FLORIDA DEPARTMENT OF
Environmental Protection

Northwest District
160 W. Government Street, Suite 308
Pensacola, FL 32502

April 28, 2020

Scott Taylor, Mill Manager
International Paper
Post Office Box 87
Cantonment, FL 32533-0087
Scott.Taylor@ipaper.com

Re: Consent Order; DEP vs. International Paper Company; International Paper Company – Pensacola Mill, Permit #FL0002526; OGC File No. 19-1453; Escambia County

Attached you will find executed Consent Order 19-1453, which addresses compliance issues with facility's industrial wastewater permit, specifically chronic toxicity violations.

We wish to make it very clear that the department's expectation is that you will work as expeditiously as possible to address the ongoing compliance issues at your facility. The department expects all timelines outlined in the attached order to be met without exception. Request for waivers or extensions are unlikely to be approved. The department will be closely monitoring your progress in implementing the required actions in this order to ensure adequate progress is being made. Additionally, the department will take appropriate enforcement actions for any violations of the facility's permit or consent order.

While we recognize that an approved Site Specific Alternative Criteria would meet the regulatory requirements of the Clean Water Act and be a path forward, timely execution is key. However, it is also important to note that if compliance is not achieved, the department may be forced to deny your application for permit renewal.

If you have any questions, please contact Krista McGraw at 850-595-0612 or Krista.McGraw@floridadep.gov.

Sincerely,

Elizabeth Mullins Orr

Elizabeth Mullins Orr
Interim Director

EMO/km
Enclosure: Revised Proposed Consent Order and Exhibits 1-4
cc: Laurie McLain, International Paper (laurie.mclain@ipaper.com)
Greg Munson (GMunson@Gunster.com)

BEFORE THE STATE OF FLORIDA
DEPARTMENT OF ENVIRONMENTAL PROTECTION

STATE OF FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION

v.

INTERNATIONAL PAPER COMPANY

______________________________/

IN THE OFFICE OF THE NORTHWEST DISTRICT

OGC FILE NO. 19-1453

CONSENT ORDER

This Consent Order (Order) is entered between the State of Florida Department of Environmental Protection (Department) and International Paper Company (Respondent) to resolve certain matters at issue between the Department and Respondent. The Department finds and Respondent admits:

1. The Department is the administrative agency of the State of Florida having the power and duty to protect Florida's air and water resources and to administer and enforce the provisions of Chapter 403, Florida Statutes (F.S.), and the rules promulgated thereunder.

2. Respondent is a "person" within the meaning of Section 403.031(5), F.S.

3. Respondent is the owner and is responsible for the operation of International Paper Company's wastewater treatment plant (Facility) located at 375 Muscogee Road, Cantonment, Escambia County, Florida (Property). Respondent owns the Property on which the Facility is located.

4. The Facility processes industrial wastewater, stormwater, and pretreated sanitary wastewaters associated with a Kraft process pulp and paper mill and discharges its wastewater to waters of the State located at approximately latitude 30° 29' 6" N, longitude 87° 21' 57" W. The Facility is subject to regulation under the Department's National Pollutant Discharge Elimination System (NPDES) permitting and enforcement program.

5. Respondent operates the Facility under Department Wastewater Permit No. FL0002526 - 008-IW1S (Permit) accompanied by Consent Order 08-0358 which was entered into by Department and Respondent on March 11, 2010 (2010 Order). On September 11, 2014, Respondent timely applied to the Department for a renewal of the NPDES Permit (Renewal) and as such, pursuant to 120.60(4) F.S., the Permit has been administratively continued pending Department final agency action on the Renewal.

6. Unless otherwise noted, this Order supersedes in all respects the 2010 Order (See Paragraphs 27 and 28).

2010 Order Background

7. The 2010 Order mandated the completion of multiple complex projects to address the continuing exceedances of water quality criteria associated with Respondent's discharge to Elevenmile Creek, a Class III freshwater creek.

