. Duplicate grab samples also were taken at the three sampling locations. Samples were taken after runoff events or at 7 to 10 day sample intervals. Water samples were placed on blue ice and then transported to laboratory for frozen storage. Samples stored frozen prior to chemical analysis.

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Four random sediment samples were taken at each sampling location. In 2003, sediment samples were taken immediately before treatment, 87 and 185 days post-treatment. In 2004, sediment samples were taken at two sampling locations immediately before treatment, and at 98 days post-treatment. The four random samples were composited and then a subsample was taken. Subsamples were placed on blue ice and then transported to laboratory for frozen storage. Samples stored frozen prior to chemical analysis.

A rain gauge was used to collect on-site precipitation. The on-site precipitation and irrigation accounted for 62.97 inches in 2003 (167% of the 30 year average) and 30.04 inches in 2004 (100% of the 30 year average) (**Tables D.1 and D.2., pp 73 to 85**). The tidal flux was measured using a flow meter connected to a pressure transducer.

2. Site Preparation and Maintenance

The test site had well maintained Bermudagrass turf. The site was amended with 12/12/12 (N/P/K) fertilizer at 275 lbs/A on February 26, 2004 and 200 lbs/A on April 8, 2004 and June 29, 2004. Pesticide applications on the site include metribuzin, prodiamine, pendimethalin, oxadiazon, MSMA, and MCB (**pp 62**).

3. Pesticide Application

The fipronil application area accounts for ~ 1.53 acres of well maintained golf fairway beside an estuary. Chipco Topchoice® insecticide was broadcast applied at 0.0109 lbs ai/A on May 20, 2003 and 0.0123 lbs ai/A on April 21, 2004. The label maximum application rate is 0.0125 lbs ai/A. Applications were made using calibrated drop spreaders. A 60 foot buffer was maintained around the estuary.

4. Analytical

A total of 264 water samples (105 in 2003 and 160 in 2004) were analyzed during this study. The sample analysis accounts for 64% of the duplicate samples in 2003 and 7.5% of the duplicate samples in 2004. Water samples were stored frozen for a maximum storage period of 342 days (**Tables F.1 and F.2, pp 90-96**). Reviewer Note: The registrant referenced storage stability data for a maximum 750 days (MRID MRID 46733902).

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Residues of fipronil in water samples were analyzed using a LC/MS/MS method entitled Insecticides, Fipronil: Method of Analysis for Possible Residues of Fipronil, MB46513, MB45950, and MB46136 in Water- Revisions 5 and 5 (August, 28, 2003). This method has method detection limit (MDL) of 0.004 μ g/L and limit of quantification (LOQ) of 0.010 μ g/L. [Reviewer Note: The method procedure requires filtration for cloudy extracts. The extracts are filtered through a nylon filtration disk after an acetonitrile extraction of surface water].

Procedural method verification in estuary water at concentrations of 10, 50, and 100 ng/L showed recoveries of 89.5% for fipronil (n=28), 93.5% for MB46513 (n=28), 93.8% for MB45950 (n=28), and 97.5% for MB46136 (n=28) (**Table IV**, **pp 36**). Method verification was conducted using estuary water at 0.010 μ g/L and 0.100 μ g/L. Average recoveries in estuary water were 86%± 10 for fipronil, 97%± 11 for MB46513, 102%± 15 for MB45950, and 98%± 11 for MB46136 (**Table I.1, pp 138**). Field spike fortifications were conducted in estuary water at the 0.10 μ g/L and 1.00 μ g/L. Recoveries ranged 77 to 86% for fipronil, 88 to 90% for MB46513, 86 to 90% for MB45950, and 99 to 103 % for MB46136 (**Table VI, pp 40**).

Residues of fipronil in sediment samples were analyzed using a LC/MS/MS method entitled Method of Analysis for Possible Residues of Fipronil, MB46513, MB45950, and MB46136 in Sediment (November 19, 2003). This method has method detection limit (MDL) of 0.030 μ g/kg and limit of quantification (LOQ) of 0.100 μ g/kg. Procedural recoveries from fortified sediment at 0.1 and 0.2 μ g/kg ranged from 80.5 to 85.5% for fipronil, 70.1 to 96.3% for MB46513, 93 to 113% for MB45950, and 95.4 to 112% for MB46136 (**Table VII, pp 41**).

B. REPORTED RESULTS

1. 2003 Fipronil Residues in Estuary Water

Fipronil residues were detected in estuary water samples (Figures 3, pp 39; Table V, pp 38). The peak concentrations of fipronil residues was $0.510 \ \mu g/L$ for fipronil at 2 days post treatment, $0.007 \ \mu g/L$ for MB 46513 at 47 days post treatment, $0.013 \ \mu g/L$ for MB 45950 at 41 days post treatment, and $0.026 \ \mu g/L$ for MB 46136 at 2 days postreatment. Fipronil was detected in all water samples from 2 to 47 days post treatment. Fipronil degradation products had more sporadic detections; MB46136 was the predominate fipronil degradate detected in surface water.

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2. 2004 Fipronil Residues in Estuary Water

There were no detectable fipronil residues in water except for a fipronil detection (0.037 μ g/L) in a 112 day posttreatment sample (**pp 40**).

3. Concentration of Fipronil Residues in Sediment

MB45950 and MB46136 were detected in sediment samples (**Table VIII, pp 42**). The peak concentration was $0.059 \ \mu g/kg$ for MB45950, and $0.043 \ \mu g/kg$ for MB46136.

B. REVIEWER COMMENTS

1. The fipronil water monitoring study (MRID 46733903) provides acceptable data on the runoff potential of fipronil and its degradation products (MB46136, MB46513, and MB 46950) and its impact on fipronil residue occurrence in estuarine surface water from use of Chipco Topchoice® on a turf in Gulf Breeze, FL. This study was submitted to fulfill a condition of registration regarding runoff concerns of fipronil residues from broadcast use of fipronil for control of fire ants. The registrant did not provide any concurrent biological monitoring of the aquatic environment to assess the impact of fipronil and its degradation products on aquatic invertebrates

2. The registrant referenced a storage stability study in a Texas runoff study (MRID 46733902). The Texas runoff study does not provide a detailed description of the storage stability study.

3. The fipronil applications were made in April and May. This application period coincides with highest precipitation months, which implies fipronil runoff and occurrence in surface water is expected to be high.















<u>Soil</u>

Soil samples for characterization were collected on 20 May 2003 from within the treated area. The samples were obtained from the top 6 inches of soil. They were shipped to AGVISE Laboratories on 29 May 2003 and received on 2 June 2003. The results are listed in Table E.1.

Parameter	Analytical Result
Sample ID	03RAFIX020-SC 0-6"
Percent Sand	96
Percent Silt	3
Percent Clay	. 1
USDA Texture	Sand
Bulk Density (gm/cc)	1.18
CEC (meq/100 g)	5.8
Percent Moisture (0 bar)	54.1
Percent Moisture (1/10 bar)	12.1
Percent Moisture (1/3 bar)	11.7
Percent Moisture (1.0 bar)	6.6
Percent Moisture (5.0 bar)	6.4
Percent Moisture (15 bar)	6.4
Percent Organic Matter	4.6
pH (1:1 soil:water)	6.5
Calcium (ppm)	574
Magnesium (ppm)	117
Sodium (ppm)	147
Potassium (ppm)	76
Hydrogen (ppm)	11

Table E.1: Soil Characterization Data



<u>Water</u>

Estuary water characterization sample was collected on 20 May 2003 from Sample Location A in the estuary. Irrigation water characterization sample was collected on 20 May 2003 from the irrigation pond east of the golf course shop building. They were shipped to AGVISE Laboratories on 29 May 2003 and received on 2 June 2003. The results are listed in Table E.2.

Parameter	Analytical Result		
Sample ID	03RAFIX020-WC (estuary water)	03RAFIX020-IWC (irrigation water)	
pH	7.7	7.4	
Sodium (ppm)	983	64	
Calcium (ppm)	38	15	
Magnesium (ppm)	108	3	
Hardness mg equivalent CaCO3 (ppm)	547	49	
Conductivity (mmhos/cm)	6.33	0.51	
Sodium Absorption Ratio	18.42	3.97	
Total Dissolved Solids (ppm)	3474	234	
Turbidity (NTU)	4.05	2.38	

Table E.2: Water Characterization Data



Test Site Maintenance

The bermudagrass turf at the test site had been well established prior to conducting this study. It was subject to regular mowing on a daily basis by the golf course ground crew. The test site area was maintained as a top level golf course facility.

Date	Product	Rate			
Fertilizer Ap	plications				
02/26/2004	12/12/12	275 lbs/A			
04/08/2004	12/12/12	200 lbs/A			
06/29/2004	12/12/12	200 lbs/A			
Chemical Applications					
10/09/2003	MSMA/Sencor (metribuzin)	32 oz. + 1.5 oz./A			
10/17/2003	MSMA/Sencor (metribuzin)	32 oz. + 1.5 oz./A			
11/03/2003	Barricade (prodiamine)	1 lb/A			
11/05/2003	Pendimethalin	0.5 gallons/A			
03/01/2004	Ronstar (oxadiazon)	3 lbs/A			
03/15/2004	Pendimethalin	0.5 gallons/A			
07/13/2004	MSMA/Sencor (metribuzin)	32 oz. + 1.5 oz./A			
07/29/2004	MSMA/Sencor (metribuzin)	32 oz. + 1.5 oz./A			
08/09/2004	MSMA/Sencor (metribuzin)	32 oz. + 1.5 oz./A			
08/19/2004	Snare MCB	100 lbs/A			

The test area had in-ground irrigation capabilities. The precipitation and irrigation applied to the test site is provided in Appendix D of this report.

BASF Reg. Doc. # 2005/7004410



5.4 Water Analysis

5.4.1 Method Verification

The analytical method for water has been validated internally and has undergone a successful independent laboratory validation. The results of these studies are presented in separate reports (References 2 and 3).

The analytical method was verified prior to beginning analysis of the field samples using a fortification solution of a mixture of fipronil and its metabolites. The verification set consisted of 2 control samples of pre-application bulk water, 3 samples of bulk water fortified at the LOQ (0.01 ng/mL), 3 samples of bulk water fortified at 10XLOQ (0.10 ng/mL). Results of the method verification were satisfactory and are shown in Table I.1 (Appendix I).

5.4.2 Procedural Recoveries

Each sample set contained at least one control sample (pre-application bulk water) fortified with a mixture of fipronil, MB46513, MB45950 and MB46136. The fortification levels were 0.005, 0.010 or 0.100 ng/mL. The procedural recoveries are summarized in Table IV. In addition, all quantitative data for the procedural recoveries are included in Appendix I.

