State of Wisconsin
Department of Natural Resources 04-05-373495

Substance Release Notification Form
Form 4400-91 (Rev. 12-01, e-form) Page 1 of 2
24-Hour Emergency Hotline Number: 1-800-943-0003

04-05-373495

Date & Military Time Of Incident: 10-19-02/ 11:10 am noticed	**Date & Military Time Reported:** 10-19-02/ 1430 PM	**Spill File #**
Person Reporting: Ray Perry	**Representing:** Proctor & Gamble	**Phone #** (920)434-2026 **Fax #** ( )
Responsible Party (RP) / Spiller: Proctor & Gamble	**RP Decision Based On:** Spill site	**Phone #** (920)434-2026 **Fax #** ( )
RP Address: 501 Eastman Avenue		**City** Green Bay **State** WI **Zip Code** 54308
RP Contact Name & Title: Ray Perry/ Environmen tal Engineer		**Phone #** (920)434-2026 **Fax #** ( )
Substance Involved: Bounty Red Ink Line Water Flexo Paper Ink (Red)	**Amount & Units Released:** unknown	**Amount & Units Recovered:** 5,000 gallons

☐ **Solid** ☐ **Semisolid** ☒ **Liquid** ☐ **Gas** **Color:** Red **Odor:** no

Exact Location Of Incident: (including street name, bldg. #, mileage, etc.) Bounty Red Ink Line Fox River, Northwest portion of the RP address. (Near the stormwater discharge pipe and the external holding tank on the northwest section of the property). Out fall 010 - N end of Property 501 Eastman Ave		**Facility Name / Property Owner:** Proctor & Gamble
☒ **City** ☐ **Village** ☐ **Township** Green Bay	**County** Brown	**Latitude/Longitude**
DNR Region: NER	**1/4 1/4 Sec T N R** ☐ **E** ☐ **W**	**Weather Conditions:** Rainy, windy

Cause Of Incident: Tote loss - Red Ink from Bounty Line - Discharge to Storm Sewer
Not found, but appears that an external holding tank to the stormwater discharge was filled with the Red Paper Ink and pumps when automatically activated emitted the substance out of the holding tank, through the discharge pipe and eventually in the Fox River.

Spilled Substance Impact To: (check **X** all that apply)

☐ Air	☐ Potential
☐ Concrete/Asphalt	☐ Potential
☐ Contained/Recovered	
☐ Groundwater	☒ Potential
☐ Private Well	☐ Potential
☐ Sanitary Sewer	☐ Potential
☐ Soil	☐ Potential
☒ Storm Sewer	☐ Potential
☒ Surface Water	☐ Potential

Name: Fox River

☐ Other:

Spill Cause/Site:
☐ Ag Coop/Food Factory
☐ Airport Facility ☐ Railroad Facility
☐ Construction, Excavation, Wrecking, Quarry, Mine
☐ Gas/Service Station/Garage/Auto Dealer/Repair Shop
☐ Hydraulic Line Break
☒ Industrial Facility ☒ Paper Mill ☐ Chemical Co.
☐ Pipeline/Terminal/Tank Farm/Oil Jobber/Wholesaler
☐ Private Property (home/farm)
☐ Public Property (city, state, church, school, etc.)
☐ Transportation Accident, Fuel Tank Spill
☐ Transportation Accident, Load Spill
☐ Utility Co. Power Generating/Transfer Facility
☐ Other:

Action Taken By Spiller:
☒ Cleanup Method:
☒ Absorbent in River
☐ Excavation
☒ Removal via Sump pit
☒ Containment
☒ Contractor Hired Onyx Spec. Services
Name: Superior

☒ Monitor
☐ No Action Needed
☐ No Action Taken
☐ Waste Destination:
☒ Other: Plugged line - Reroute to WWTP System

Injuries? ☐ Yes ☒ No If **yes** how many? **Has An Evacuation Occurred?** ☐ Yes ☒ No **Potential?** ☐ Yes ☒ No

Are There Any Resource Damages? ☐ Yes ☐ No ☒ Potential **What Kind?** Water quality

Other Agencies Notified: (check first column, if notified; check both columns, if on the scene)

☐ ☐ Fire Department	☒ ☒ Local DNR	☐ ☐ EPA
☒ ☐ Local Law Enforcement	☐ ☐ Div. Emerg. Mgt.	☐ ☐ Nat'l Resp Ctr 800-442-8802
☐ ☐ LEPC or Local Emer. Mgt.	☐ ☐ Coast Guard	☐ ☐ Chemtrec 800-424-9300
☐ ☐ Level A/Level B Team	☐ ☐ DHFS 608-258-0099	☐ ☐ Other:

Incident Commander:
Phone # ( )

Prepared By: Benjamin J. Treml	**Phone #** 492-5825	**Date:** 10-23-02	**Rpt'd To DATCP?** ☐ Yes ☒ No
Person Notified: Benjamin J. Treml	**Phone #** 492-5825	**Date:** 10-19-02	**Time:** 14:30
Investigated By: Benjamin J. Treml 10-23-02	**Sign:**	**Date:**	**Incident Closed?** ☒ Yes ☒ No **Date:** 12-19-02
Spill Coordinator Signoff:	**Date:** 12-19-02	**Transferred To:** ERP ☐ DATCP ☐ **Date:** **Case #** NO	**NFA Letter Sent?** ☐ Yes ☒ No **Spill Packet Sent?** ☒ Yes ☐ No **To:**

State of Wisconsin Substance Release Report (Cont'd)
Form 4401-91 Rev 12-01

Date and Military Time Of Incident: 10-19-02/ 11:10 AM	**Responsible Party:** Proctor & Gamble

Additional Comments : Warden Treml was notified and on scene at 3:00 pm. Warden Treml initially met with Ray Perry and Vicki Kurtz and went to the spill location along the Fox River. Kurtz indicated that she was notified of the spill from an off-duty employee fishing in the river. Kurtz stated that she could not tend to the matter until approximately 12:00 pm due to being assigned to a machine with no relief. Perry indicated that he was notified at approximately 1:30 pm that day. Kurtz also stated that when she went to the location after notification, she originally saw the red substance. Later, Kurtz did not see the substance.

At approximatley 3:00 pm, Warden Treml observed a red subtance in the Fox River, which seemed to be water saluable and contained along the shoreline with a nortwest wind. Warden Treml took photographs with issued digital camera, while Kurtz took a water sample for Proctor & Gamble. Warden Treml contacted a boater, who was able to bring Warden Treml to the discharge pipe with the boat. Warden Treml observed a faint shade of red in the River. Warden Treml notified Chronart and Superior.