8. Corrective actions implemented by Respondent pursuant to the 2010 Order include but are not limited to: (i) the construction of improvements to the Facility's wastewater treatment system; (ii) the construction of an effluent transmission pipeline; (iii) the construction of a receiving wetland effluent distribution system (EDS); (iv) the implementation of surface water quality monitoring programs; and (v) establishment, maintenance, and perpetual management of a 1,188 acre conservation easement.

9. Despite Respondent's implementation of the 2010 Order's corrective actions, full and continued compliance with the Permit's final effluent limitations has not been achieved. More specifically, on nineteen occasions from 2015 to 2020, Respondent reported effluent quality monitoring results which failed to meet the Facility's permit limits for chronic whole effluent toxicity for the *Ceriodadphnia dubia* species IC_{25} (hereinafter, Exceedances) and each of which are a violation of Permit

Condition I.F.1, and Rules 62-302.530(20), 62-302.530(62), 62-4.241(1) F.A.C., and Section 403.161(1) F.S.

10. Respondent has studied and continues to study causes for the Exceedances which are summarized as follows:

Chronic Toxicity Study to Date

11. In December 2012, Respondent submitted a Toxicity Control Plan (2012 TCP) to the Department for approval. The Department subsequently approved the 2012 TCP and since that time, with appropriate Department approvals, five investigations of effluent quality have been conducted pursuant to the TCP: (1) a preliminary toxicity evaluation; (2) a Facility characterization trial; (3) a toxicity identification evaluation (TIE); (4) an effluent monitoring permit compliance point evaluation; and (5) a copper contribution assessment. The results of these studies have been previously submitted to the Department and are summarized in Exhibit 1 to this Order.

12. Since July 2019, the Facility has undertaken additional chronic bioassay investigations, including split sampling and analysis with multiple certified laboratories. Despite proper split sample collection and preservation, sample analysis produced differing results between the laboratories. Subsequent investigations into these differing results led to the hypothesis that the sensitivity of laboratory specific test cultures of *Ceriodaphnia dubia* to inorganic salt or ion composition within the effluent could produce mixed results. Sodium, potassium, manganese and calcium ions are present in all freshwaters and are essential chemicals that are required by aquatic organisms; however, increases or imbalances in these ions can result in chronic effects to sensitive organisms, such as *Ceriodaphnia dubia.*

13. A work plan (hereinafter, Salt Ion Composition Work Plan or Plan) shall be developed by Respondent to analyze the impact of the salt ion composition of Respondent's treated effluent on bioassay results. If the Salt Ion Composition Work Plan fails to confirm that Exceedances are due to

ionic imbalance, then the contribution of Emerald Coast Utility Authority's (ECUA) reclaimed water on the Respondent's chronic bioassay failures will be evaluated (hereinafter, ECUA Contribution Removal or Removal). The specific requirements of the Plan and Removal are detailed below and in Exhibit 3, but it is important to verify the salt ion composition hypothesis first because discontinuing the use of ECUA reclaimed water as source water could introduce additional variability into the verification of the salt ion composition hypothesis.

Moderating Provisions

14. Pursuant to the 2010 Order, Respondent undertook long-term monitoring in the receiving waters, in part, to determine whether to propose the adoption of Type II Site Specific Alternative Criteria (SSAC) pursuant to Rule 62-302.800(2), F.A.C., or pursue other moderating provisions if appropriate. If needed and approved, the SSAC would replace the generally applicable Class III freshwater criteria identified in Rule 62-302.530, F.A.C., for pH, specific conductance, and dissolved oxygen (DO) in the EDS.

15. Based on the long-term comprehensive monitoring described above, Respondent has demonstrated that the freshwater surface waters within the EDS do not attain the generally applicable Class III water quality criteria for DO, specific conductance, and pH, and appropriate moderating provisions may be necessary.