Analyte	Fortification Level (ng/mL)	Number	Mean Percent Recovery
	0.010	4	89.9
Fipronil	0.050	5	90.3
	0.100	19	88.2
	Mean	28	89.5
· · · · · · · · · · · · · · · · · · ·	0.010	4	92.9
MB46513	0.050	5	88.7
	0.100	19	98.8
	Mean	28	93.5
	0.010	4	95.7
MB45950	0.050	5	88.1
	0.100	19	97.6
	Mean	28	93.8
· .	0.010	4	98.9
MB46136	0.050	5	96.9
	0.100	19	96.7
· · · · · · · · · · · · · · · · · · ·	Mean	28	97.5

Table IV: Mean Procedural Recoveries from Fortified Bulk Water



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Table D.I. 2003 Daily Chinate Data	Table	D.1 .	2003	Daily	Climate Data
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		-	On-Site	NC	DAA Weather	Data ^a
	Days After	Irrigation	Rainfall	Rainfall	Maximum	Minimum
Date	Application	(inches)	(inches)	(inches)	Temp. (°F)	Temp. (°F)
01 May 2003				0.00	81	69
02 May 2003				0.00	85	67
03 May 2003				0.58	80	65
04 May 2003				0.00	84	68
05 May 2003				0.00	81	75
06 May 2003				0.00	82	77
07 May 2003				0.00	84	76
08 May 2003				0.00	84	76
09 May 2003				0.00	85	75
10 May 2003				0.00	86	76
11 May 2003				0.03	89	72
12 May 2003				0.00	86	68
13 May 2003				Т	85	64
14 May 2003				0.00	84	61
15 May 2003				0.30	85	73
16 May 2003				T	87	70
17 May 2003				0.00	86	77
18 May 2003				3.51	84	69
19 May 2003	-1	0.00	3.75	0.05	82	69
20 May 2003 ^b	0	0.00	0.00	0.01	82	71
21 May 2003	1	0.11	0.30	0.64	81	71
22 May 2003	2	0.00	2.50	1.14	83	69
23 May 2003	3	0.14	0.00	0.00	85	67
24 May 2003	4	0.14	0.00	0.00	83	65
25 May 2003	5	0.14	0.00	0.00	84	64
26 May 2003	6	0.14	0.00	0.00	87	69
27 May 2003	7	0.14	0.00	0.00	82	65
28 May 2003	8	0.14	0.00	0.00	84	62
29 May 2003	9	0.14	0.00	0.00	88	65
30 May 2003	10	0.14	0.00	0.00	91	69
31 May 2003	11	0.14	0.00	0.00	87	74
Total Rainfall (19	9-31 May)	1.37	6.55	6.26		
Monthly Normal	Rainfall (1973-	-2002) ^a	4.30		•	

^a Obtained from Pensacola Airport, Pensacola, FL .

^b Date of Application of Test Substance and Initiation of Sampling.



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	-		On-Site	NC	DAA Weather	Data ^a
	Days After	Irrigation	Rainfall	Rainfall	Maximum	Minimum
Date	Application	(inches)	(inches)	(inches)	Temp. (°F)	Temp. (°F)
01 Jun 2003	12	0.14	0.00	0.00	88	76
02 Jun 2003	13	0.14	0.00	0.09	89	71
03 Jun 2003	14	0.12	0.20	0.94	85	70
04 Jun 2003	15	0.14	0.00	0.00	86	73
05 Jun 2003	16	0.14	0.00	Т	86	72
06 Jun 2003	17	0.14	0.00	2.16	78	71
07 Jun 2003	18	0.00	1.50	0.16	82	75
08 Jun 2003	19	0.90	0.50	0.24	88	73
09 Jun 2003	20	0.08	0.60	Т	88	73
10 Jun 2003	21	0.11	0.00	Т	88	74
11 Jun 2003	22	0.11	0.00	0.14	88	75
12 Jun 2003	23	0.11	0.00	0.11	87	74
13 Jun 2003	24	0.11	0.00	0.20	87	74
14 Jun 2003	25	0.00	1.25	0.50	89	69
15 Jun 2003	26	0.11	0.00	0.37	87	74
16 Jun 2003	27	0.11	0.30	Т	89	74
17 Jun 2003	28	0.00	1.80	0.15	78	72
18 Jun 2003	29	0.12	0.20	1.01	85	73
19 Jun 2003	30	0.11	0.10	1.29	86	75
20 Jun 2003	31	0.00	1.00	0.73	89	74
21 Jun 2003	32	0.11	0.30	0.31	87	73
22 Jun 2003	33	0.11	0.30	0.20	86	74
23 Jun 2003	34	0.13	0.10	0.00	87	74
24 Jun 2003	35	0.14	0.00	0.02	87	77
25 Jun 2003	36	0.14	0.00	Т	88	74
26 Jun 2003	37	0.14	0.00	0.00	87	71
27 Jun 2003	38	0.14	0.00	Т	87	72
28 Jun 2003	39	0.00	1.00	0.16	81	73
29 Jun 2003	40	0.00	0.50	0.45	83	73
30 Jun 2003	41	0.00	4.20	5.26	80	70
Total Rainfall	·	3.60	13.85	14.49		
Monthly Normal	Rainfall (1973-	2002) ^a	6.22			

^a Obtained from Pensacola Airport, Pensacola, FL .



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			On-Site	NC	AA Weather	Data ^a
	Days After	Irrigation	Rainfall	Rainfall	Maximum	Minimum
Date	Application	(inches)	(inches)	(inches)	Temp. (°F)	Temp. (°F)
01 Jul 2003	42	0.00	4.20	1.44	85	74
02 Jul 2003	43	0.00	1.70	0.33	83	75
03 Jul 2003	44	0.14	0.00	0.24	86	73
04 Jul 2003	45	0.90	0.50	0.49	81	71
05 Jul 2003	46	0.00	1.50	2.29	79	70
06 Jul 2003	47	0.14	0.00	Т	88	72
07 Jul 2003	48	0.14	0.00	0.01	87	75
08 Jul 2003	49	0.14	0.00	0.00	87	76
09 Jul 2003	50	0.14	0.00	0.00	89	75
10 Jul 2003	51	0.60	0.80	0.00	91	77
11 Jul 2003	52	0.14	0.00	Т	91	76
12 Jul 2003	53	0.00	1.00	0.22	89	72
13 Jul 2003	54	0.14	0.00	0.00	87	73
14 Jul 2003	55	0.10	0.40	0.05	82	74
15 Jul 2003	56	0.14	0.00	0.00	86	74
16 Jul 2003	57	0.14	0.00	0.00	90	72
17 Jul 2003	58	0.12	0.20	0.63	92	71
18 Jul 2003	59	0.14	0.00	0.00	89	72
19 Jul 2003	60	0.13	0.10	1.42	89	71
20 Jul 2003	61	0.14	0.00	0.04	88	71
21 Jul 2003	62	0.90	0.50	0.49	82	72
22 Jul 2003	63	0.20	1.20	1.11	89	71
23 Jul 2003	64	0.00	1.25	0.33	80	72
24 Jul 2003	65	0.70	0.75	0.40	86	73
25 Jul 2003	66	0.14	0.00	0.00	86	72
26 Jul 2003	67	0.14	0.00	0.01	86	73
27 Jul 2003	68	0.14	0.00	0.01	88	74
28 Jul 2003	69	0.14	0.00	0.01	89	75
29 Jul 2003	70	0.90	0.50	0.29	87	71
30 Jul 2003	71	0.14	0.00	0.01	88	71
31 Jul 2003	72	0.14	0.50	0.16	89	74
Total Rainfall		6.93	15.10	9.98		
Monthly Normal	Rainfall (1973-	-2002) ^a	8.28			

Table D 1 2003 Daily Climate Data (continued)

^a Obtained from Pensacola Airport, Pensacola, FL .



			On-Site	NC)AA Weather	Data ^a
н 	Days After	Irrigation	Rainfall	Rainfall	Maximum	Minimum
Date	Application	(inches)	(inches)	(inches)	Temp. (°F)	Temp. (°F)
01 Aug 2003	73	0.14	0.00	1.11	86	73
02 Aug 2003	74	0.14	0.00	0.29	86	73
03 Aug 2003	75	0.60	0.80	0.02	88	75
04 Aug 2003	76	0.13	0.10	0.18	90	75
05 Aug 2003	77	0.60	0.80	0.01	89	77
06 Aug 2003	78	0.90	0.50	0.56	87	74
07 Aug 2003	79	0.80	0.60	0.21	85	74
08 Aug 2003	80	0.80	0.60	0.00	89	73
09 Aug 2003	81	0.14	0.00	0.00	91	72
10 Aug 2003	82	0.14	0.00	0.00	89	73
11 Aug 2003	83	0.14	0.00	0.01	87	73
12 Aug 2003	84	0.60	0.80	0.80	80	72
13 Aug 2003	85	0.90	0.50	1.00	89	73
14 Aug 2003	86	0.14	0.00	0.00	93	73
15 Aug 2003	87	b	b	0.01	91	76
16 Aug 2003	88	b	^b	0.05	90	76
17 Aug 2003	89	b	^b	0.67	88	76
18 Aug 2003	90	b	b	0.31	89	75
19 Aug 2003	91	b	b	0.00	90	75
20 Aug 2003	92	b	^b	0.03	88	76
21 Aug 2003	93	^b	^b	0.12	84	75
22 Aug 2003	94	b	^b	0.23	86	74
23 Aug 2003	95	^b	b	0.10	89	74
24 Aug 2003	96	^b	^b	0.00	89	74
25 Aug 2003	97	^b	^b	Т	91	76
26 Aug 2003	98	b	^b	Т	90	75
27 Aug 2003	99	^b	b	0.00	89	76
28 Aug 2003	100	 b	^b	0.00	90	76
29 Aug 2003	101	b	^b	0.07	90	76
30 Aug 2003	102	b	^b	0.12	90	75
31 Aug 2003	103	b	^b	Т	89	75
Total Rainfall			4.70			
		6.17	(8/1-			
		(8/1-8/14)	8/14)	5.90		
Monthly Normal	Rainfall (1973-	-2002) ^a	6.10		-	

Table D.1. 2003 Daily Climate Data (continued)

^a Obtained from Pensacola Airport, Pensacola, FL .

^b Data not available.



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			On-Site	NC	AA Weather	Data ^a
	Days After	Irrigation	Rainfall	Rainfall	Maximum	Minimum
Date	Application	(inches)	(inches)	(inches)	Temp. (°F)	Temp. (°F)
01 Sep 2003	104	^b	b	0.00	90	76
02 Sep 2003	105	b	b	0.00	91	77
03 Sep 2003	106	^b	^b	0.00	89	75
04 Sep 2003	107	^b	b	0.35	94	74
05 Sep 2003	108	^b	^b	0.01	91	73
06 Sep 2003	109	b	^b	0.05	80	73
07 Sep 2003	110	b	^b	0.00	83	70
08 Sep 2003	111	^b	b	0.00	86	70
09 Sep 2003	112	^b	b	0.00	85	71
10 Sep 2003	113	^b	b	0.00	88	73
11 Sep 2003	114	^b	 b	0.00	87	70
12 Sep 2003	115	^b	^b	0.00	85	68
13 Sep 2003	116	^b	^b	0.78	86	72
14 Sep 2003	117	^b	^b	0.18	85	72
15 Sep 2003	118	b	^b	0.00	88	70
16 Sep 2003	119	 b	b	0.00	87	67
17 Sep 2003	120	b	b	0.00	88	68
18 Sep 2003	121	b	^b	0.00	88	68
19 Sep 2003	122	^b	^b	0.00	87	69
20 Sep 2003	123	b	^b	0.00	89	71
21 Sep 2003	124	^b	b	0.62	83	74
22 Sep 2003	125	b	^b	0.72	82	69
23 Sep 2003	126	b	^b	0.00	87	66
24 Sep 2003	127	b	b	0.00	87	66
25 Sep 2003	128	b	^b	0.15	84	71
26 Sep 2003	129	 _b	^b	0.00	84	70
27 Sep 2003	130	b	^b	Т	85	72
28 Sep 2003	131	^b	^b	0.00	83	62
29 Sep 2003	132	b	b	0.00	75	56
30 Sep 2003	133	b	^b	0.00	79	56
Total Rainfall		^b	^b	2.86		
Monthly Normal	Rainfall (1973-	-2002) ^a	4.15			

Table D.1.	2003 Daily	Climate Data	(continued)
			· · · · · · · · · · · · · · · · · · ·

^a Obtained from Pensacola Airport, Pensacola, FL.

^b Data not available.