Superior arrived at the scene. Green Bay Packaging (northern property border) was also adised to check their systems for the dye. Warden Treml and Superior looked at the intake and discharge at Green Bay Packaging and could not find any indications of the red dye. City Works was dispatched and advised Warden Treml where the City discharge pipe was located. Warden Treml and Superior could not find any Red dye substance.

Superio found the substance to be nuteual. It was later discovered that the red dye was contained at an external holding tank, with three automatic sub-pumps removing the substance when activated. It was then tracked back to converting for a bounty machine. MSDS sheets indicated that the substance was not hazardous to the environment. Perry and other engineers (Matt Johnson and Sharon Umentum) were not sure if converting was the source.

Superior put a boom around the red dye material and a deflection boom by Green Bay Packaging intake. Superior also left a pump station for Proctor & Gamble to retrieve the material from holding tank until the source was found.

Warden Treml left a spill packet with Pery and informed him to notify Chronart with any questions or further reoccurances.

Case Activity Report: ☐ Yes ☒ No CAR#: (Please, attach copy of all CAR and other documentation)

Enforcement Action: ☒ Yes ☐ No (Explain Below)

Warden Treml informed Perry that response to the spill and reporting the spilll to the DNR was unaccpetable. Spill was first notified at 11:10 am and Kurtz did not respond until approximatley 12:00 pm. Perry was not notified until 1:30 PM and Warden Treml was not notified until 2:30 pm. This done in the form of a verbal warning.

Chronert, Roxanne N.

From: Gerdman, David A
Sent: Thursday, December 05, 2002 3:53 PM
To: Chronert, Roxanne N.
Subject: Procter & Gamble Spill Report

Roxanne, apparently Procter & Gamble will be sending you (if they haven't sent it already) a follow-up to the October spill of red ink into the Fox River. Could you please send me a copy of their follow-up report? The address here is 2220 East County Hwy. V, Mishicot 54228. Thanks.

I'll be meeting with Keith Latva and Ray Perry on Dec. 20th at 8 at the north gate to look at the piping modifications, if you need to re-visit the site.

Dave Gerdman

December 2, 2002

Ms. Roxanne Nelezen Chronert
Spills Coordinator
P.O. Box 10448
Green Bay, WI 54307-0448

Dear Ms. Chronert;

Subject: No Further Action Report for Procter & Gamble Red Ink Spill to Fox River

Summary of Incident:
At approximately 11:10 a.m. on Sat., October 19th an off duty Procter & Gamble (P&G) employee that was fishing on the Fox River noticed a red discharge from our 010 outfall at the north end of our Fox River Plant property. This is our stormwater discharge and back up treated water discharge to the river. The employee then called this information in to his department. That department notified our Raw Materials department who in turn called the guard and requested that I be notified. At approximately 12:50 p.m. I was notified at home by the guard. I responded to the call by coming in to the plant to investigate. I went out to the 010 outfall and noticed no red discharge in the river, but the algae along the bank was stained red for about 50 yards upstream of the discharge and about 20 yards downstream of the discharge. I then checked our 001 outfall which is located about 100 yards upstream of the 010 outfall. This is our main treated water discharge and there was no red discharge or staining at this location. I also checked the flume handling treated water and stormwater from the south end of our plant – there was no red liquid in this area at that time. I then called in the spill at approximately 2:00 p.m. to the Wisconsin DNR. Warden Ben Treml called me back at about 2:30 p.m. and asked to visit the site. He arrived at about 2:45 p.m. and we proceeded to the 010 outfall. At that time a red discharge was observed. Onyx Superior Special Services, Inc. was called in to contain the spill and begin clean up operations. Onyx Superior determined that the pH was neutral for the sample obtained from the river. We then began to trace the origin of the spill and discovered that the red material was located in our north lift station sump pit. An electrician from our Raw Materials Dept. turned off the power to the lift pump to prevent further discharge of the material and Onyx called in a 5,600 gallon tanker truck to begin removing this material from the sump pit. In addition, a boom was placed in the river around our 010 outfall to attempt to contain any possible additional discharge. On call resources from our Raw Materials Dept. and a past Environmental Dept. employee were called in to assist in the problem solving.

A Bounty red ink was discovered within the ink storage spill protection area in a floor pit. We traced this line to a floor drain located near one of our paper machines elsewhere in the plant. The valve to this ink drain line was shut off at this time as a precautionary measure. This floor drain was believed to drain to a sump that pumped the water to a dissolved air flotation unit and then to Green Bay Metropolitan Sewerage District (GBMSD). However, we suspected that somehow this drain was discharging to the stormwater lift station. We began checking grated manhole covers for evidence of the red material and found none. The plan then was to pump the lift station down to the point that we could flush the floor drain at the paper machine and verify that it was somehow connected to the stormwater system. After the tanker was full the lift station still contained red liquid and another tanker was called in to continue the removal operation. This tanker was full by about 2:15 a.m. Sunday, October 20th. At this time there was still red liquid in the lift station sump pit and it was determined to resume pumping operations later that day or Monday depending on the weather.

On Monday, October 21st at about 8:00 a.m. we checked the 010 and 001 outfalls as well as the flume area and found no red liquid at any of these points. A sample of the material was sent to EnChem for a heavy

metals analysis. Later on that morning a third tanker resumed the pumping operations at the north lift station sump pit. About 2:30 p.m. we checked on the status of the pumping operations and found that the third tanker was full and the sample was clear. We then checked on the 010 outfall with Emery Coonen, Onyx Superior. There was a steady rain falling at the time. We discovered a red discharge at the 010 outfall at that time so we checked the 001 outfall which was clear. We then checked the flume area and found red liquid in the pit leading to the gravity fed 010 outfall. We contacted the Wisconsin DNR at about 3:00 p.m. This gravity fed line is connected to the line from the north lift station between the pit and the river. The grade on this line is minimal and we concluded that the red material had been trapped in the gravity fed line and forced back into this line due to back pressure from the river. We worked with Bounty converting to isolate their ink storage area and the drain pump feeding the floor drain at the paper machine was locked out to prevent any further pumping of the spilled ink. We worked with Onyx Superior to set up pumping operations at the south site beginning Monday night as soon as another tanker could be on site. In addition to the tanker a frack tank was also delivered and about 20,000 gallons was pumped out Monday night. Tuesday morning, October 22nd there was still red liquid in the south area pit and plans were set up to resume pumping this area. That evening a second frack tank was delivered and another 20,000 gallons was pumped out. Upon completion of this pumping the liquid in this pit was clear.