16. On February 24, 2020 Respondent submitted a petition for rulemaking pursuant to Rule 62-302.800(2), F.A.C., to establish SSAC for DO, specific conductance and pH in the freshwater EDS. Any proposed agency action with respect to the adoption of SSAC (or any other type of moderating provision) will be subject to administrative review under Chapter 120, F.S. Any proposed moderating provision will also be subject to the EPA's review and approval pursuant to 40 CFR 131.

Having reached a resolution of the matter Respondent and the Department mutually agree and it is

ORDERED:

17. Within 30 days of the effective date of this Order, Respondent shall pay the Department $190,000.00 in civil penalties for the Exceedances and $1,000.00 for costs and expenses incurred by the Department during the investigation of this matter and the preparation and tracking of this Order.

18. In addition to making the penalty payment of $191,000.00 as set forth in paragraph 17 above, Respondent shall implement a mitigation project, which must first be approved by the Department. The mitigation project must be either an environmental enhancement or an environmental restoration project. The Department may also consider the donation of environmentally sensitive land as the implementation of a mitigation project. The dollar value of the mitigation project shall be equivalent to at least $1,000,000.00. Respondent shall comply with all the requirements and time frames in Exhibit 2 (Mitigation Project) attached and incorporated to this Order.

Moderating Provision - Corrective Action

19. Respondent shall continue to cooperate with the Department in the processing of the SSAC Petition for Rulemaking pursuant to Rule 62-302.800(2), F.A.C. If a proposed SSAC is adopted by the Department and approved by EPA, the SSAC will be incorporated into the Renewal. In the event that either the Department denies the Petition for SSAC Rulemaking or the Department subsequently determines that a variance is the more appropriate moderating provision, the Respondent shall apply for a variance for designated parameter(s) within 30 days of written notification by the Department. If needed and approved by the Department and the EPA, the variance will be incorporated into the Renewal. The Respondent shall comply with all requests for additional information from the

Department within 30 days of the date of the request, but may receive additional time upon written request to, and approval by, the Department.

Level II WQBEL – Rule 62-650.500, F.A.C.

20. Within 30 days of the effective date of this Order, Respondent shall commence a Level II Water Quality Based Effluent Limitation (WQBEL) study to address the low DO in Tee and Wicker Lakes and a downstream portion of Elevenmile Creek. Based on the long-term comprehensive monitoring described above, Respondent has demonstrated that Tee and Wicker Lakes and a downstream portion of Elevenmile Creek do not attain the generally applicable Class III marine water quality criteria for DO. With respect to the Renewal, the purpose of the Level II WQBEL is to provide reasonable assurance that Respondent's discharge does not cause or contribute to the low DO in Tee and Wicker Lakes and a downstream portion of Elevenmile Creek. The Level II WQBEL shall also provide reasonable assurance that Respondent's discharge does not cause or contribute to exceedances of Upper Perdido Bay's new estuary specific numeric interpretation of the narrative nutrient criterion (NNC), Rule 62-302.532(1)(k)2, F.A.C., and the revised dissolved oxygen percent saturation criteria in Rule 62-302.533, F.A.C. As a condition precedent to agency action on the Renewal, based upon the Level II WQBEL, the Respondent shall provide reasonable assurance that the discharge is consistent with the antidegradation requirements of Rules 62-4.242 and 62-302.300, F.A.C. The Level II WQBEL shall comply with the applicable requirements of Rule 62-650.500, F.A.C., and be complete and submitted to the Department on or before April 15, 2021.