· · · · · · · · · · · · · · · · · · ·			On-Site	NC	AA Weather	Data ^a
	Days After	Irrigation	Rainfall	Rainfall	Maximum	Minimum
Date	Application	(inches)	(inches)	(inches)	Temp. (°F)	Temp. (°F)
01 Oct 2003	134	b	b	0.00	82	61
02 Oct 2003	135	b	^b	0.00	76	55
03 Oct 2003	136	^b	^b	0.00	77	55
04 Oct 2003	137	b	^b	0.00	81	63
05 Oct 2003	138	b	^b	0.00	82	63
06 Oct 2003	139	b	^b	Т	82	66
07 Oct 2003	140	b	^b	0.00	83	66
08 Oct 2003	141	b	^b	Т	85	68
09 Oct 2003	142	b	b	0.01	79	66
10 Oct 2003	143	b	^b	1.25	76	69
11 Oct 2003	144	b	^b	0.29	71	68
12 Oct 2003	145	^b	^b	0.00	78	68
13 Oct 2003	146	^b	b	0.00	81	69
14 Oct 2003	147	b	b	Т	86	62
15 Oct 2003	148	 b	^b	0.00	76	52
16 Oct 2003	149	^b	^b	0.00	75	50
17 Oct 2003	150	^b	^b	0.00	79	54
18 Oct 2003	151	^b	^b	0.00	77	58
19 Oct 2003	152	b	^b	0.00	80	54
20 Oct 2003	153	 b	b	0.00	83	53
21 Oct 2003	154	^b	 b	0.00	86	58
22 Oct 2003	155	^b	 b	0.00	88	64
23 Oct 2003	156	^b ,	^b	0.00	81	61
24 Oct 2003	157	b	^b	0.00	82	58
25 Oct 2003	158	b	b	0.00	80	64
26 Oct 2003	159	b	b	0.89	79	67
27 Oct 2003	160	^b	b	0.13	70	57
28 Oct 2003	161	 b	b	0.01	67	51
29 Oct 2003	162	^b	b	0.00	76	50
30 Oct 2003	163	b	b	0.00	77	56
31 Oct 2003	164	b	b	0.00	79	61
Total Rainfall		^b	^b	2.58		······································
Monthly Normal	Rainfall (1973-	2002) ^a	4.38			

Table D.1. 2003 Daily Climate Data (continued)

^a Obtained from Pensacola Airport, Pensacola, FL.

^b Data not available.



			On-Site	NC	DAA Weather	Data ^a
Date	Days After Application	Irrigation (inches)	Rainfall (inches)	Rainfall (inches)	Maximum Temp. (°F)	Minimum Temp. (°F)
01 Nov 2003	165	0.14	0.00	0.00	82	63
02 Nov 2003	166	0.00	0.00	0.00	84	62
03 Nov 2003	167	0.00	0.00	T	84	58
04 Nov 2003	168	0.14	0.00	0.30	79	70
05 Nov 2003	169	0.00	0.00	Т	82	70
06 Nov 2003	170	0.00	0.00	0.00	86	68
07 Nov 2003	171	0.14	0.00	0.00	80	63
08 Nov 2003	172	0.00	0.00	0.00	77	58
09 Nov 2003	173	0.00	0.00	0.00	68	58
10 Nov 2003	174	0.14	0.00	0.00	74	53
11 Nov 2003	175	0.00	0.00	0.00	77	58
12 Nov 2003	176	0.00	0.00	Т	82	59
13 Nov 2003	177	0.14	0.00	0.00	71	49
14 Nov 2003	178	0.00	0.00	0.00	66	41
15 Nov 2003	179	0.00	0.00	0.00	76	47
16 Nov 2003	180	0.14	0.00	Т	82	58
17 Nov 2003	181	0.00	0.00	Т	77	65
18 Nov 2003	182	0.00	0.00	1.00	79	66
19 Nov 2003	183	0.00	1.75	0.00	68	48
20 Nov 2003	184	0.00	0.00	0.00	75	46
21 Nov 2003	185 ^b	0.00	0.00	0.00	74	47
22 Nov 2003	186	0.14	0.00	0.00	74	50
23 Nov 2003	187	0.00	0.00	0.26	78	60
24 Nov 2003	188	0.00	0.62	0.43	70	38
25 Nov 2003	189	0.00	0.00	0.00	55	33
26 Nov 2003	190	0.00	0.00	0.00	68	39
27 Nov 2003	191	0.00	0.00	0.55	81	63
28 Nov 2003	192	0.00	1.6	0.10	69	41
29 Nov 2003	193	0.00	0.00	0.00	55	33
30 Nov 2003	194	0.00	0.00	0.00	60	32
Total Rainfall		0.98	3.97	2.64		
Monthly Normal	Rainfall (1973-	-2002) ^a	3.91			

Table D.1. 2003 Daily Climate Data (continued)

^a Obtained from Pensacola Airport, Pensacola, FL .

^b Field Phase Complete



Table D.2. 2004 Daily Climate Data

Air Relative Estuary On-site NOAA													
		Temp	eratur	e (°C)	Hu	midity	(%)	Temp	peratur	e (°C)	Rain.	Rain.	Irrig.
Date	DAA	Min	Max	Avg	Min	Max	Avg	Min	Max	Avg	(in.)	(in.)	(in.)
04/01/04	-20	9.8	26.6	17.1	16.8	62.3	40.4	9.3	26.5	16.5	0.01	0.00	
04/02/04	-19	8.0	17.1	12.9	33.1	74.3	58.0	7.7	23.7	16.6	0.03	0.00	
04/03/04	-18	11.2	23.9	16.2	20.4	84.8	52.8	19.8	23.2	21.4	0.01	0.00	
04/04/04	-17	11.8	23.9	16.8	21.5	81.5	49.6	20.8	24.3	22.2	0.02	0.00	
04/05/04	-16	12.6	26.3	17.5	18.5	70.1	48.6	20.6	25.3	22.4	0.02	0.00	
04/06/04	-15	11.4	18.3	15.0	27.5	76.9	46.5	18.5	23.1	20.8	0.00	0.00	
·04/07/04	-14	12.7	20.5	17.3	35.4	78.1	64.4	18.4	23.9	20.8	0.48	0.94	
04/08/04	-13	17.5	21.6	18.5	69.1	90.8	85.7	19.3	21.0	20.0	0.00	0.00	
04/09/04	-12	18.4	22.1	20.2	73.0	90.7	82.5	19.6	22.9	21.2	0.06	0.00	
04/10/04	-11	14.9	23.4	19.3	53.1	90.2	75.2	20.6	24.0	22.2	0.00	0.00	
04/11/04	-10	15.3	23.4	19.8	58.5	91.9	78.6	20.8	26.2	23.4	0.31	0.24	
04/12/04	-9	16.9	23.0	19.1	55.3	90.9	81.3	20.0	23.2	21.3	0.01	0.01	
04/13/04	-8	16.2	21.6	19.5	64.8	90.3	81.5	20.0	22.4	21.3	0.00	0.00	
04/14/04	-7	10.1	16.2	11.7	47.2	74.8	59.8	16.1	22.2	18.7	0.00	0.00	
04/15/04	-6	10.3	21.9	15.1	21.6	62.1	41.5	14.0	18.8	16.5	0.00	0.00	
04/16/04	-5	9.8	20.9	15.2	27.0	83.4	54.5	17.0	25.1	20.0	0.00	0.00	
04/17/04	-4	11.0	21.2	17.2	40.6	90.7	70.4	19.9	28.1	23.2	0.02	0.00	
04/18/04	-3	13.2	20.9	17.3	65.6	91.6	80.8	20.6	28.7	24.0	0.00	0.00	
04/19/04	-2	13.7	21.7	17.7	68.1	93.1	84.5	21.1	28.8	24.6	0.01 ^b	0.00	
04/20/04	-1	14.3	21.3	18.5	58.1	93.1	81.6	21.8	28.1	24.4	0.01 ^b	0.00	0.14
04/21/04	0	18.6	21.0	19.7	62.0	87.4	78.9	21.6	26.8	23.5	0.00	0.00	
04/22/04	1	19.8	22.1	20.7	72.3	89.1	83.5	21.0	25.0	22.7	0.01	Т	
04/23/04	2	20.1	22.6	21.1	76.6	90.2	83.6	21.2	25.9	23.1	0.08	0.00	0.14
04/24/04	3	19.5	22.6	20.9	73.3	90.4	83.6	21.4	26.6	23.8	0.05	0.00	
04/25/04	4	17.8	23.4	21.2	73.5	92.6	84.8	22.7	27.8	24.8	0.00	0.00	
04/26/04	5	20.5	23.8	22.0	71.0	91.2	82.6	22.9	27.6	25.1	0.36	0.53	0.14
04/27/04	6	19.0	24.5	21.4	68.2	92.2	79.5	23.4	26.9	25.0	0.12	0.00	
04/28/04	7	15.7	26.4	20.5	28.2	70.4	50.2	22.0	26.8	24.3	0.00	0.00	
04/29/04	8	15.6	22.3	19.6	30.7	70.7	51.8	21.0	25.5	23.4	0.21	0.70	0.14
04/30/04	9	16.2	21.8	20.4	62.4	89.9	79.0	19.0	22.9	22.0	0.44	0.45	
Total 2.26 2.87 0.56													
						Cumu	lative R	Rainfall	and Irrig	gation	2.82		
					Mo	nthly No	ormal R	Rainfall	(1973-2	2002) ^a	3.92		
^a Obtained	from P	ensaco	la Airpo	ort, Pen	sacola	, FL .							
° Rain gau	Rain gauge calibration water input values of 0.89 and 0.96 inches were removed from data totals												
on 04/19	/04 and	04/20/	04, r esp	pectivel	у.								