Tuesday, October 22nd investigations continued on the source of the spill and it was discovered that about 250 to 300 gallons of magenta red ink (MSDS sheet enclosed) had been spilled within the spill containment area in Bounty Converting. That ink had been collected and then pumped to the floor drain by the paper machine between 4:00 a.m. and 6:00 a.m. on Saturday, October 19th with the belief that it would eventually end up at GBMSD. Also on Tuesday, our in house lab determined that the color samples indicated that this was likely the same material involved in the spill, and the lab concluded that this material's pH was about neutral. We checked the MSDS sheet for the magenta red ink and there was no indication of any toxicity issues. We then began to look for ways the ink could have gotten to the stormwater sewer system. We investigated the floor drain by the paper machine and found that it most likely did not go where we initially thought and may be connected to the storm sewer system.

Wednesday, October 23rd we hired a contractor to trace the line from the floor drain near the paper machine. The contractor was unable to trace the line due to number of bends in the pipe. We then worked with Emery Coonen to obtain a safe dye to trace these lines. Emery obtained a blue dye and delivered it to the plant.

Thursday morning, October 24th we began tracing the floor drain line with the blue dye. The blue dye was poured into the floor drain and then flushed with water. The blue dye showed up in the north lift station sump pit as well as a manhole within about 2 hours confirming our suspicions that the floor drain was connected to the storm sewer system. At this time plans were put in place to plug this floor drain and other drains in the area that were also determined to be connected.

* R.N. CHRONICLE

Friday morning, October 25th we met with you and David Gerdman from the Wisconsin DNR and Emery Coonen from Onyx Superior to review our clean up, problem solving, and action plan to prevent future occurances. Later that day we received the analytical test results from EnChem (copy enclosed) confirming this material is within limits for heavy metals.

Action Plan to Prevent Future Occurances:

1. Temporarily plug the floor drains by the paper machine. Done on 10/22/02.
2. Permanently plug the floor drains by the paper machine. Done on 10/25/02.
3. Update the drawings of that area. Done on 11/08/02
4. Extend the ink drain line to a floor drain verified to go through our dissolved air flotation system and then to GBMSD. Completed by 11/01/02.
5. Confirm with GBMSD their willingness to take this ink. Completed by 11/08/02.
6. Review with the site the need to get Environmental Department resources involved immediately in the event of any future spills. Completed on 10/25/02..
7. Make arrangements with Onyx Superior to dispose of the remainder of the concentrated ink spilled in Bounty Converting. Done on 11/13/02.

8. Make arrangements with Onyx Superior and GBMSD to dispose of the 55,000 gallons of red liquid pumped out of our sump pits. Completed by 11/22/02

If you have any questions, feel free to give me a call at 430-2026.

Sincerely,

Ray D. Perry

Ray D. Perry
Site Environmental Leader

Enclosures:
Map
EnChem Analytical Results
MSDS for ink

ENCHEM ANALYTICAL REPORT

SEVERN TRENT SERVICES

STL Chicago

SEVERN TRENT LABORATORIES
ANALYTICAL REPORT

JOB NUMBER: 212910

Prepared For:

EnChem
1241 Bellevue St.
Suite 9
Green Bay, WI 54302

Project: Offloads

Attention: Laurie Woelfel

Date: 10/25/2002

Linda S Mackley
Signature

10-25-02
Date

Name: Linda S. Mackley

Title: Project Manager

E-Mail: lmackley@stl-inc.com

STL Chicago
2417 Bond Street
University Park, IL 60466

PHONE: (708) 534-5200
FAX..: (708) 534-5211

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Severn Trent Laboratories - Chicago
METALS CASE NARRATIVE

Client: EnChem
Project: OffLoads
STL Job #: 212910

Date Rec'd: 10/23/02

1. This narrative covers the Metals analysis of samples in the above Job #212910.

   Method Ref: USEPA SW-846

2. All analyses were performed within the required holding times.

3. All Initial and Continuing Calibration Verification (ICV/CCV's) were within control limits except for Lead & Zinc.
   Lead in the sample was less than the RL.
   Zinc – All Matrix QC's were within control limit. Also the Zinc in undigested sample confirmed the reported value. Therefore, reanalysis was not performed).

4. All Initial and Continuing Calibration Blanks (ICB/CCB's) were within control limits.

5. All ICP Interference Check Samples (ICSA and ICSAB) were within control limits.

6. All Laboratory Control Sample (LCS) recoveries were within the 80-120% control limits.

7. All Method blank concentrations were less than the Reporting Limits (RL) except for Zinc. (Please refer to comments on item3 above).

8. All Serial dilution analysis were within control limits.

   All Matrix spike recoveries were within the 75-125% control limits..

   All Duplicate results were within the 20% RPD control limits for sample concentration greater than 4X the RL or +\- the RL for sample concentration less than 4X the RL...

Mani S. Iyer
Metals Section Manager

10/25/02
Date

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SAMPLE INFORMATION
Date: 10/25/2002

Job Number.: 212910
Customer...: EnChem
Attn.....: Laurie Woelfel

Project Number.....: 20002620
Customer Project ID....: OFFLOADS
Project Description....: Offloads

Laboratory Sample ID	Customer Sample ID	Sample Matrix	Date Sampled	Time Sampled	Date Received	Time Received
212910-1	827384-001	Water	10/21/2002	10:50	10/23/2002	09:20

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LABORATORY TEST RESULTS

Job Number: 212910 — Date:10/25/2002

CUSTOMER: EnChem — PROJECT: OFFLOADS — ATTN: Laurie Woelfel

Customer Sample ID: 827384-001
Date Sampled.....: 10/21/2002
Time Sampled.....: 10:50
Sample Matrix.....: Water

Laboratory Sample ID: 212910-1
Date Received.....: 10/23/2002
Time Received.....: 09:20

TEST METHOD	PARAMETER/TEST DESCRIPTION	SAMPLE RESULT	Q	FLAGS	MDL	RL	DILUTION	UNITS	BATCH	DT	DATE/TIME	TECH
335.2	Cyanide, Total (Tit., Spec.)											
	Cyanide, Total	ND	U	*	0.0032	0.010	1	mg/L	66620		10/25/02 1241	rnm
150.1	pH (Water)											
	pH	7.60			0.20	0.20	1	pH Units	66398		10/23/02 1652	nrp
7470A	Mercury (CVAA)											
	Mercury	ND	U		0.000065	0.00020	1	mg/L	66509		10/24/02 1348	gok
6010B	Metals Analysis (ICAP Trace)											
	Arsenic	ND	U		0.0052	0.010	1	mg/L	66589		10/24/02 1447	lmr
	Cadmium	ND	U		0.00044	0.0020	1	mg/L	66589		10/24/02 1447	lmr
	Chromium	0.0020	B		0.0015	0.010	1	mg/L	66589		10/24/02 1447	lmr
	Copper	0.23			0.0016	0.010	1	mg/L	66589		10/24/02 1447	lmr
	Lead	ND	U		0.0029	0.0050	1	mg/L	66589		10/24/02 1447	lmr
	Nickel	ND	U		0.0019	0.010	1	mg/L	66589		10/24/02 1447	lmr
	Zinc	0.091		H*	0.010	0.020	1	mg/L	66589		10/24/02 1447	lmr

* In Description = Dry Wgt.