Interim Limits, Deadlines for Compliance with Applicable Permit Conditions and Effluent Limitations

21. Commencing immediately on the effective date of this Order and continuing until January 1, 2022 or final agency action on the Renewal, whichever occurs later, any discharge of

wastewater from Outfall #D-003 shall comply with the interim discharge limits and monitoring requirements set forth in Table 1 below:

Table 1 - Interim Limits at D-003

	Discharge Limitations				Monitoring Requirements		
Parameters	**Units**	**Monthly Avg.**	**Daily Max.**	**Other-specify**	**Frequency**	**Sample Type**	**Sample Point**
pH	standard units	NA	Range 5.0-8.5	NA	Daily	Grab or Instantaneous	EFF-3
Specific Conductance	µmhos/cm	Report	2,500	NA	3/week	Instantaneous	EFF-3
Turbidity	NTU	50	Report	NA	3/week	Grab	EFF-3
Dissolved Oxygen	mg/L	Report	Report	Report Minimum	3/week	Instantaneous	EFF-4

Salt Ion Composition Work Plan

22. Respondent shall complete the Salt Ion Composition Work Plan in accordance with the following terms:

A. Within 30 days of the effective date of this Order, Respondent shall submit results of its Laboratory protocol evaluation, where laboratory performance, qualifications and experience for multiple bioassay labs shall be evaluated to determine (a) the lab's ability to meet the pertinent testing requirements, found in EPA-821-R-02-013, and (b) the lab's capability to respond immediately by performing a Toxicity Identification Evaluation (TIE) on toxic samples, if necessary.

B. Within 45 days of the effective date of this Order, Respondent shall submit a Salt Ion Composition Plan of Study (SIC POS) to the Department for approval. Laboratories selected to participate in the study shall have certification from the Florida Department of Health Environmental Laboratory Certification Program for *Ceriodaphnia dubia*, Survival and Reproduction Test method 1002.0, found in Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, EPA-821-R-02-013. The SIC POS shall comport with requirements in Exhibit 3 attached and incorporated to this Order.

C. Within 10 days of approval by the Department, Respondent shall initiate implementation of SIC POS.

D. By November 30, 2020, Respondent shall complete the approved SIC POS.

E. By December 31, 2020, Respondent shall submit to the Department a final report of approved SIC POS findings.

23. The Department shall complete its review of the final report in paragraph 22.E by January 21, 2021. If the Department determines the final report in paragraph 22.E. demonstrates that the salt ion composition is the cause of bioassay failures, then by January 31, 2021, the Respondent shall apply for a variance from the requirements of Rules 62-4.241(1)(b) and 62-620.620(3), F.A.C., which if needed and approved would be incorporated into the Renewal.

24. In addition to the foregoing, beginning on the first day of the first full month after the effective date of this Order, Respondent shall commence monthly *Ceriodaphnia dubia* chronic toxicity testing (hereinafter, CD Test). Respondent shall demonstrate compliance by passing six consecutive months of valid passing CD Tests before January 1, 2022, or by obtaining an appropriate moderating provision. There shall be no less than 3 weeks and no more than 6 weeks between the CD Tests. Each CD test shall be conducted in accordance with the requirements as described in Exhibit 4 attached and incorporated to this Order. CD Test data shall be submitted to the Department monthly utilizing the electronic reporting system EzDMR, along with the complete bioassay laboratory report for each analysis. If a monthly CD Test for *Ceriodadphnia dubia* species is invalid, as established in EPA methods EPA-821-R-02-013, another CD Test must be initiated within 14 days after the last day of the invalid CD Test. Chronic toxicity testing with *Pimephales promelas* shall continue to be in accordance with the requirements of the Permit.

<u>Removal of ECUA Source Water</u>

25. Concurrent with the Salt Ion Composition Work Plan in Paragraphs 22 and 23, the Respondent shall propose a Plan of Study (hereinafter, ECUA POS) to remove ECUA's reclaimed water

as a source water for the Facility. Completing the ECUA POS will only be necessary if the Department determines the final report in paragraph 22.E does not demonstrate that the salt ion composition is the cause of the bioassay failures, or if Respondent fails to pass at least six consecutive months of chronic toxicity tests before January 1, 2022. The ECUA POS shall not include the use of ECUA reclaimed water as a source water for the Facility. During this time period, the effluent treatment system would be purged of any potential ECUA impact and monthly toxicity tests will continue to be conducted. Additionally, during the trial period, toxicity testing may be conducted on synthetic mixtures of ECUA and Respondent's effluent.