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Tabl	le D.2	. 2004	Daily	Climate	Data	(continued))
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	-	Air Relative Estuary On-site NOAA											
		Temp	peratur	e (°C)	Hu	midity	(%)	Tem	peratur	e (°C)	Rain.	Rain.	Irrig.
Date	DAA	Min	Max	Avg	Min	Max	Avg	Min	Max	Avg	(in.)	(in.)	(in.)
05/01/04	10	18.2	21.1	19.8	83.1	91.8	89.2	20.7	22.3	21.8	0.00	0.00	
05/02/04	11	19.5	23.2	21.7	86.7	93.2	90.9	20.9	26.9	23.5	0.24	0.44	
05/03/04	12	15.6	22.8	20.2	69.2	91.2	80.8	22.8	25.0	24.0	0.04	0.00	
05/04/04	13	13.5	24.3	18.1	24.2	80.4	54.9	20.7	24.3	22.3	0.00	0.00	0.14
05/05/04	14	12.5	21.5	16.8	28.9	75.6	58.8	18.3	25.7	21.2	0.01	0.00	
05/06/04	15	13.9	23.3	19.5	40.4	88.8	71.1	19.5	27.1	22.7	0.04	0.00	
05/07/04	16	16.4	25.5	21.3	36.7	91.3	71.6	20.8	27.9	23.8	0.05	0.00	0.14
05/08/04	17	16.9	25.9	21.7	43.9	90.3	71.2	21.5	28.5	24.6	0.02	0.00	
05/09/04	18	18.8	30.3	23.3	33.8	87.0	67.7	23.6	27.5	25.3	0.00	0.00	
05/10/04	19	20.8	25.4	23.4	59.1	88.5	75.7	23.9	29.3	26.2	0.01	0.00	0.14
05/11/04	20	19.1	24.6	22.7	66.6	89.9	80.9	23.1	28.8	26.0	0.00	0.00	
05/12/04	21	20.5	24.4	22.8	64.7	89.7	80.3	24.0	28.9	26.5	0.00	0.00	
05/13/04	22	22.1	24.6	23.3	76.6	89.4	84.1	25.1	27.8	26.5	0.00	Т	0.14
05/14/04	23	22.4	24.5	23.3	78.5	90.2	86.3	25.3	27.5	26.3	0.00	0.00	
05/15/04	24	22.0	24.5	23.2	75.8	87.5	81.8	24.6	28.6	26.4	0.07	0.00	
05/16/04	25	21.0	25.2	23.1	76.8	88.1	83.5	24.6	28.1	26.5	0.00	0.00	0.14
05/17/04	26	21.1	25.3	23.1	74.8	88.6	82.7	25.3	28.9	26.8	0.02	0.00	
05/18/04	27	19.9	25.1	23.2	75.8	90.1	83.2	24.7	29.2	26.9	0.03	0.00	
05/19/04	28	20.0	25.4	23.4	76.0	91.6	84.8	26.0	30.1	27.7	0.01	0.00	0.14
05/20/04	29	21.2	25.3	23.7	67.2	91.2	80.3	26.5	31.2	28.4	0.02	Т	
05/21/04	30	19.9	26.2	23.8	63.7	91.2	78.5	26.1	30.5	28.1	0.01	0.00	
05/22/04	31	21.4	28.0	24.9	44.3	90.6	73.7	26.2	30.8	28.1	0.00	0.00	0.14
05/23/04	32	21.8	26.5	24.8	64.1	84.4	74.2	25.4	30.8	27.7	0.00	Т	
05/24/04	33	24.0	26.6	25.3	66.6	80.2	75.6	24.9	29.9	27.2	0.00	0.00	
05/25/04	34	24.3	27.0	25.6	64.5	78.3	72.7	25.4	30.6	27.5	0.00	0.00	0.14
05/26/04	35	23.1	27.5	25.6	59.6	76.2	69.0	25.5	30.6	27.9	0.00	0.00	
05/27/04	36	24.2	27.8	25.9	57.1	76.1	68.8	25.7	30.7	28.2	0.00	0.00	
05/28/04	37	24.4	27.5	26.0	64.3	78.9	72.7	26.0	31.3	28.4	0.00	0.00	0.14
05/29/04	38	23.6	27.6	25.7	62.7	78.7	72.9	26.0	31.3	28.4	0.00	Т	
05/30/04	39	24.0	27.7	26.1	73.8	86.9	79.6	26.5	32.3	28.8	0.00	Т	
05/31/04	40	25.7	28.2	27.0	77.3	87.0	82.3	27.4	32.6	29.6	0.55	0.48	
												· .	
Total 1.12 0.92 1.26													
						Cumu	lative R	ainfall	and Irrig	gation	2.38		
					Mor	nthly No	ormal R	ainfall	(1973-2	.002) ^a	4.30		
^a Obtained	from P	ensaco	la Airpo	ort. Pen	sacola.	FL.							

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	Fable D.2.	2004 Daily	Climate Data	(continued)	ļ
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-		Air Relative Estuar							y	On-site	NOAA	<u> </u>	
		Temp	peratur	e (°C)	Hu	midity	<u>(%)</u>	Tem	peratur	e (°C)	Rain.	Rain.	Irrig.
Date	DAA	Min	Max	Avg	Min	Max	Avg	Min	Max	Avg	(in.)	(in.)	(in.)
06/01/04	41	23.0	28.4	26.3	69.5	88.9	81.3	26.9	32.3	28.6	0.97	1.58	
06/02/04	42	20.0	27.1	23.9	63.2	89.8	82.4	24.9	27.9	26.9	0.16	0.20	
06/03/04	43	20.8	26.3	23.9	71.1	87.9	80.1	24.6	27.3	25.5	0.37	0.23	
06/04/04	44	19.8	26.9	24.4	69.2	90.8	80.9	24.4	27.0	25.5	0.03	0.00	
06/05/04	45	23.0	27.3	25.0	61.3	88.2	76.9	24.8	29.6	26.8	0.07	Т	
06/06/04	46	21.5	26.7	24.5	61.0	86.6	77.7	25.8	29.9	27.7	0.18	0.13	
06/07/04	47	22.6	26.8	25.0	63.9	88.6	79.1	26.2	29.7	28.3	0.02	0.01	
06/08/04	48	22.2	27.8	25.3	67.0	90.4	81.2	25.8	31.1	28.5	0.04	0.02	
06/09/04	49	23.0	27.9	26.2	69.6	90.8	80.5	26.6	32.1	29.3	0.00	Т	
06/10/04	50	23.1	28.8	26.4	59.4	91.0	75.9	27.7	33.3	30.5	0.00	0.24	
06/11/04	51	23.7	29.0	27.0	57.9	87.7	73.1	28.4	34.3	31.6	0.00	0.03	
06/12/04	52	26.1	29.2	27.7	66.8	79.4	73.7	29.4	33.2	30.8	0.00	Т	
06/13/04	53	26.0	29.6	28.0	59.9	83.4	73.6	28.7	33.9	30.6	0.15	0.84	
06/14/04	54	23.9	28.8	27.1	66.2	89.4	76.1	29.1	33.2	30.7	2.24	3.09	
06/15/04	55	22.0	27.1	24.3	75.3	91.5	86.1	26.0	29.7	27.6	0.30	0.22	
06/16/04	56	23.2	28.6	26.7	76.8	89.3	82.7	25.7	30.2	27.0	0.00	Т	
06/17/04	57	26.8	29.4	27.9	73.2	86.0	80.8	27.2	32.4	28.6	0.02	Т	
06/18/04	58	25.3	29.5	27.8	70.3	88.4	79.0	27.7	32.0	30.0	0.03	Т	
06/19/04	59	25.0	30.4	28.1	58.4	89.6	75.8	28.8	34.0	31.5	0.01	Т	
06/20/04	60	24.1	30.9	27.9	61.0	87.1	76.5	28.6	34.4	32.1	0.61	2.40	
06/21/04	61	21.8	31.7	26.6	45.8	90.0	79.2	30.0	32.3	31.1	0.10	Т	
06/22/04	62	22.5	29.9	26.7	63.7	91.7	78.5	29.7	32.8	30.9	0.66	0.72	
06/23/04	63	20.7	29.0	25.4	60.2	88.9	78.6	26.7	32.0	29.2	0.29	0.22	
06/24/04	64	22.9	29.8	26.9	62.4	88.8	78.0	26.2	30.0	28.0	0.17	0.79	
06/25/04	65	23.4	29.3	27.8	69.0	87.3	75.6	26.4	32.7	29.6	0.65	0.18	-
06/26/04	66	21.9	29.1	26.6	60.6	90.2	74.7	27.2	31.3	29.0	0.03	Т	
06/27/04	67	24.3	29.7	27.4	54.5	84.0	72.7	27.2	33.5	29.7	0.05	Т	
06/28/04	68	23.5	29.7	26.8	58.5	79.6	71.3	27.4	31.6	29.2	0.13	0.28	
06/29/04	69	22.8	29.2	25.7	65.9	87.7	77.7	27.2	30.8	29.3	0.34	0.33	
06/30/04 70 20.8 27.3 24.8 61.6 88.5 80.7 27.1 29.4 28.7 0.02 T													
	Total 7.64 11.51 0.00												
						Cumu	lative R	ainfall	and Irrig	gation	7.64		
					Mor	nthly No	ormal R	ainfall	(1973-2	2002) ^a	6.22		
										······································	··- · · · · · · · · · · · · · · · · · ·		
^a Obtained	Obtained from Pensacola Airport Pensacola El												

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Tab	le D.2.	2004]	Daily	Climate Data	(continued))
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		Air Relative Estuary						y.	On-site	NOAA			
		Temp	peratur	e (°C)	Hu	midity	(%)	Temp	peratur	e (°C)	Rain.	Rain.	Irrig.
Date	DAA	Min	Max	Avg	Min	Max	Avg	Min	Max	Avg	(in.)	(in.)	(in.)
07/01/04	71	22.9	28.2	25.7	68.2	89.2	80.3	27.2	32.5	29.3	0.01	Т	
07/02/04	72	22.8	29.1	26.4	67.5	86.4	78.2	28.2	32.6	29.9	0.11	1.25	0.14
07/03/04	73	23.4	28.7	26.4	64.9	87.3	77.8	28.0	32.4	30.2	0.00	0.13	
07/04/04	74	24.2	28.8	27.2	62.1	83.6	73.3	27.2	31.7	29.1	0.04	0.05	
07/05/04	75	25.5	30.1	28.2	65.1	81.8	73.1	27.1	32.9	29.5	0.03	0.11	
07/06/04	76	26.8	30.3	28.8	62.2	77.8	70.6	28.0	33.7	30.2	0.04	0.65	
07/07/04	77	25.2	30.7	27.8	57.8	84.5	73.7	29.5	34.7	31.6	0.05	Т	
07/08/04	78	23.7	30.5	27.4	55.9	87.7	73.3	30.3	34.8	32.0	0.04	Т	
07/09/04	79	22.8	28.6	26.3	65.2	84.8	73.1	28.6	31.4	30.2	0.00	Т	0.14
07/10/04	80	24.3	29.9	28.2	59.6	82.2	68.8	27.9	34.7	30.8	0.03	0.12	
07/11/04	81	26.2	32.6	28.8	57.3	86.7	71.1	29.5	36.0	32.4	0.03	Т	
07/12/04	82	25.6	31.6	28.7	55.7	85.9	71.9	31.5	36.1	33.2	0.07	0.00	0.14
07/13/04	83	26.2	33.3	29.2	46.4	79.3	68.2	30.1	33.3	31.6	0.76	2.46	
07/14/04	84	22.7	32.2	27.2	48.7	88.1	75.6	29.3	33.3	30.4	1.14	0.98	
07/15/04	85	22.6	30.2	26.7	66.0	90.8	76.9	26.9	33.0	29.6	0.00	0.08	
07/16/04	86	23.5	31.5	28.3	60.1	83.9	75.1	28.3	31.2	29.5	0.02	0.07	
07/17/04	87	23.5	30.3	27.2	55.9	85.7	69.8	26.3	29.5	27.8	1.70	0.41	
07/18/04	88	22.6	27.8	25.6	73.7	90.5	80.7	26.2	28.3	27.7	0.14	0.69	
07/19/04	89	23.3	28.6	26.6	63.1	88.9	80.0	26.4	30.2	28.3	0.00	Т	
07/20/04	90	25.5	31.1	28.5	49.1	79.9	66.0	28.3	32.3	30.8	0.03	0.00	
07/21/04	91	23.5	29.9	26.9	54.0	82.7	67.4	28.5	32.2	30.3	0.07	0.00	
07/22/04	92	23.5	30.6	27.0	54.9	89.0	72.5	28.5	33.2	31.0	0.03	0.00	
07/23/04	93	24.3	32.7	27.8	55.3	81.5	68.3	29.3	34.5	31.5	0.05	0.00	0.14
07/24/04	94	25.6	33.0	28.7	46.7	81.4	69.4	30.0	36.0	32.3	0.13	0.22	
07/25/04	95	26.0	33.6	29.3	46.8	84.3	70.1	31.0	36.4	33.0	0.03	0.31	
07/26/04	96	26.7	30.1	28.4	70.1	84.0	77.0	30.9	35.4	33.0	1.00	0.27	0.14
07/27/04	97	23.4	28.8	26.5	61.4	89.0	79.6	28.8	32.3	30.7	0.06	0.63	
07/28/04	98	24.3	29.5	27.0	64.2	89.1	77.4	29.1	34.1	30.9	0.00	0.00	
07/29/04	99	23.8	30.4	27.6	58.6	84.6	74.5	30.5	35.5	32.2	0.04	Т	
07/30/04	100	24.5	30.3	27.7	65.4	87.4	76.6	30.3	34.2	31.6	0.52	0.33	i .
07/31/04 101 23.1 30.3 27.7 65.7 89.9 77.7 29.2 32.8 30.6 0.00 T													
										Total	6.17	8.76	0.70
						Cumu	lative R	ainfall	and Irrig	gation	6.87		
					Moi	nthly No	ormal R	ainfall	(1973-2	2002) ^a	8.28		
^a Obtained	l from P	ensaco	la Airpo	ort, Pen	sacola,	FL.							