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LABORATORY CHRONICLE

Job Number: 212910 Date: 10/25/2002

CUSTOMER: EnChem PROJECT: OFFLOADS ATTN: Laurie Woelfel

Lab ID: 212910-1 Client ID: 827384-001 Date Recvd: 10/23/2002 Sample Date: 10/21/2002

METHOD	DESCRIPTION	RUN#	BATCH#	PREP BT #(S)	DATE/TIME ANALYZED		DILUTION
3010A	Acid Digestion (ICAP)	1	66356		10/23/2002	1600	
335.2	Cyanide, Total (Tit., Spec.)	1	66620	66620	10/25/2002	1241	
EDD	Electronic Data Deliverable	1					
7470A	Mercury (CVAA)	1	66509	66504	10/24/2002	1348	
6010B	Metals Analysis (ICAP Trace)	1	66589	66356	10/24/2002	1447	
7470/7471	SW846 Digestion (Hg)	1	66504		10/24/2002	0930	
150.1	pH (Water)	1	66398	66398	10/23/2002	1652	

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QUALITY CONTROL RESULTS

Job Number.: 212910 Report Date.: 10/25/2002

CUSTOMER: EnChem PROJECT: OFFLOADS ATTN: Laurie Woelfel

QC Type	Description	Reag. Code	Lab ID	Dilution Factor	Date	Time

Test Method.....: 6010B Analyst...: lmr
Method Description.: Metals Analysis (ICAP Trace) Batch.....: 66589

LCS	Laboratory Control Sample	M02ISPK004	66356		10/24/2002	1416

Parameter/Test Description	Units	QC Result	QC Result	True Value	Orig. Value	QC Calc.	*	Limits	F
Arsenic	mg/L	0.09490		0.10000	0.00520 U	95	%	80-120	
Cadmium	mg/L	0.04852		0.05000	0.00044 U	97	%	80-120	
Chromium	mg/L	0.20024		0.20000	0.00150 U	100	%	80-120	
Copper	mg/L	0.24397		0.25000	0.00160 U	98	%	80-120	
Lead	mg/L	0.09970		0.10000	0.00290 U	100	%	80-120	
Nickel	mg/L	0.48707		0.50000	0.00190 U	97	%	80-120	
Zinc	mg/L	0.51061		0.50000	0.10368	102	%	80-120	

* %=% REC, R=RPD, A=ABS Diff., D=% Diff.

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QUALITY CONTROL RESULTS

Job Number.: 212910 Report Date.: 10/25/2002

CUSTOMER: EnChem PROJECT: OFFLOADS ATTN: Laurie Woelfel

QC Type	Description	Reag. Code	Lab ID	Dilution Factor	Date	Time
MB	Method Blank	66356	66356		10/24/2002	1410

Test Method.....: 6010B Analyst...: lmr
Method Description.: Metals Analysis (ICAP Trace) Batch.....: 66589

Parameter/Test Description	Units	QC Result	QC Result	True Value	Orig. Value	QC Calc.	*	Limits	F
Arsenic	mg/L	0.00520 U							
Cadmium	mg/L	0.00044 U							
Chromium	mg/L	0.00150 U							
Copper	mg/L	0.00160 U							
Lead	mg/L	0.00290 U							
Nickel	mg/L	0.00190 U							
Zinc	mg/L	0.10368							H

 * %=% REC, R=RPD, A=ABS Diff., D=% Diff.

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QUALITY CONTROL RESULTS

Job Number.: 212910 Report Date.: 10/25/2002

CUSTOMER: EnChem PROJECT: OFFLOADS ATTN: Laurie Woelfel

QC Type	Description	Reag. Code	Lab ID	Dilution Factor	Date	Time
MD	Method Duplicate		212910-1		10/24/2002	1453

Test Method.....: 6010B Analyst...: lmr
Method Description.: Metals Analysis (ICAP Trace) Batch.....: 66589

Parameter/Test Description	Units	QC Result	QC Result	True Value	Orig. Value	QC Calc.	*	Limits	F
Arsenic	mg/L	0.00520 U			0.00520 U	0.00482	A	0.01000	
Cadmium	mg/L	0.00044 U			0.00044 U				
Chromium	mg/L	0.00150 U			0.00201 B	0.00128	A	0.01000	
Copper	mg/L	0.23564			0.23333	1.0	R	20.0	
Lead	mg/L	0.00290 U			0.00290 U	0.00090	A	0.00500	
Nickel	mg/L	0.00190 U			0.00190 U	0.00106	A	0.01000	
Zinc	mg/L	0.09163			0.09146	0.00017	A	0.02000	

* %=% REC, R=RPD, A=ABS Diff., D=% Diff.

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QUALITY CONTROL RESULTS

Job Number.: 212910 Report Date.: 10/25/2002

CUSTOMER: EnChem PROJECT: OFFLOADS ATTN: Laurie Woelfel

QC Type	Description	Reag. Code	Lab ID	Dilution Factor	Date	Time
MS	Matrix Spike	M02ISPK004	212910-1		10/24/2002	1532

Test Method.....: 6010B Analyst...: lmr
Method Description.: Metals Analysis (ICAP Trace) Batch.....: 66589

Parameter/Test Description	Units	QC Result	QC Result	True Value	Orig. Value	QC Calc.	*	Limits	F
Arsenic	mg/L	0.10482		0.10000	0.00520 U	105	%	75-125	
Cadmium	mg/L	0.04814		0.05000	0.00044 U	96	%	75-125	
Chromium	mg/L	0.20098		0.20000	0.00201 B	100	%	75-125	
Copper	mg/L	0.46948		0.25000	0.23333	94	%	75-125	
Lead	mg/L	0.10032		0.10000	0.00290 U	100	%	75-125	
Nickel	mg/L	0.48155		0.50000	0.00190 U	96	%	75-125	
Zinc	mg/L	0.58176		0.50000	0.09146	98	%	75-125	

 * %=% REC, R=RPD, A=ABS Diff., D=% Diff.