More specifically and additionally, the ECUA POS shall comply with the following:

A. By June 1, 2020, Respondent shall submit its ECUA POS to the Department for review and approval.

B. By January 31, 2021, Respondent shall initiate the Department approved ECUA POS if necessary (assuming Department approval of the POS no later than August 1, 2020).

C. By October 1, 2021, Respondent shall complete the ECUA POS.

D. By November 1, 2021, Respondent shall submit a final report of the ECUA POS findings. Respondent shall include a plan for corrective action in the final report for Department review and approval.

26. By January 1, 2022, Respondent shall complete corrective actions contained in the Department approved ECUA Reclaimed Water Study findings report referenced in Paragraph 25. D. above, and demonstrate compliance with Permit Condition I.F.1, and Rules 62-302.530(20), 62-302.530(62), 62-4.241(1) F.A.C.

27. Respondent shall continue to comply with the corrective actions with respect to Long-Term Monitoring and Land Management Practices as required by paragraph 14(d) of the 2010 Order.

28. Respondent shall continue to comply with the corrective actions with respect to the Conservation Easement as required by paragraph 14(e) of the 2010 Order.

29. Respondent shall pay the Department stipulated penalties as follows:

a. $10,000.00 for each valid CD Test as required by paragraph 24 that is not in compliance with the limit as listed on Exhibit 4 attached and incorporated to this Order, starting April 1, 2020 until this Order is closed.

b. $5,000 per day for noncompliance with the deadlines for corrective actions in paragraphs 18 through 23, and 25 through 28.

The Department may demand stipulated penalties at any time after violations occur. Respondent shall pay stipulated penalties owed within 30 days of the Department's issuance of written demand for payment and shall do so as further described in paragraphs 33 and 34 below. Nothing in this paragraph shall prevent the Department from filing suit to specifically enforce any terms of this Order.

30. Nothing in this Order shall prevent the Department from filing suit to specifically enforce any terms of this Order. Any stipulated penalties assessed under paragraph 29 shall be in addition to the civil penalties, mitigation project, Department costs and expenses agreed to in paragraphs 17 and 18 of this Order.

31. Every calendar quarter after the effective date of this Order, and continuing until all corrective actions have been completed, Respondent shall submit in writing to the Department a report containing information concerning the status and progress of projects being completed under this Order, information as to compliance or noncompliance with the applicable requirements of this Order including construction requirements and effluent limitations, and any reasons for noncompliance. These reports shall also include a projection of the work to be performed pursuant to this Order during the 12-month period which will follow the report. Respondent shall submit the reports to the Department within 30 days of the end of each quarter. Quarterly reports shall be due as follows: January-March reports shall be submitted by April 30; April-June reports shall be submitted by July 30; July-September reports shall

be submitted by October 30; and October-December reports shall be submitted by January 1 of the following year.

32. Within 90 days of the effective date of this Order, Respondent shall submit a written estimate of the total cost of the corrective actions required by this Order to the Department. The written estimate shall identify the information the Respondent relied upon to provide the estimate.

33. Respondent shall make all payments required by this Order by cashier's check, money order or on-line payment. Cashier's check or money order shall be made payable to the "Department of Environmental Protection" and shall include both the OGC number assigned to this Order and the notation "Water Quality Assurance Trust Fund." Online payments by e-check can be made by going to the DEP Business Portal at: http://www.fldepportal.com/go/pay/. It will take a number of days after this Order becomes final, effective and filed with the Clerk of the Department before online payment option is available.

34. Except as otherwise provided, all submittals and payments required by this Order shall be sent to Assistant District Director, Department of Environmental Protection, Northwest District Office 160 West Government Street Suite 308 Pensacola, FL 32502.