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Table D.2. 2004 Daily Climate Data (continued)

		Temp	Air beratur	e (°C)	Hu	Relative Humidity (%)			Estuary Temperature (°C)			NOAA Rain.	Irria.
Date	DAA			· ·					r		Rain.		
		Min	Max	Avg	Min	Max	Avg	Min	Max	Avg	(in.)	(in.)	(in.)
08/01/04	102	25.2	31.0	28.9	63.5	85.3	71.5	28.4	31.6	29.7	0.04	0.01	
08/02/04	103	25.9	32.1	28.1	55.9	86.6	75.5	29.1	33.5	31.4	1.77	T	
08/03/04	104	23.0	30.5	27.0	73.4	104.5	84.2	28.3	32.3	30.4	0.00	0.00	
08/04/04	105	25.2	31.0	28.5	56.5	82.7	71.5	28.4	33.1	30.6	0.04	0.00	
08/05/04	106	24.5	31.4	28.4	55.7	87.2	71.1	30.5	35.0	32.1	0.06	0.00	
08/06/04	107	26.9	32.6	29.6	45.8	77.9	66.4	31.4	35.7	33.0	0.05	0.00	
08/07/04	108	24.8	33.7	28.3	44.3	84.3	67.9	30.5	33.9	32.1	0.04	0.00	0.14
08/08/04	_109	23.0	30.8	26.7	44.0	77.6	61.4	28.1	32.5	30.6	0.01	Т	
08/09/04	110	24.2	28.8	26.5	47.8	80.8	66.6	27.7	31.2	29.1	0.47	0.89	
08/10/04	111	23.8	28.9	26.5	71.3	90.0	82.0	27.4	30.1	28.5	0.98	0.98	
08/11/04	112	23.0	28.5	26.2	67.6	91.0	82.6	26.9	29.6	28.1	0.96	0.01	
08/12/04	113	22.8	27.5	25.7	75.3	115.0	93.1	26.9	28.7	27.9	0.25	1.92	
08/13/04	114	22.5	29.3	24.9	68.6	99.1	83.9	26.5	30.4	28.0	0.03	0.00	
08/14/04	115	19.5	.29.3	23.5	46.1	89.7	69.3	26.6	29.6	28.0	0.04	0.00	
08/15/04	116	17.2	30.6	22.8	32.5	76.1	58.3	22.9	27.1	25.3	0.05	0.00	
08/16/04	117	19.2	31.6	24.4	38.2	75.1	60.1	23.6	27.8	25.4	0.03	0.00	
08/17/04 118 20.7 28.9 25.1 55.7 76.8 67.4 25.0 30.4 27.0 0.04 0.00 0.1					0.14								
08/18/04	119	21.6	30.9	25.9	39.4	76.0	60.6	26.2	31.5	28.2	0.03	0.00	
08/19/04	120	21.5	29.4	25.7	42.1	81.1	64.6	27.1	31.7	28.9	0.05	Т	
08/20/04	121	22.5	29.0	26.5	62.6	86.1	75.3	28.0	33.3	30.3	0.03	1.18	0.14
08/21/04	122	23.3	29.2	27.6	70.4	87.3	78.7	28.1	32.7	30.3	0.82	0.24	
08/22/04	123	23.4	29.8	26.1	64.1	87.9	80.4	29.3	33.7	30.8	0.00	Т	
08/23/04	124	22.8	30.5	26.0	60.2	89.3	77.7	30.0	35.5	31.9	0.21	0.67	
08/24/04	125	21.9	29.6	25.5	59.9	89.0	79.6	30.1	33.8	31.2	0.00	Т	
08/25/04	126	23.6	29.9	27.1	61.6	87.7	76.3	29.8	34.5	31.6	0.02	0.02	
08/26/04	127	25.3	30.6	27.5	60.2	89.8	77.0	30.3	34.5	31.7	0.14	Т	
08/27/04	128	24.0	29.5	26.8	63.5	88.6	79.8	29.9	32.8	31.0	0.03	0.00	
08/28/04	129	24.3	30.3	27.5	62.3	89.8	77.6	29.9	33.4	31.1	0.04	0.00	
08/29/04	130	24.7	29.8	27.5	63.6	90.0	78.0	29.6	33.1	31.0	0.05	Т	
08/30/04	131	23.6	29.7	26.6	65.8	88.5	78.2	29.0	32.2	30.4	0.17	0.22	
08/31/04	132	73 ^a	28.3	b	b	88.2	b	28.5	30.3	29.4	0.35	0.20	
	Total 6.80 6.34 0.42												
						Cumu	lative R	ainfall	and Irrig	gation	7.22		
					Mo	onthly No	ormal R	ainfall	(1973-2	.002) ^a	6.89		
^a Obtained	from P	ensaco	la Airpo	ort, Pen	sacola,	FL.							
Probe ma	^o Probe malfunctioned, reliable data not available.												

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Table D.2. 2004 Daily Climate Data (continued)

Air Relative Estuary On-site NOAA													
]	Temp	eratur	e (°C)	Hu	midity	(%)	Temp	peratur	e (°C)	Rain.	Rain.	Irrig.
Date	DAA	Min	Max	Avg	Min	Max	Avg	Min	Max	Avg	(in.)	(in.)	(in.)
09/01/04	133	70 ^a	26.8	b	b	89.2	b	25.7	29.1	28.1	0.06	0.71	-
09/02/04	134	23.0	29.1	25.8	66.2	90.7	79.1	27.4	34.0	29.9	0.01	0.00	
09/03/04	135	23.5	30.4	27.0	63.9	89.2	77.9	29.7	34.0	31.7	0.09	0.00	
09/04/04	136	23.9	29.8	26.8	56.5	86.6	76.8	30.5	33.9	32.4	0.05	0.00	
09/05/04	137	74 ^a	33.9	b	b	80.9	b	30.4	33.0	31.7	0.07	0.00	0.14
09/06/04	138	23.7	34.1	27.8	45.2	81.1	67.2	28.4	31.6	29.9	0.09	Т	
09/07/04	139	24.4	29.2	26.3	60.2	74.6	68.5	25.9	29.1	27.0	0.00	Т	
09/08/04	140	24.3	31.7	27.4	50.7	79.8	64.2	24.6	27.6	26.0	0.06	0.00	0.14
09/09/04	141	22.8	28.9	25.9	61.9	80.9	72.7	25.9	32.0	28.3	0.05	0.00	
09/10/04	142	23.4	29.5	26.5	59.9	87.9	74.2	27.4	32.7	29.5	0.03	0.00	
09/11/04	143	23.3	29.6	25.8	61.9	87.1	77.1	28.4	31.1	29.5	0.05	0.00	0.14
09/12/04	144	22.4	29.0	24.8	63.2	90.0	81.0	27.6	30.3	28.7	0.06	0.27	
09/13/04	145	22.4	28.9	25.9	62.0	89.5	76.9	27.4	30.3	28.7	0.01	1.10	
09/14/04	146 ^c	23.2	29.1	25.4	61.4	85.4	77.2	27.2	29.3	28.0	0.00	0.00	
09/15/04 147 ^d 3.19 ^e 3.56													
09/16/04 148 NA 1.86													
09/17/04 149 NA T													
09/18/04	150										NA	0.00	
09/19/04	151										NA	0.00	
09/20/04	152										NA	0.00	
09/21/04	153										NA	0.00	
09/22/04	154										NA	0.00	
09/23/04	155										NA	0.00	
09/24/04	156									-	NA	0.00	
09/25/04	157									-	NA	0.00	
09/26/04	158						-				NA	0.00	
09/27/04	159		1							-	NA	0.00	
09/28/04	160			-							NA	0.00	
09/29/04	161										NA	Т	
09/30/04	162		-								NA	0.00	
			· · ·										
										Total	3.82	7.50	0.42
						Cumu	lative R	lainfall	and Irri	gation	4.24		
	Monthly Normal Rainfall (1973-2002) ^a 6.10												
^a Obtained	l from P	ensaco	la Airpo	ort, Per	isacola	, FL .							
^b Probe m	alfunction	oned, re	eliable o	lata no	t availa	ble.							
^c Data coll	lected th	nrough	11:00 A	M Sep	tember	14, 200	04.				-		
^d A dash () indic	ates da	ta not a	vailabl	e due te	o dama	ge from	Hurric	ane Iva	n.			
^e Data col	^e Data collected through 11:08 PM September 15, 2004.												

03RAFIX020



Table F.1. Critical Dates – 2003 Samples

Sample ID 03RAFIX020-	Sample Event	Sample Date	DAA	Shipping Date	Receipt Date	Anal. Set	Anal. Date	Storage (Days)
·····				2003			1	
001	Pre-Trt	19 May 03	-1	28 Jul 03	13 Aug 03	02	10 Sep 03	114
003	Pre-Trt	19 May 03	-1	28 Jul 03	13 Aug 03	02	10 Sep 03	114
005	Pre-Trt	19 May 03	-1	28 Jul 03	13 Aug 03	02	10 Sep 03	114
007	1	20 May 03	0	28 Jul 03	13 Aug 03	02	10 Sep 03	113
009	1	20 May 03	0	28 Jul 03	13 Aug 03	02	10 Sep 03	113
011	1	20 May 03	0	28 Jul 03	13 Aug 03	02	10 Sep 03	113
013	2	21 May 03	1	28 Jul 03	13 Aug 03	02	10 Sep 03	112
015	2	21 May 03	1	28 Jul 03	13 Aug 03	02	10 Sep 03	112
017	2	21 May 03	1	28 Jul 03	13 Aug 03	02	10 Sep 03	112
019	3	22 May 03	2	28 Jul 03	13 Aug 03	02	10 Sep 03	111
020	3	22 May 03	2	28 Jul 03	13 Aug 03	08	23 Jan 04	246
021	3	22 May 03	2	28 Jul 03	13 Aug 03	03	11 Sep 03	112
022	3	22 May 03	2	28 Jul 03	13 Aug 03	08	23 Jan 04	246
023	3	22 May 03	2	28 Jul 03	13 Aug 03	03	11 Sep 03	112
024	3	22 May 03	2	28 Jul 03	13 Aug 03	08	23 Jan 04	246
025	4	28 May 03	8	28 Jul 03	13 Aug 03	03	11 Sep 03	106
027	4	28 May 03	8	28 Jul 03	13 Aug 03	03	11 Sep 03	106
029	4	28 May 03	8	28 Jul 03	13 Aug 03	03	11 Sep 03	106
031	5	05 Jun 03	16	28 Jul 03	13 Aug 03	03	11 Sep 03	98
033	5	05 Jun 03	16	28 Jul 03	13 Aug 03	03	11 Sep 03	98
035	5	05 Jun 03	16	28 Jul 03	13 Aug 03	03	11 Sep 03	98
037	6	08 Jun 03	19	28 Jul 03	13 Aug 03	03	11 Sep 03	95
039	6	08 Jun 03	19	28 Jul 03	13 Aug 03	03	11 Sep 03	95
041	6	08 Jun 03	19	28 Jul 03	13 Aug 03	03	11 Sep 03	95
043	7	13 Jun 03	24	28 Jul 03	13 Aug 03	03	11 Sep 03	90
045	7	13 Jun 03	24	28 Jul 03	13 Aug 03	03	11 Sep 03	90
047	7	13 Jun 03	24	28 Jul 03	13 Aug 03	03	11 Sep 03	90
049	8	19 Jun 03	30	28 Jul 03	13 Aug 03	03	11 Sep 03	84
051	8	19 Jun 03	30	28 Jul 03	13 Aug 03	03	11 Sep 03	84
053	8	19 Jun 03	30	28 Jul 03	13 Aug 03	03	11 Sep 03	84
055	9	25 Jun 03	36	28 Jul 03	13 Aug 03	03	11 Sep 03	78
057	9	25 Jun 03	36	28 Jul 03	13 Aug 03	03	11 Sep 03	78
059	9	25 Jun 03	36	28 Jul 03	13 Aug 03	03	11 Sep 03	78
061	10	30 Jun 03	41	28 Jul 03	13 Aug 03	04	12 Sep 03	74
063	10	30 Jun 03	41	28 Jul 03	13 Aug 03	04	12 Sep 03	· 74
065	10	30 Jun 03	41	28 Jul 03	13 Aug 03	04	12 Sep 03	74
067	11	02 Jul 03	43	28 Jul 03	13 Aug 03	04	12 Sep 03	72
069	11	02 Jul 03	43	28 Jul 03	13 Aug 03	04	12 Sep 03	72
071	11	02 Jul 03	43	28 Jul 03	13 Aug 03	04	12 Sep 03	72
073	12	06 Jul 03	47	28 Jul 03	13 Aug 03	04	12 Sep 03	68
075	12	06 Jul 03	47	28 Jul 03	13 Aug 03	04	12 Sep 03	68
077	12	06 Jul 03	47	28 Jul 03	13 Aug 03	04	12 Sep 03	68