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QUALITY CONTROL RESULTS

Job Number.: 212910 — Report Date.: 10/25/2002

CUSTOMER: EnChem — PROJECT: OFFLOADS — ATTN: Laurie Woelfel

QC Type	Description	Reag. Code	Lab ID	Dilution Factor	Date	Time
MSD	Matrix Spike Duplicate	M021SPK004	212910-1		10/24/2002	1538

Test Method.....: 60108 — Analyst...: lmr
Method Description.: Metals Analysis (ICAP Trace) — Batch.....: 66589

Parameter/Test Description	Units	QC Result	QC Result	True Value	Orig. Value	QC Calc.	*	Limits	F
Arsenic	mg/L	0.10029	0.10482	0.10000	0.00520 U	100	%	75-125	
						4.9	R	20	
Cadmium	mg/L	0.04816	0.04814	0.05000	0.00044 U	96	%	75-125	
						0.0	R	20	
Chromium	mg/L	0.20230	0.20098	0.20000	0.00201 B	101	%	75-125	
						1.0	R	20	
Copper	mg/L	0.47642	0.46948	0.25000	0.23333	97	%	75-125	
						3.1	R	20	
Lead	mg/L	0.10080	0.10032	0.10000	0.00290 U	101	%	75-125	
						1.0	R	20	
Nickel	mg/L	0.48458	0.48155	0.50000	0.00190 U	97	%	75-125	
						1.0	R	20	
Zinc	mg/L	0.59017	0.58176	0.50000	0.09146	100	%	75-125	
						2.0	R	20	

* %=% REC, R=RPD, A=ABS Diff., D=% Diff.

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QUALITY CONTROL RESULTS

Job Number.: 212910 Report Date.: 10/25/2002

CUSTOMER: EnChem. PROJECT: OFFLOADS ATTN: Laurie Woelfel

QC Type	Description	Reag. Code	Lab ID	Dilution Factor	Date	Time
SD	Serial Dilution		212910-1		10/24/2002	1544

Test Method.....: 6010B Analyst...: lmr
Method Description.: Metals Analysis (ICAP Trace) Batch.....: 66589

Parameter/Test Description	Units	QC Result	QC Result	True Value	Orig. Value	QC Calc.	*	Limits	F
Arsenic	mg/L	0.00520 U			0.00520 U				
Cadmium	mg/L	0.00044 U			0.00044 U				
Chromium	mg/L	0.00150 U			0.00201 B				
Copper	mg/L	0.04971			0.23333	6.5	D	10.0	
Lead	mg/L	0.00290 U			0.00290 U				
Nickel	mg/L	0.00439 B			0.00190 U				
Zinc	mg/L	0.02661			0.09146				

* %=% REC, R=RPD, A=ABS Diff., D=% Diff.

STL Chicago is part of Severn Trent Laboratories, Inc.

QUALITY CONTROL RESULTS

Job Number.: 212910 Report Date.: 10/25/2002

CUSTOMER: EnChem PROJECT: OFFLOADS ATTN: Laurie Woelfel

Test Method.....: 335.2 Batch.....: 66620 Analyst...: rnm
Method Description.: Cyanide, Total (Tit., Spec.) Test Code.: CN
Parameter.....: Cyanide, Total

QC	Lab ID	Reagent	Units	QC Result		QC Result	True Value	Orig. Value		QC Calc.	F	*	Limits	Date	Time
MB	66620		mg/L	0.00320	U									10/25/2002	1236
LCS	66620	I02FSTCN2	mg/l	0.09240			0.09800	0.00320	U	94		%	80-120	10/25/2002	1236

Test Method.....: 150.1 Batch.....: 66398 Analyst...: nrp
Method Description.: pH (Water) Test Code.: PH
Parameter.....: pH

QC	Lab ID	Reagent	Units	QC Result	QC Result	True Value	Orig. Value	QC Calc.	F	*	Limits	Date	Time
LCS	66398	I02CPH7B	pH Units	6.98000		7.00000		0.02000		A	0.20000	10/23/2002	1649
LCD	66398	I02CPH7B	pH Units	6.98000		7.00000		0.02000		A	0.20000	10/23/2002	1650
MDP	212910-1		pH Units	7.62000			7.60000	0.02000		A	0.20000	10/23/2002	1653

Test Method.....: 7470A Batch.....: 66509 Analyst...: gok
Method Description.: Mercury (CVAA) Test Code.: HG
Parameter.....: Mercury

QC	Lab ID	Reagent	Units	QC Result		QC Result	True Value	Orig. Value		QC Calc.	F	*	Limits	Date	Time
MB	66504		ug/L	0.06	U									10/24/2002	1249
LCS	66504	M02ESTK010	ug/L	2.09			2.00	0.06	U	105		%	80-120	10/24/2002	1251

 * %=% REC, R=RPD, A=ABS Diff., D=% Diff.

QUALITY ASSURANCE METHODS

REFERENCES AND NOTES

Report Date: 10/25/2002

REPORT COMMENTS

1) All pages of this report are integral parts of the analytical data. Therefore, this report should be reproduced only in its entirety.
2) Soil, sediment and sludge sample results are reported on a "dry weight" basis except when analyzed for landfill disposal or incineration parameters. All other solid matrix samples are reported on an "as received" basis unless noted differently.
3) Reporting limits are adjusted for sample size used, dilutions and moisture content if applicable.
4) The test results for the noted analytical method(s) meet the requirements of NELAC. Lab Cert. ID# 100201
5) Arizona Environmental Laboratory License number AZ0603.
6) According to 40CFR Part 136.3, pH, Chlorine Residual and Dissolved Oxygen analyses are to be performed immediately after aqueous sample collection. When these parameters are not indicated as field (e.g. pH Field) they were not analyzed immediately, but as soon as possible on laboratory receipt.

Glossary of flags, qualifiers and abbreviations (any number of which may appear in the report)

Inorganic Qualifiers (Q-Column)

U	Analyte was not detected at or above the stated limit.
<	Not detected at or above the reporting limit.
J	Result is less than the RL, but greater than or equal to the method detection limit.
B	Result is less than the CRDL/RL, but greater than or equal to the IDL/MDL.
S	Result was determined by the Method of Standard Additions.
F	AFCEE: Result is less than the RL, but greater than or equal to the method detection limit.