35. Respondent shall allow all authorized representatives of the Department access to the Facility and the Property at reasonable times for the purpose of determining compliance with the terms of this Order and the rules and statutes administered by the Department.

36. In the event of a sale or conveyance of the Facility or of the Property upon which the Facility is located, if all of the requirements of this Order have not been fully satisfied, Respondent shall, at least 30 days prior to the sale or conveyance of the Facility or Property, (a) notify the Department of such sale or conveyance, (b) provide the name and address of the purchaser, operator, or person(s) in control of the Facility, and (c) provide a copy of this Order with all attachments to the purchaser,

operator, or person(s) in control of the Facility. The sale or conveyance of the Facility or the Property does not relieve Respondent of the obligations imposed in this Order.

37. If any event occurs which causes delay or the reasonable likelihood of delay in complying with the requirements of this Order, Respondent shall have the burden of proving the delay was or will be caused by circumstances beyond the reasonable control of Respondent and could not have been or cannot be overcome by Respondent's due diligence. Neither economic circumstances nor the failure of a contractor, subcontractor, materialman, or other agent (collectively referred to as "contractor") to whom responsibility for performance is delegated to meet contractually imposed deadlines shall be considered circumstances beyond the control of Respondent (unless the cause of the contractor's late performance was also beyond the contractor's control). Administrative or judicial challenges by third parties unrelated to Respondent are considered circumstances beyond the control of Respondent. Upon occurrence of an event causing delay, or upon becoming aware of a potential for delay, Respondent shall notify the Department by the next working day and shall, within seven calendar days notify the Department in writing of (a) the anticipated length and cause of the delay, (b) the measures taken or to be taken to prevent or minimize the delay, and (c) the timetable by which Respondent intends to implement these measures. If the parties can agree that the delay or anticipated delay has been or will be caused by circumstances beyond the reasonable control of Respondent, the time for performance hereunder shall be extended. The agreement to extend compliance must identify the provision or provisions extended, the new compliance date or dates, and the additional measures Respondent must take to avoid or minimize the delay, if any. Failure of Respondent to comply with the notice requirements of this paragraph in a timely manner constitutes a waiver of Respondent's right to request an extension of time for compliance for those circumstances

38. This Order is a settlement of the Department's civil and administrative authority arising under Florida law to resolve the matters addressed herein. This Order is not a settlement of any criminal liabilities which may arise under Florida law, nor is it a settlement of any violation which may be prosecuted criminally or civilly under federal law. Entry of this Order does not relieve Respondent of the need to comply with applicable federal, state, or local laws, rules, or ordinances.

39. The Department hereby expressly reserves the right to initiate appropriate legal action to address any violations of statutes or rules administered by the Department that are not specifically resolved by this Order.

40. Respondent is fully aware that a violation of the terms of this Order for which there is no stipulated penalty may subject Respondent to judicial imposition of damages, civil penalties up to $10,000.00 per day per violation, and criminal penalties.

41. Respondent acknowledges and waives its right to an administrative hearing pursuant to sections 120.569 and 120.57, F.S., on the terms of this Order. Respondent also acknowledges and waives its right to appeal the terms of this Order pursuant to section 120.68, F.S. Respondent retains all rights to participate in any and all proceedings initiated by third parties.

42. Electronic signatures or other versions of the parties' signatures, such as .pdf or facsimile, shall be valid and have the same force and effect as originals. No modifications of the terms of this Order will be effective until reduced to writing, executed by both Respondent and the Department, and filed with the clerk of the Department.

43. The terms and conditions set forth in this Order may be enforced in a court of competent jurisdiction pursuant to sections 120.69 and 403.121, F.S. Failure to comply with the terms of this Order constitutes a violation of section 403.161(1)(b), F.S.