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 $a = 1 \ldots \ldots k$



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Sample ID 03RAFIX020-	Sample Event	Sample Date	DAA	Shipping Date	Receipt Date	Anal. Set	Anal. Date	Storage (Days)
079	13	11 Jul 03	52	28 Jul 03	13 Aug 03	04	12 Sep 03	63
081	13	11 Jul 03	52	28 Jul 03	13 Aug 03	04	12 Sep 03	63
083	13	11 Jul 03	52	28 Jul 03	13 Aug 03	04	12 Sep 03	63
085	14	18 Jul 03	59	28 Jul 03	13 Aug 03	04	12 Sep 03	56
087	14	18 Jul 03	59	28 Jul 03	13 Aug 03	04	12 Sep 03	56
089	14	18 Jul 03	59	28 Jul 03	13 Aug 03	04	12 Sep 03	56
091	15	21 Jul 03	62	28 Jul 03	13 Aug 03	04	12 Sep 03	53
093	15	21 Jul 03	62	28 Jul 03	13 Aug 03	04	12 Sep 03	53
095	15	21 Jul 03	62	28 Jul 03	13 Aug 03	04	12 Sep 03	53
097	16	24 Jul 03	65	28 Jul 03	13 Aug 03	04	12 Sep 03	50
099	16	24 Jul 03	65	28 Jul 03	13 Aug 03	04	12 Sep 03	50
101	16	24 Jul 03	65	28 Jul 03	13 Aug 03	06	17 Sep 03	55
103	17	31 Jul 03	72	09 Sep 03	12 Sep 05	06	17 Sep 03	48
105	17	31 Jul 03	72	09 Sep 03	12 Sep 05	06	17 Sep 03	48
107	17	31 Jul 03	72	09 Sep 03	12 Sep 05	06	17 Sep 03	48
109	18	07 Aug 03	79	09 Sep 03	12 Sep 05	06	17 Sep 03	41
111	18	07 Aug 03	79	09 Sep 03	12 Sep 05	06	17 Sep 03	41
113	18	07 Aug 03	79	09 Sep 03	12 Sep 05	06	17 Sep 03	41
115	19	15 Aug 03	87	09 Sep 03	12 Sep 05	06	17 Sep 03	33
117	19	15 Aug 03	87	09 Sep 03	12 Sep 05	06	17 Sep 03	33
119	19	15 Aug 03	87	09 Sep 03	12 Sep 05	06	17 Sep 03	33
121	20	22 Aug 03	94	09 Sep 03	12 Sep 05	06	17 Sep 03	26
123	20	22 Aug 03	94	09 Sep 03	12 Sep 05	06	17 Sep 03	26
125	20	22 Aug 03	94	09 Sep 03	12 Sep 05	06	17 Sep 03	26
127	21	28 Aug 03	100	09 Sep 03	12 Sep 05	06	17 Sep 03	20
129	21	28 Aug 03	100	09 Sep 03	12 Sep 05	06	17 Sep 03	20
131	21	28 Aug 03	100	09 Sep 03	12 Sep 05	06	17 Sep 03	20
133	22	12 Sep 03	115	08 Dec 03	12 Jan 04	07	13 Jan 04	123
134	22	12 Sep 03	115	08 Dec 03	12 Jan 04	07	13 Jan 04	123
135	22	12 Sep 03	115	08 Dec 03	12 Jan 04	07	13 Jan 04	123
136	22	12 Sep 03	115	08 Dec 03	12 Jan 04	07	13 Jan 04	123
137	22	12 Sep 03	115	08 Dec 03	12 Jan 04	07	13 Jan 04	123
138	22	12 Sep 03	115	08 Dec 03	12 Jan 04	07	13 Jan 04	123
139	23	23 Sep 03	126	08 Dec 03	12 Jan 04	07	13 Jan 04	112
140	23	23 Sep 03	126	08 Dec 03	12 Jan 04	07	13 Jan 04	112
141	23	23 Sep 03	126	08 Dec 03	12 Jan 04	07	13 Jan 04	112
142	23	23 Sep 03	126	08 Dec 03	12 Jan 04	07	13 Jan 04	112
143	23	23 Sep 03	126	08 Dec 03	12 Jan 04	07	13 Jan 04	112
144	23	23 Sep 03	126	08 Dec 03	12 Jan 04	07	13 Jan 04	112
145	24	09 Oct 03	142	08 Dec 03	12 Jan 04	07	13 Jan 04	96
146	24	09 Oct 03	142	08 Dec 03	12 Ian 04	07	13 Jan 04	96

Table F.1. Critical Dates – 2003 Samples (continued)



Sample ID 03RAFIX020-	Sample Event	Sample Date	DAA	Shipping Date	Receipt Date	Anal. Set	Anal. Date	Storage (Days)		
2003 Sediment										
201	Pre-Trt	19 May 03	-1	28 Jul 03	13 Aug 03	10	01 Apr 04	318		
203	Pre-Trt	19 May 03	-1	28 Jul 03	13 Aug 03	10	01 Apr 04	318		
205	Pre-Trt	19 May 03	-1	28 Jul 03	13 Aug 03	10	01 Apr 04	318		
207	90	15 Aug 03	87	09 Sep 03	12 Sep 05	10	01 Apr 04	230		
209	90	15 Aug 03	87	09 Sep 03	12 Sep 05	11	22 Jul 04	342		
211	90	15 Aug 03	87	09 Sep 03	12 Sep 05	11	22 Jul 04	342		
213	180	21 Nov 03	185	08 Dec 03	21 Nov 04	11	22 Jul 04	244		
215	180	21 Nov 03	185	08 Dec 03	21 Nov 04	11	22 Jul 04	244		
217	180	21 Nov 03	185	08 Dec 03	21 Nov 04	11	22 Jul 04	244		

Table F.1. Critical Dates – 2003 Samples (continued)



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Table F.2. Critical Dates – 2004 Samples

Sample ID 03RAFIX020-	Sample Event	Sample Date	DAA	Shipping Date	Receipt Date	Anal. Set	Anal. Date	Storage (Days)
	1.1.1.1.1	Dair	<u> </u>		Date	500	Dar	(Dajs)
1002	T 1	20 1	0	2004	T 07 T-104	1 14	00.2104	107
2002		29 Apr 04	δ 0	30 Jun 04	0/Ju104	14	02 NOV 04	107
2003	1	29 Apr 04	<u>ð</u>	30 Jun 04	07 Jul 04	14	17 Aug 04	100
2005		30 Apr 04	9	30 Jun 04	07 Jul 04		17 Aug 04	112
2005	1	30 Apr 04	9	30 Jun 04	07 5104	15	20 Aug 04	250
3005	1	30 Apr 04	9	30 Jun 04	07 Jul 04	10	14 Jan US	196
1007	1	30 Apr 04	9	30 Jun 04	07 Jul 04	14	02 NOV 04	180
2007	1	30 Apr 04	9	30 Jun 04	07 5104	14	02 NOV 04	180
3007		30 Apr 04	9	30 Jun 04	07 Jul 04	18	14 Jan 05	259
1009	1	30 Apr 04	<u> </u>	30 Jun 04	07 Jul 04	14	02 Nov 04	186
2009	1	30 Apr 04	9	30 Jun 04	07 Jul 04	14	02 Nov 04	186
1011		30 Apr 04	9	<u>30 Jun 04</u>	07 Jul 04	12	17 Aug 04	109
2011	1	30 Apr 04	9	<u>30 Jun 04</u>	07 Jul 04	13	20 Aug 04	112
1013		30 Apr 04	9	30 Jun 04	07 Jul 04	12	17 Aug 04	109
2013	1	30 Apr 04	9	30 Jun 04	07 Jul 04	13	20 Aug 04	112
1027	2	02 May 04	11	30 Jun 04	07 Jul 04	14	02 Nov 04	184
2027	2	02 May 04	11	30 Jun 04	07 Jul 04	14	02 Nov 04	184
1029	2	02 May 04	11	30 Jun 04	07 Jul 04	12	17 Aug 04	107
2029	2	02 May 04	11	30 Jun 04	07 Jul 04	13	20 Aug 04	110
1075	4	31 May 04	40	30 Jun 04	07 Jul 04	14	02 Nov 04	155
2075	4	31 May 04	40	30 Jun 04	07 Jul 04	14	02 Nov 04	155
3075	4	31 May 04	40	30 Jun 04	07 Jul 04	18	14 Jan 05	228
1077	4	31 May 04	40	30 Jun 04	07 Jul 04	12	17 Aug 04	78
2077	4	31 May 04	40	30 Jun 04	07 Jul 04	13	20 Aug 04	81
3077	4	31 May 04	40	30 Jun 04	07 Jul 04	18	14 Jan 05	228
1083	4	01 Jun 04	41	30 Jun 04	07 Jul 04	14	02 Nov 04	154
2083	4	01 Jun 04	41	30 Jun 04	07 Jul 04	14	02 Nov 04	154
1085	4	01 Jun 04	41	30 Jun 04	07 Jul 04	12	17 Aug 04	77
2085	4	01 Jun 04	41	30 Jun 04	07 Jul 04	13	20 Aug 04	80
3085	4	01 Jun 04	41	30 Jun 04	07 Jul 04	18	14 Jan 05	227
3087	4	01 Jun 04	41	30 Jun 04	07 Jul 04	18	14 Jan 05	227
3089	4	01 Jun 04	41	30 Jun 04	07 Jul 04	18	14 Jan 05	227
1111	5	03 Jun 04	43	30 Jun 04	07 Jul 04	14	02 Nov 04	152
2111	5	03 Jun 04	43	30 Jun 04	07 Jul 04	14	02 Nov 04	152
1113	5	03 Jun 04	43	30 Jun 04	07 Jul 04	12	17 Aug 04	75
2113	5	03 Jun 04	43	30 Jun 04	07 Jul 04	13	20 Aug 04	78
3115	5	03 Jun 04	43	30 Jun 04	07 Jul 04	18	14 Jan 05	225
3117	5	04 Jun 04	44	30 Jun 04	07 Jul 04	18	14 Jan 05	224
1147	7	14 Jun 04	54	30 Jun 04	07 Jul 04	14	02 Nov 04	141
2147	7	14 Jun 04	54	30 Jun 04	07 Jul 04	14	02 Nov 04	141
1149	7	14 Jun 04	54	30 Jun 04	07 Jul 04	12	17 Aug 04	64
2149	7	14 Jun 04	54	30 Jun 04	07 Jul 04	13	20 Aug 04	67
1151	7	14 Jun 04	54	30 Jun 04	07 Jul 04	12	17 Aug 04	64
2151	7	14 Jun 04	54	30 Jun 04	07 Jul 04	13	20 Aug 04	67
3151	7	14 Jun 04	54	30 Jun 04	07 Jul 04	18	14 Jan 05	214
1153	7	14 Jun 04	54	30 Jun 04	07 Jul 04	14	02 Nov 04	141