Inorganic Flags (Flag Column)

'	ICV,CCV,ICB,CCB,ISA,ISB,CRI,CRA,MRL: Instrument related QC exceed the upper or lower control limits.
*	LCS, LCD, MD: Batch QC exceeds the upper or lower control limits.
+	MSA correlation coefficient is less than 0.995.
4	MS, MSD: The analyte present in the original sample is 4 times greater than the matrix spike concentration; therefore, control limits are not applicable.
E	SD: Serial dilution exceeds the control limits.
H	MB, EB1, EB2, EB3: Batch QC is greater than reporting limit or had a negative instrument reading lower than the absolute value of the reporting limit.
N	MS, MSD: Spike recovery exceeds the upper or lower control limits.
W	AS(GFAA) Post-digestion spike was outside 85-115% control limits.

Organic Qualifiers (Q - Column)

U	Analyte was not detected at or above the stated limit.
ND	Compound not detected.
J	Result is an estimated value below the reporting limit or a tentatively identified compound (TIC).
Q	Result was qualitatively confirmed, but not quantified.
C	Pesticide identification was confirmed by GC/MS.
Y	The chromatographic response resembles a typical fuel pattern.
Z	The chromatographic response does not resemble a typical fuel pattern.
E	Result exceeded calibration range, secondary dilution required.
F	AFCEE:Result is an estimated value below the reporting limit or a tentatively identified compound (TIC)

Organic Flags (Flags Column)

B	MB: Batch QC is greater than reporting limit.
*	LCS, LCD, ELC, ELD, CV, MS, MSD, Surrogate: Batch QC exceeds the upper or lower control limits.
'	EB1, EB2, EB3, MLE: Batch QC is greater than reporting Limit
A	Concentration exceeds the instrument calibration range
a	Concentration is below the method Reporting Limit (RL)
B	Compound was found in the blank and sample.
D	Surrogate or matrix spike recoveries were not obtained because the extract was diluted for analysis; also compounds analyzed at a dilution will be flagged with a D.
H	Alternate peak selection upon analytical review
I	Indicates the presence of an interfence, recovery is not calculated.
M	Manually integrated compound.

QUALITY ASSURANCE METHODS

REFERENCES AND NOTES

Report Date: 10/25/2002

P The lower of the two values is reported when the % difference between the results of two GC columns is greater than 25%.

Abbreviations

AS	Post Digestion Spike (GFAA Samples - See Note 1 below)
Batch	Designation given to identify a specific extraction, digestion, preparation set, or analysis set
CAP	Capillary Column CCB Continuing Calibration Blank
CCV	Continuing Calibration Verification
CF	Confirmation analysis of original
C1	Confirmation analysis of A1 or D1
C2	Confirmation analysis of A2 or D2
C3	Confirmation analysis of A3 or D3
CRA	Low Level Standard Check - GFAA; Mercury
CRI	Low Level Standard Check - ICP
CV	Calibration Verification Standard
Dil Fac	Dilution Factor - Secondary dilution analysis
D1	Dilution 1
D2	Dilution 2
D3	Dilution 3
DLFac	Detection Limit Factor
DSH	Distilled Standard - High Level
DSL	Distilled Standard - Low Level
DSM	Distilled Standard - Medium Level
EB1	Extraction Blank 1
EB2	Extraction Blank 2
FB3	DI Blank
ELC	Method Extracted LCS
ELD	Method Extracted LCD
ICAL	Initial calibration
ICB	Initial Calibration Blank
ICV	Initial Calibration Verification
IDL	Instrument Detection Limit
ISA	Interference Check Sample A - ICAP
ISB	Interference Check Sample B - ICAP
Job No.	The first six digits of the sample ID which refers to a specific client, project and sample group Lab ID An B number unique laboratory identification
LCD	Laboratory Control Standard Duplicate
LCS	Laboratory Control Standard with reagent grade water or a matrix free from the analyte of interest
MB	Method Blank or (PB) Preparation Blank
MD	Method Duplicate
MDL	Method Detection Limit
MLE	Medium Level Extraction Blank
MRL	Method Reporting Limit Standard
MSA	Method of Standard Additions
MS	Matrix Spike
MSD	Matrix Spike Duplicate
ND	Not Detected
PREPF	Preparation factor used by the Laboratory's Information Management System (LIMS)
PDS	Post Digestion Spike (ICAP)
RA	Re-analysis of original
A1	Re-analysis of D1
A2	Re-analysis of D2
A3	Re-analysis of D3
RD	Re-extraction of dilution
RE	Re-extraction of original
RC	Re-extraction Confirmation
RL	Reporting Limit
RPD	Relative Percent Difference of duplicate (unrounded) analyses
RRF	Relative Response Factor

QUALITY ASSURANCE METHODS

REFERENCES AND NOTES

Report Date: 10/25/2002

RT	Retention Time
RTW	Retention Time Window Sample ID A 9 digit number unique for each sample, the first six digits are referred as the job number
SCB	Seeded Control Blank
SD	Serial Dilution (Calculated when sample concentration exceeds 50 times the MDL)
UCB	Unseeded Control Blank
SSV	Second Source Verification Standard
SLCS	Solid Laboratory Control Standard(LCS)
PHC	pH Calibration Check LCSP pH Laboratory Control Sample
LCDP	pH Laboratory Control Sample Duplicate
MDPH	pH Sample Duplicate
MDFP	Flashpoint Sample Duplicate
LCFP	Flashpoint LCS
G1	Gelex Check Standard Range 0-1
G2	Gelex Check Standard Range 1-10
G3	Gelex Check Standard Range 10-100
G4	Gelex Check Standard Range 100-1000

Note 1: The Post Spike Designation on Batch QC for GFAA is designated with an "S" added to the current abbreviation used. EX. LCS S=LCS Post Spike (GFAA); MS$=MS Post Spike (GFAA)

Note 2: The MD calculates an absolute difference (A) when the sample concentration is less than 5 times the reporting limit. The control limit is represented as +/- the RL.