44. This Order is a final order of the Department pursuant to section 120.52(7), F.S., and it is final and effective on the date filed with the Clerk of the Department unless a Petition for Administrative Hearing is filed in accordance with Chapter 120, F.S. Upon the timely filing of a petition, this Order will not be effective until further order of the Department.

45. Respondent shall publish the following notice in a newspaper of daily circulation in Escambia County, Florida. The notice shall be published one time only within 30 days of the effective date of the Order. Respondent shall provide a certified copy of the published notice to the Department within 10 days of publication.

STATE OF FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION

NOTICE OF CONSENT ORDER

The Department of Environmental Protection ("Department") gives notice of agency action of entering a Consent Order with INTERNATIONAL PAPER COMPANY pursuant to section 120.57(4), Florida Statutes. The Consent Order addresses the industrial wastewater effluent quality issues at International Paper Company located at 375 Muscogee Road, Cantonment, Escambia County, Florida. The Consent Order is available for public inspection on the department's website at: https://floridadep.gov/northwest/nw-compliance-assurance/content/international-paper-npdes-wastewater-permit.

Persons who are not parties to this Consent Order, but whose substantial interests are affected by it, have a right to petition for an administrative hearing under sections 120.569 and 120.57, Florida Statutes. Because the administrative hearing process is designed to formulate final agency action, the filing of a petition concerning this Consent Order means that the Department's final action may be different from the position it has taken in the Consent Order.

The petition for administrative hearing must contain all the following information:
The OGC Number assigned to this Consent Order;

a. The name, address, and telephone number of each petitioner; the name, address, and telephone number of the petitioner's representative, if any, which shall be the address for service purposes during the proceeding;

b. An explanation of how the petitioner's substantial interests will be affected by the Consent Order;

c. A statement of when and how the petitioner received notice of the Consent Order;

d. Either a statement of all material facts disputed by the petitioner or a statement that the petitioner does not dispute any material facts;

e. A statement of the specific facts the petitioner contends warrant reversal or modification of the Consent Order;

f. A statement of the rules or statutes the petitioner contends require reversal or modification of the Consent Order; and

g. A statement of the relief sought by the petitioner, stating precisely the action petitioner wishes the Department to take with respect to the Consent Order.

The petition must be filed (received) at the Department's Office of General Counsel, 3900 Commonwealth Boulevard, MS# 35, Tallahassee, Florida 323993000 or via electronic correspondence at Agency_Clerk@dep.state.fl.us within 21 days of receipt of this notice. A copy of the petition must also be mailed at the time of filing to the District Office at Northwest District Office 160 W Government Street Suite 308 Pensacola, FL 32502. Failure to file a petition within the 21-day period constitutes a person's waiver of the right to request an administrative hearing and to participate as a party to this proceeding under sections 120.569 and 120.57, Florida Statutes. Before the deadline for filing a petition, a person whose substantial interests are affected by this Consent Order may choose to pursue mediation as an alternative remedy under section 120.573, Florida Statutes. Choosing mediation will not adversely affect such person's right to request an administrative hearing if mediation does not result in a settlement. Additional information about mediation is provided in section 120.573, Florida Statutes and Rule 62-110.106(12), Florida Administrative Code.

46. Rules referenced in this Order are available at http://www.dep.state.fl.us/legal/Rules/rulelist.htm.

FOR RESPONDENT:

INTERNATIONAL PAPER COMPANY

By: Scott Taylor

Name: Scott Taylor

Title: International Paper Pensacola Mill Manager

DONE AND ORDERED this 28th day of April, 2020, in Pensacola, Florida.

STATE OF FLORIDA DEPARTMENT
OF ENVIRONMENTAL PROTECTION

Elizabeth Mullins Orr

Elizabeth Mullins Orr
Interim District Director
Northwest District

Filed, on this date, pursuant to section 120.52, F.S., with the designated Department Clerk, receipt of which is hereby acknowledged.