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Sample ID 03RAFIX020-	Sample Event	Sample Date	DAA	Shipping Date	Receipt Date	Anal. Set	Anal. Date	Storage (Days)
2153	7	14 Jun 04	54	30 Jun 04	07 Jul 04	14	02 Nov 04	141
3153	7	14 Jun 04	54	30 Jun 04	07 Jul 04	18	14 Jan 05	214
1155	7	14 Jun 04	54	30 Jun 04	07 Jul 04	14	02 Nov 04	141
2155	7	14 Jun 04	54	30 Jun 04	07 Jul 04	14	02 Nov 04	141
3155	7	14 Jun 04	54	30 Jun 04	07 Jul 04	18	14 Jan 05	214
1157	7	15 Jun 04	55	30 Jun 04	07 Jul 04	14	02 Nov 04	140
2157	7	15 Jun 04	55	30 Jun 04	07 Jul 04	14	02 Nov 04	140
3157	7	15 Jun 04	55	30 Jun 04	07 Jul 04	18	14 Jan 05	213
1159	7	15 Jun 04	55	30 Jun 04	07 Jul 04	12	17 Aug 04	63
2159	7	15 Jun 04	55	30 Jun 04	07 Jul 04	13	20 Aug 04	66
1195	9	20 Jun 04	60	30 Jun 04	07 Jul 04	14	02 Nov 04	135
2195	9	20 Jun 04	60	30 Jun 04	07 Jul 04	14	02 Nov 04	135
1197	9	20 Jun 04	60	30 Jun 04	07 Jul 04	13	20 Aug 04	61
2197	9	20 Jun 04	60	30 Jun 04	07 Jul 04	13	20 Aug 04	61
3197	9	20 Jun 04	60	30 Jun 04	07 Jul 04	18	14 Jan 05	208
3199	9	20 Jun 04	60	30 Jun 04	07 Jul 04	18	14 Jan 05	208
3201	9	21 Jun 04	61	30 Jun 04	07 Jul 04	18	14 Jan 05	207
1221	10	22 Jun 04	62	30 Jun 04	07 Jul 04	15	08 Nov 04	139
2221	10	22 Jun 04	62	30 Jun 04	07 Jul 04	15	08 Nov 04	139
1223	10	23 Jun 04	63	30 Jun 04	07 Jul 04	15	08 Nov 04	138
2223	10	23 Jun 04	63	30 Jun 04	07 Jul 04	15	08 Nov 04	138
3223	10	23 Jun 04	63	30 Jun 04	07 Jul 04	18	14 Jan 05	205
1235	10	24 Jun 04	64	30 Jun 04	07 Jul 04	13	20 Aug 04	57
2235	10	24 Jun 04	64	30 Jun 04	07 Jul 04	13	20 Aug 04	57
3235	10	24 Jun 04	64	30 Jun 04	07 Jul 04	18	14 Jan 05	204
1237	10	24 Jun 04	64	30 Jun 04	07 Jul 04	13	20 Aug 04	57
2237	10	24 Jun 04	64	30 Jun 04	07 Jul 04	13	20 Aug 04	57
1243	11	25 Jun 04	65	30 Jun 04	07 Jul 04	15	08 Nov 04	136
2243	11	25 Jun 04	65	30 Jun 04	07 Jul 04	15	08 Nov 04	136
1245	11	25 Jun 04	65	30 Jun 04	07 Jul 04	13	20 Aug 04	56
1247	11	25 Jun 04	65	30 Jun 04	07 Jul 04	15	08 Nov 04	136
2247	11	25 Jun 04	65	30 Jun 04	07 Jul 04	15	08 Nov 04	136
3253	11	25 Jun 04	65	30 Jun 04	07 Jul 04	18	14 Jan 05	203
3255	11	26 Jun 04	66	30 Jun 04	07 Jul 04	18	14 Jan 05	202
1267	12	29 Jun 04	69	07 Sep 04	27 Sep 04	15	08 Nov 04	132
2267	12	29 Jun 04	69	07 Sep 04	27 Sep 04	15	08 Nov 04	132
1269	12	29 Jun 04	69	07 Sep 04	27 Sep 04	15	08 Nov 04	132
2269	12	29 Jun 04	69	07 Sep 04	27 Sep 04	15	08 Nov 04	132
1291	13	13 Jul 04	83	07 Sep 04	27 Sep 04	15	08 Nov 04	118
2291	13	13 Jul 04	83	07 Sep 04	27 Sep 04	15	08 Nov 04	118
3291	13	13 Jul 04	83	07 Sep 04	27 Sep 04	18	14 Jan 05	185
1293	13	13 Jul 04	83	07 Sep 04	27 Sep 04	15	08 Nov 04	118
2293	13	13 Jul 04	83	07 Sep 04	27 Sep 04	15	08 Nov 04	118
3293	13	13 Jul 04	83	07 Sep 04	27 Sep 04	18	14 Jan 05	185
3295	13	14 Jul 04	84	07 Sep 04	27 Sep 04	18	14 Jan 05	184
1297	13	14 Jul 04	84	07 Sep 04	27 Sep 04	15	08 Nov 04	117

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Sample ID 03RAFIX020-	Sample Event	Sample Date	DAA	Shipping Date	Receipt Date	Anal. Set	Anal. Date	Storage (Days)
2297	13	14 Jul 04	84	07 Sep 04	27 Sep 04	15	08 Nov 04	117
1299	13	14 Jul 04	84	07 Sep 04	27 Sep 04	15	08 Nov 04	117
2299	13	14 Jul 04	84	07 Sep 04	27 Sep 04	15	08 Nov 04	117
1301	13	14 Jul 04	84	07 Sep 04	27 Sep 04	15	08 Nov 04	117
2301	13	14 Jul 04	84	07 Sep 04	27 Sep 04	15	08 Nov 04	117
3303	13	14 Jul 04	84	07 Sep 04	27 Sep 04	18	14 Jan 05	184
3305	13	14 Jul 04	84	07 Sep 04	27 Sep 04	18	14 Jan 05	184
1315	14	17 Jul 04	87	07 Sep 04	27 Sep 04	15	08 Nov 04	114
2315	14	17 Jul 04	87	07 Sep 04	27 Sep 04	15	08 Nov 04	114
1317	14	17 Jul 04	87	07 Sep 04	27 Sep 04	16	22 Nov 04	128
2317	14	<u>17 Jul 04</u>	87	07 Sep 04	27 Sep 04	16	22 Nov 04	128
1319	14	17 Jul 04	87	07 Sep 04	27 Sep 04	16	22 Nov 04	128
2319	14	<u>17 Jul 04</u>	87	07 Sep 04	27 Sep 04	16	22 Nov 04	128
3319	14	17 Jul 04	87	07 Sep 04	27 Sep 04	18	14 Jan 05	181
3321	14	<u>17 Jul 04</u>	87	07 Sep 04	27 Sep 04	18	14 Jan 05	181
3323	14	17 Jul 04	87	07 Sep 04	27 Sep 04	18	14 Jan 05	181
1327	14	18 Jul 04	88	07 Sep 04	27 Sep 04	16	22 Nov 04	127
2327	14	<u>18 Jul 04</u>	88	07 Sep 04	27 Sep 04	16	22 Nov 04	127
1339	15	<u>26 Jul 04</u>	96	07 Sep 04	27 Sep 04	16	22 Nov 04	119
2339	15	<u>26 Jul 04</u>	96	07 Sep 04	27 Sep 04	16	22 Nov 04	119
1341	15	26 Jul 04	96	07 Sep 04	27 Sep 04	16	22 Nov 04	119
2341	15	<u>26 Jul 04</u>	96	07 Sep 04	27 Sep 04	16	22 Nov 04	119
3341	15	<u>26 Jul 04</u>	96	07 Sep 04	27 Sep 04	18	14 Jan 05	172
1363	16	<u>30 Jul 04</u>	100	07 Sep 04	27 Sep 04	16	22 Nov 04	115
2363	16	<u>30 Jul 04</u>	100	07 Sep 04	27 Sep 04	16	22 Nov 04	115
1365	16	<u>30 Jul 04</u>	100	07 Sep 04	27 Sep 04	16	22 Nov 04	115
2365	10	<u>30 Jul 04</u>	100	07 Sep 04	27 Sep 04	16	22 Nov 04	115
1367	16	<u>30 Jul 04</u>	100	07 Sep 04	27 Sep 04	16	22 Nov 04	115
2307	10	<u>30 Jul 04</u>	100	07 Sep 04	27 Sep 04	10	22 NOV 04	115
1387	17	$02 \operatorname{Aug} 04$	103	07 Sep 04	27 Sep 04	10	22 NOV 04	112
1280	17	$02 \operatorname{Aug} 04$	103	07 Sep 04	27 Sep 04	10	22 Nov 04	112
2280	17	02 Aug 04	103	07 Sep 04	27 Sep 04	10	22 Nov 04	112
1201	17	02 Aug 04	103	07 Sep 04	27 Sep 04	10	22 NOV 04	112
2201	17	$02 \operatorname{Aug} 04$	103	07 Sep 04	27 Sep 04	10	22 Nov 04	112
1303	17	$02 \operatorname{Aug} 04$	103	07 Sep 04	27 Sep 04	10	22 Nov 04	112
2303	17	$02 \operatorname{Aug} 04$	103	07 Sep 04	27 Sep 04	16	22 Nov 04	112
3305	17	02 Aug 04	103	07 Sep 04	27 Sen 04	10	14 Jan 05	165
1435	10	09 Aug 04	110	07 Sep 04	27 Sen 04	17	06 Dec 04	105
2435	19	09 Aug 04	110	07 Sep 04	27 Sen 04	17	06 Dec 04	119
1437	19	09 Aug 04	110	07 Sep 04	27 Sep 04	17	06 Dec 04	110
2437	19	09 Aug 04	110	07 Sep 04	27 Sep 04	17	06 Dec 04	110
3439	19	09 Aug 04	110	07 Sen 04	27 Sen 04	18	14 Jan 05	158
1449	19	10 Aug 04	111	07 Sen 04	27 Sen 04	17	06 Dec 04	118
2449	19	10 Aug 04	111	07 Sep 04	27 Sep 04	17	06 Dec 04	118
1451	19	10 Aug 04	111	07 Sep 04	27 Sep 04	17	06 Dec 04	118

Table F.2. Critical Dates – 2004 Samples (continued)