STL Chicago

EN CHEM INC.
1241 Bellevue St., Suite 9
Green Bay, WI 54302
920-469-2436
FAX 920-469-8827

212910

CHAIN OF CUSTODY

96175

Page ____ of ____

(Please Print Legibly)
Company Name: En-Chem
Branch or Location: Green Bay
Project Contact: Laurie Woelfel
Telephone: X 404
Project Number: 827384
Project Name:
Project State: WI
Sampled By (Print):

P.O. # ____ Quote # ____
Mail Report To: Laurie Woelfel
Company:
Address:
Invoice To:
Company:
Address:
Mail Invoice To:

*Preservation Codes
A=None B=HCL C=H2SO4 D=HNO3 E=EnCore F=Methanol G=NaOH
H = Sodium Bisulfate Solution I = Sodium Thiosulfate J = Other

Data Package Options - (please circle if requested)
Sample Results Only (no QC)
EPA Level II (Subject to Surcharge)
EPA Level III (Subject to Surcharge)
EPA Level IV (Subject to Surcharge)

Regulatory Program: UST, RCRA, SDWA, NPDES, CERCLA

Matrix Codes: W=Water, S=Soil, A=Air, C=Charcoal, B=Biota, Sl=Sludge

FILTERED? (YES/NO)
PRESERVATION (CODE)
Due 10-24
ANALYSES REQUESTED: Arsenic, Cadmium, Chromium, Copper, Cyanide, Lead, Mercury, Nickel, Zinc, Pb
TOTAL # OF BOTTLES SENT

LABORATORY ID (Lab Use Only)	FIELD ID	COLLECTION DATE	COLLECTION TIME	MATRIX	CLIENT COMMENTS	LAB COMMENTS (Lab Use Only)
1	827384 001	10-21	10:50			2) 1000 ml amber 1) 250 ml poly
	Due 10/24 OK per Linda M.					

Rush Turnaround Time Requested (TAT) - Prelim
(Rush TAT subject to approval/surcharge)
Date Needed:
Transmit Prelim Rush Results by (circle):
Phone Fax E-Mail
Phone #:
Fax #:
E-Mail Address:

Samples on HOLD are subject to special pricing and release of liability

Relinquished By:	Date/Time:	Received By:	Date/Time:
Annette Yanke	10-21-02		
Relinquished By:	Date/Time:	Received By:	Date/Time:
Relinquished By:	Date/Time:	Received By:	Date/Time:
Relinquished By:	Date/Time:	Received By:	Date/Time:
Relinquished By:	Date/Time:	Received By:	Date/Time:

En-Chem Project No: 827384
Sample Receipt Temp:
Sample Receipt pH (if applicable):
Cooler Custody Seals Present / Not Present:
Intact / Not Intact:

Version 2.0: 1/02

ESTIMATE #9417
October 23, 2002

We are pleased to estimate the following:

A) Alter Flite 2 Roll Angle Overhead Mold per SK-0802021-3 (Omni mold no.# OMNI-126)

Reduce the height of the socket interface by approximately .25" in the existing one cavity mold. - **$200.00**

B) Alter Flite 3 Roll Angle Overhead Mold per SK-0802022-3 (Omni mold no.# OMNI-127)

Reduce the height of the socket interface by approximately .25" in the existing one cavity mold. - **$200.00**

Pricing is subject to review of the final drawings. Alterations are to be complete approximately two (2) weeks A.R.O. or less. Tooling payment is due net upon receipt. Any questions, please feel free to contact us.

Sincerely,

Terry L. Elkins - Applications Engineer

Omni Technologies, Inc. USA
80 Brown Street
Greendale, IN USA 47025

Office Phone: (812) 539-4144
Office Fax: (812) 539-4437

E-Mail Address: terrye3@zoomtown.com

http://www.omnitechnologies.com

Chronert, Roxanne N.

From: Chronert, Roxanne N.
Sent: Tuesday, October 22, 2002 2:01 PM
To: 'Cory King - US CG'; Bougie, Cheryl; Gerdman, David A
Cc: 'Emery Coonen - Superior'; Treml, Benjamin J
Subject: P&G Red Ink Spill

I have a nasty cold and talking is tough. So I thought an e-mail would work best.

I just got off the phone with Emery Coonen, ONYX Emergency Services, 920-960-9440. So far 35,000 gallons of water have been pumped from the two pits. These are the pits that contain the storm water prior to discharge to the Fox River via outfall 010. (I am not going to try to explain the pits and how they are tied into the outfalls 001 and 010, the sump in the ink area, and distinguish where the storm and sanitary lines run. I have talked to Emery, Ben Treml, Br. Co Marine Warden, and Keith Latva and think I have a basic understanding but as soon as I write it down I will get something wrong. It is confusing enough without getting wrong information.)

P&G has someone checking the pits and outfalls every half hour 24 hours a day. If any red ink is noted they have ONYX on standby to immediately pump the water from the pits again. This should eliminate any additional release to the Fox River.

Enchem will have all the analytical completed by Thursday afternoon. Per my discussions with Emery, Ben and Keith the biggest concern with the ink may be copper. Once the analytical is complete GBMSD will make a determination as to if they can take the 35,000 gallons of pumped water.

P&G cannot find the source of the ink in the pits. The ink is stored in the center of the plant there is a containment pit around the ink. The pit is not connected to the storm sewer. P&G has all their engineers working on this issue to find the source. They are checking to make sure a storm line has not been tied into the containment pit or the sanitary lines from the Bounty area. The process waste water from the Bounty line normally goes to the sanitary sewer. They are also checking all their plumbing plans for some other explanation.

If they cannot find the source by Thursday when they get the lab results I will probably have them start putting in some borings to see if there was a historic release, a line is broken underground, etc., etc., etc. I am not requiring this yet because I am hoping they will be able to find the source and want to give them that opportunity since they have been successful in eliminating the release to the Fox River. I will try to set up a meeting on Thursday or Friday (after all the lab results are in) with P&G so I can see the site, have them explain their lines and what they have done to find the source. I will let you all know any details on the time and place. Depending on your schedule you are welcome to come.

I will keep you updated on any new information. I will be out most of tomorrow in court. If you need to contact me ASAP call my mobile phone at 920-362-2072. I am be leaving a little early today. I am going to try to sleep off this cold!