			· ·	-						
Sample ID 03R A FIX 020-	Sample Event	Sample Date	DAA	Shipping Date	Receipt Date	Anal. Set	Anal. Date	Storage (Days)		
0511711121020-	Livent	Date		Batt	Dutt			(===j=)		
2451	19	10 Aug 04	111	07 Sep 04	27 Sep 04	17	06 Dec 04	118		
3451	19	10 Aug 04	111	07 Sep 04	27 Sep 04	18	14 Jan 05	157		
1453	19	10 Aug 04	111	07 Sep 04	27 Sep 04	17	06 Dec 04	118		
2453	19	10 Aug 04	111	07 Sep 04	27 Sep 04	17	06 Dec 04	118		
1459	20	11 Aug 04	112	07 Sep 04	27 Sep 04	17	06 Dec 04	117		
2459	20	11 Aug 04	112	07 Sep 04	27 Sep 04	17	06 Dec 04	117		
1460	20	11 Aug 04	112	07 Sep 04	27 Sep 04	18	14 Jan 05	156		
1461	20	11 Aug 04	112	07 Sep 04	27 Sep 04	17	06 Dec 04	117		
2461	20	11 Aug 04	112	07 Sep 04	27 Sep 04	17	06 Dec 04	117		
1463	20	11 Aug 04	112	07 Sep 04	27 Sep 04	17	06 Dec 04	117		
2463	20	11 Aug 04	112	07 Sep 04	27 Sep 04	17	06 Dec 04	117		
3465	20	11 Aug 04	112	07 Sep 04	27 Sep 04	18	14 Jan 05	156		
3467	20	11 Aug 04	112	07 Sep 04	27 Sep 04	18	14 Jan 05	156		
1473	20	12 Aug 04	113	07 Sep 04	27 Sep 04	17	06 Dec 04	116		
2473	20	12 Aug 04	113	07 Sep 04	27 Sep 04	17	06 Dec 04	116		
1475	20	12 Aug 04	113	07 Sep 04	27 Sep 04	17	06 Dec 04	116		
2475	20	12 Aug 04	113	07 Sep 04	27 Sep 04	17	06 Dec 04	116		
1483	21	21 Aug 04	122	07 Sep 04	27 Sep 04	17	06 Dec 04	107		
2483	21	21 Aug 04	122	07 Sep 04	27 Sep 04	17	06 Dec 04	107		
1485	21	21 Aug 04	122	07 Sep 04	27 Sep 04	17	06 Dec 04	107		
2485	21	21 Aug 04	122	07 Sep 04	27 Sep 04	17	06 Dec 04	107		
3493	21	22 Aug 04	123	07 Sep 04	27 Sep 04	18	14 Jan 05	145		
	2004 Sediment									
219	Pre-Trt	19 Apr 04	-2	17 Dec 04	06 Jan 05	19	17 Jan 05	273		
221	Pre-Trt	19 Apr 04	-2	17 Dec 04	06 Jan 05	19	17 Jan 05	273		
223	90	28 Jul 04	98	07 Sep 04	27 Sep 04	19	17 Jan 05	173		
225	90	28 Jul 04	98	07 Sep 04	27 Sep 04	19	17 Jan 05	173		

Table F.2. Critical Dates – 2004 Samples (continued)



Table I.1. Water Method Verification Results

Sample	Fortification		Percent]	Recovery	
Identification	Level (ng/mL)	Fipronil	MB46513	MB45950	MB46136
03RAFIX020-Bulk1-					
UTC-1	0.000	ND	ND	ND	ND
03RAFIX020-Bulk1-					
UTC-2	0.000	ND	ND	ND	ND
03RAFIX020-Bulk1-					
10ppt-1	0.010	78.6	93.1	72.8	80.0
03RAFIX020-Bulk1-					
10ppt-2	0.010	85.5	77.3	107.2	92.4
03RAFIX020-Bulk1-					
10ppt-3	0.010	105.0	97.3	115.4	113.9
03RAFIX020-Bulk1-					
100ppt-1	0.100	80.3	104.5	102.3	97.0
03RAFIX020-Bulk1-					
100ppt-2	0.100	84.8	104.3	106.8	101.2
03RAFIX020-Bulk1-					
100ppt-3	0.100	85.6	107.5	107.2	104.0
Mean Recov	86 ± 10	97 ± 11	102 ± 15	98 ± 11	
		n=6	n=6	n=6	n=6



5.5 Sediment Analysis

5.5.1 Method of Analysis

During the course of this study, a method of analysis was developed for the sediment samples. The acceptable recoveries observed in the sample sets demonstrated the capability of the method to extract fipronil-related residues from sediment. The procedural recoveries in this study are provided in Table VII.

Table VII: Mean Procedural Recoveries from Fortified Sediment

Sample ID	Analytical	Fortification	Percent Recovery					
Number	Set Number	Level (ppb)	Fipronil	MB46513	MB45950	MB46136		
03RAFIX020-Bulk-200 ppt-040104	10 ¹	0.20	81.6	95.8	113.9	112.2		
03RAFIX020-Bulk-200 ppt-072204	112	0.20	80.5	70.1	93.8	95.4		
03RAFIX020-100 ppt- 011705	19 ³	0.10	85.5	96.3	109.1	103.8		

¹ 2003 Set #10

² 2003 Set #11

³ 2004 Set #19

5.5.2 Analytical Results of Sediment Samples

In 2003, nine sediment samples were collected and analyzed. In 2004, four sediment samples were collected and analyzed. Sediment samples from the estuary were analyzed to evaluate the possibility of fipronil related residues. For sediment the LOQ was 0.100 ng/mL (ppb), and the MDL was 0.030 ng/mL (ppb) for fipronil and its metabolites, MB46513, MB45950, and MB46136. In 2003, sediment samples were collected from each of the three sampling points prior to treatment, and at 87 and 185 days after application, and were analyzed for fipronil-related residues in the estuary sediment. In 2004, sediment samples were collected from each of the two runoff sampling points prior to treatment, and at 98 days after application, and were analyzed for fipronil-related residues in the estuary sediment. The results are summarized in Table VIII.



:	Sampl	e		· · ·	Fipronil	MB46513	MB45950	MB46136
ID ^a	Loc. ^b	Date	DAT	Rainfall ^c	(ppb)	(ppb)	(ppb)	(ppb)
-019	A	22 May 03	2	2.5	0.341	N.D	N.D	0.018
-020	A ^d	22 May 03	2		0.342	N.D	N.D	0.017
-021	В	22 May 03	2		0.505	N.D	N.D	0.022
-022	B ^d	22 May 03	2		0.460	N.D	N.D	0.022
-023	C	22 May 03	2		0.510	N.D	N.D	0.023
-024	Cd	22 May 03	2		0.494	N.D	N.D	0.026
					<loq< td=""><td></td><td></td><td></td></loq<>			
-061	A	30 Jun 03	41	4.2	(0.010)	N.D	0.013	0.018
-063	В	30 Jun 03	41		N.D	N.D	N.D	N.D
					<loq< td=""><td></td><td><loq< td=""><td></td></loq<></td></loq<>		<loq< td=""><td></td></loq<>	
-065	C	30 Jun 03	41		(0.008)	N.D	(0.006)	0.013
					<loq< td=""><td></td><td></td><td></td></loq<>			
-067	A	02 Jul 03	43	1.7	(0.004)	N.D	N.D	N.D
					<loq< td=""><td></td><td><loq< td=""><td><loq< td=""></loq<></td></loq<></td></loq<>		<loq< td=""><td><loq< td=""></loq<></td></loq<>	<loq< td=""></loq<>
-069	B	02 Jul 03	43		(0.005)	N.D	(0.004)	(0.007)
					<loq< td=""><td></td><td></td><td></td></loq<>			
-071	<u>C</u>	02 Jul 03	43		(0.005)	N.D	N.D	N.D
					<loq< td=""><td><loq< td=""><td><loq< td=""><td><loq< td=""></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td><loq< td=""><td><loq< td=""></loq<></td></loq<></td></loq<>	<loq< td=""><td><loq< td=""></loq<></td></loq<>	<loq< td=""></loq<>
-073		06 Jul 03	47	1.5	(0.008)	(0.007)	(0.004)	(0.008)
	_				<loq< td=""><td></td><td></td><td><loq< td=""></loq<></td></loq<>			<loq< td=""></loq<>
-075	B	06 Jul 03	47		(0.009)	N.D	N.D	(0.009)
					<loq< td=""><td><loq< td=""><td></td><td></td></loq<></td></loq<>	<loq< td=""><td></td><td></td></loq<>		
-077	C	06 Jul 03	47	1	(0.007)	(0.004)	N.D	N.D

Table V: Residues in 2003 Estuary Water Samples

^a Sample ID prefix: 03RAFIX020-

^b Loc.: Sample Collection Location

[°] Rainfall: Rainfall that occurred within ≈24 hours prior to sampling

^d Duplicate sample analysis





Figure 3: Fipronil Residues and Rainfall +Irrigation for 2003



2004 Water Samples

A total of 160 estuary water samples collected in 2004 were analyzed. Samples collected and analyzed were those at designated sampling periods in addition to those collected for several days following a significant rainfall event. This ensured that any fipronil-related residue arising from runoff would be detected.

None of the 2004 water samples analyzed contained detectable residues of parent fipronil or any metabolites except for a single sample collected 112 days after application from sampling point A. That sample had residues of parent fipronil at 0.037 ppb. The duplicate sample from that interval and sampling point was also analyzed and no detectable residues were present.

The analytical results for the 2003 and 2004 estuary and sediment samples analyzed are summarized in Appendix I.

5.4.4 Analytical Results of Field Recovery Water Samples

Field recovery water samples were prepared to establish the stability of fipronil related residues during storage at the test site, subsequent transfer to the analytical facility, and further storage prior to analysis. Field recovery samples were prepared on 21 November 2003 by fortifying duplicate 100 mL samples of pre-application (bulk) estuary water with a mixture of fipronil, MB46513, MB45950, and MB46136 to yield concentrations of 0.100 ng/mL and 1.00 ng/mL. The results are summarized in Table VI and demonstrate good accountability for the analyte after storage and transfer to the analytical facility. All quantitative data for the field recovery samples are included in Appendix I.

	Fortification	 	Percent	Recovery	
Sample	Level (ng/mL)	Fipronil	MB46513	MB45950	MB46136
03RAFIX020-303 - 306	0.100	77 ±10	90 ±12	90 ±8	103 ±6
03RAFIX020-307 - 310	1.00	86 ±14	88 ±15	86 ±6	99±5

Table VI: Recoveries from Field Fortified Water Samples

5.4.5 Storage Stability

The majority of the water samples were analyzed within approximately 70 to 180 days of collection each year. A few samples were analyzed late in the study with a storage interval extending beyond 180 days. Frozen storage stability studies in water have been conducted and demonstrate the stability of fipronil and its metabolites in water over storage periods up to 25 months⁴.

88 ±15

86 ±6





Sample ID 03RAFIX020-	Sample Location	Days after Appl	Sample Date	Anal Set #	Analytical Results (ppb)			
					Fipronil	MB46513	MB45950	MB46136
2003								
201	A	-1	19 May 03	10	N.D	N.D	N.D	N.D
203	В	-1	19 May 03	10	N.D	N.D	N.D	N.D
205	C	-1	19 May 03	10	N.D	N.D	N.D	N.D
207	Α	87	15 Aug 03	10	N.D	N.D	N.D	N.D
209	В	87	15 Aug 03	11	N.D	N.D	N.D	N.D
							<loq< td=""><td></td></loq<>	
211	C	87	15 Aug 03	11	N.D	N.D	(0.035)	N.D
213	А	185	21 Nov 03	11	N.D	N.D	N.D	N.D
215	В	185	21 Nov 03	11	N.D	N.D	N.D	N.D
217	C	185	21 Nov 03	11	N.D	N.D	N.D	N.D
2004								
219	A	-2	19 Apr 04	19	N.D	N.D	N.D	N.D
221	В	-2	19 Apr 04	19	N.D	N.D	N.D	N.D
223	A	98	28 Jul 04	19	N.D	N.D	N.D	N.D
225	В	98	28 Jul 04	19	N.D	N.D	<loq (0.059)</loq 	<loq (0.043)</loq

Table VIII: Analytical Results of Sediment Samples

In 2003, the sediment samples did not contain parent fipronil residues in any of the samples. The only fipronil-related metabolite detected above the MDL but below the LOQ was MB45950 at 87 days. In 2004, the sediment samples did not contain parent fipronil residues in any of the samples. The only fipronil-related metabolite detected above the MDL but below the LOQ was MB45950 and MB46136 at 98 days. Sediment samples were not collected at 180 days in 2004 due to hurricane Ivan site destruction.

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