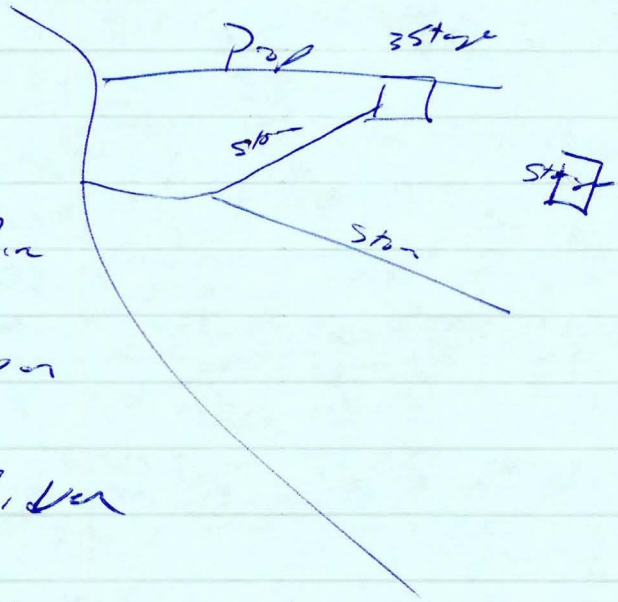
I hope to send this message to Keith Latva 430-3848 and or Ray Perry 430-2026 as soon as I receive a call back with their e-mail address.

Roxanne Nelezen Chronert
Spills Coordinator - Hydrogeologist
Wisconsin Department of Natural Resources
1125 N Military Avenue
PO Box 10448
Green Bay WI 54307-0448
Phone: 920-492-5592
Fax: 920-492-5859
roxanne.chronert@dnr.state.wi.us

Visit us on the web at www.dnr.state.wi.us/org/aw/rr

NOTE: On June 16, 2002 the e-mail format for WDNR changed to firstname.lastname@dnr.state.wi.us Messages sent to the old address (chronr) will be forwarded to the new address (roxanne.chronert) for an undetermined amount of time.

001 - from DAF other plants



010 Sample A1

Section 5

Pump Sat to Mon
15,000 gal by Mon
Clean in P.t
but saw Red @ River

Other line coming from plant gravity feed
Red back up line

300 gallon loss in Storage - lost prior to Sat.
Seal on drain line on tote
or seal in pump (Tues ?)

375 gallons per tote
10 drop / tote

4:00 Sat am turned on sump under containment. Went to 14 new Treatment System. Thought.

≈13:00 Sat. No Red coming out. Staining on skids.
≈15:00 Sat. Red coming out. Bon Superior.

Mon. - Clean in N line 48" line storm line coming

3 Dry Drains
go to storm.

WED - survey pipes

Thurs Blue Pond Dye -
in @ 14F -

Discharge

April 1998 - last change to 14F

Ink system in 8yrs ago

Temp Plug in dry end of 14F pressure area

Bladder Plug
- Wash H_2O — 14F } usually dry
- Wash H_2O — ~~Dye~~ Ink

Permanent Fix - will do as soon as get estimate + Confirm it is going

10 days - perman. plugs

Valve is lockedout

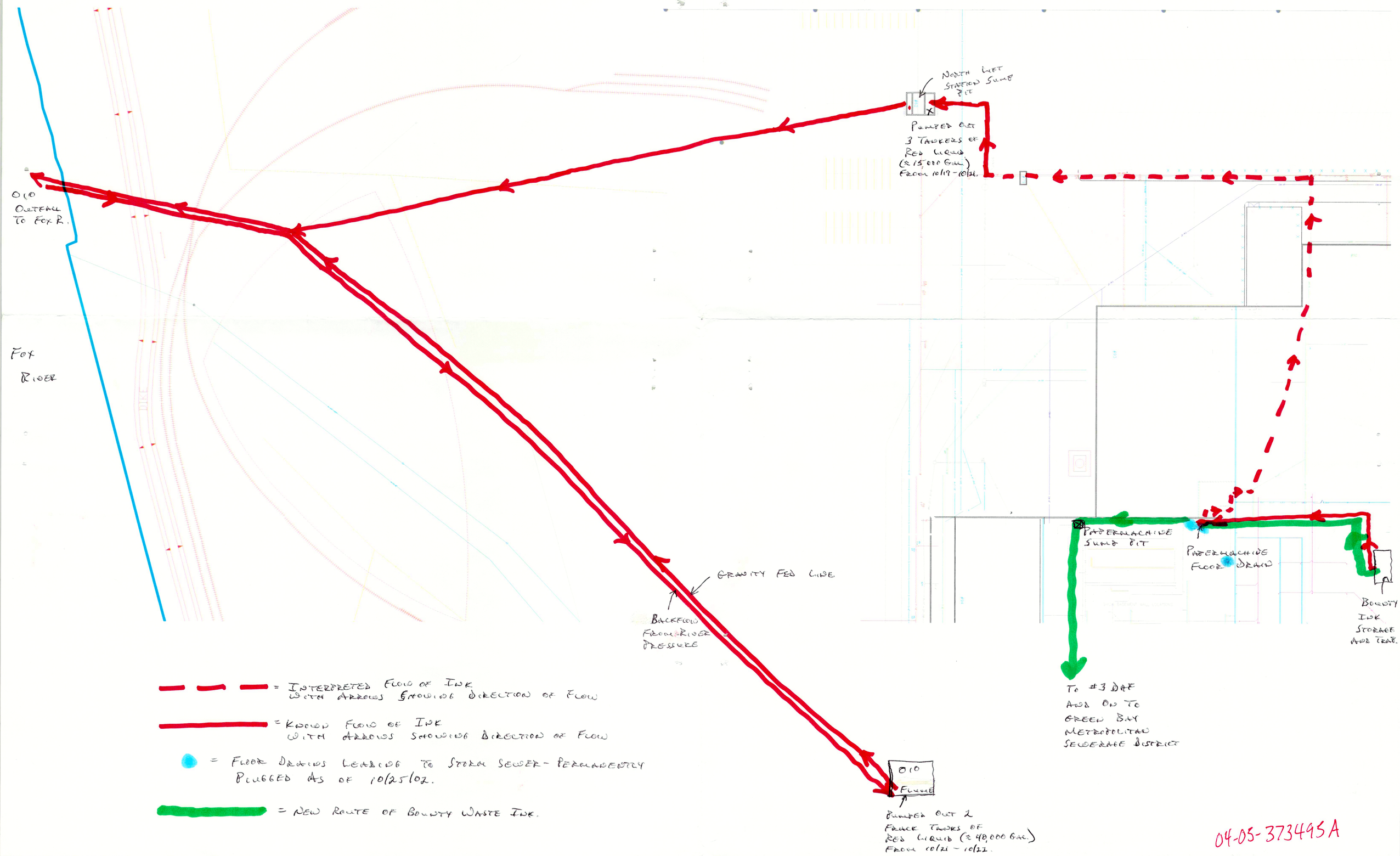
Sat afternoon - valve shut

Sump - off + Locked out

10-19-02
P & G RED INK SPILL

10-19-02
P & G RED INK SPILL

10-19-02
P & G RED INK SPILL



04-05-373495A