Mary H. Curle

Clerk

April 28, 2020

Date

Copies furnished to:
Lea Crandall, Agency Clerk
Mail Station 35

Exhibit 1 to Consent Order OGC Case No.19-1453

<u>Previous Bioassay Characterization Studies</u>

- **Preliminary toxicity evaluation (2013)**: A total of 23 constituents were identified as potentially influencing WET exceedances in final Mill effluent. Target constituents were identified based on the chemical analyses of effluent and an exceedance above an effects-based screening concentration. Of the 15 constituents observed in the Mill effluents, target constituents with toxicity quotients above 100 included didecyl dimethyl ammonium chloride (DDAC), hydroquinone, sulfide, nonylphenol ethoxylate (NPE), and total quaternary amines. Target constituents identified in reclaim water include chlorine, copper, bis(2-ethylhexyl)phthalate, acrolein, xylenes, total phenols, and total AOX.
- **Mill Trial (2013):** The Mill trial characterized the relative contribution of Mill process waste streams and reclaim water to constituents in Mill effluent. The Mill trial identified a refined list of constituents potentially associated with WET exceedances. A summary is presented below:
    - Paired samples for chronic WET testing and laboratory chemical analysis were collected across reclaim water flows ranging from 0 to 3,600 gallons per minute (GPM). Non-metric multidimensional scaling (NMDS) was used to identify patterns in the chemical composition of the chronic toxicity tests.
    - Constituents specific to the reclaim water include acrolein and total phenols, which exhibited a statistically significant increase in Mill effluent as the reclaim water was introduced to the Mill. Constituents specific to the Mill include total long chain fatty acids (LCFAs), linoleic acid, hydroquinone, and DDAC. The LCFAs were also observed in the ECUA effluents, albeit at lower concentrations.
    - The Mill trial results suggest that there is a critical combination of constituents in Mill and reclaim water that may contribute to WET. Also, dilution effects from the reclaim water could also influence toxicity:
        - Mill bioassay samples that passed in April were characterized by elevated LCFA and hydroquinone, with low total phenol and acrolein concentrations. Mill samples that passed in May were characterized by low LCFAs and hydroquinones, with high levels of total phenols and acrolein. Samples that did not attain the reproductive endpoint had moderate combinations of LCFAs, hydroquinones, total phenols, and acrolein.
- **Toxicity identification evaluation (2014)**: A TIE was conducted on samples of wastewater effluent and receiving water. General TIE conclusions are presented below:
    - WET exceedances at the Mill are intermittent and a preliminary correlation analysis suggests that the exceedances may be (at least in part) associated with individual LCFA fractions.
    - Spiking study results suggest that the toxic effects of the LCFAs can potentially be masked by other constituents within the effluent.
    - Constituents in reclaim water may potentially influence the toxicity of the Mill effluent. The influences could be synergistic or antagonistic.
    - Constituents potentially experience mixing and attenuation before reaching the EDS, and within the EDS itself.

- Chronic toxicity was not observed within the EDS. Both acute and chronic toxicity were observed in natural background waters on the EDS project site (resulting from an acidic pH).
- The macroinvertebrate community in the EDS has not been adversely affected by the constituents in the effluent.

- **Effluent monitoring permit compliance point evaluation (2016-2018)**: The study evaluated moving the current effluent compliance point from the final effluent mixbox (FEMB) at EFF-3, to the point of discharge in the receiving water (EFF-4).The purpose of the study was to (1) support modifying the collection of WET samples to more accurately reflect the August 2011 upgrades to the WWTS, and (2) assess whether regulatory relief from WET requirements could be achieved. The analytical data collected at EFF-4 suggested that moving the effluent compliance point will not achieve the desired regulatory relief:
  - There was no statistically significant reduction of key constituents at EFF-4.
  - WET was observed at EFF-4, potentially associated with phenols and specific conductivity.

  It was concluded that before effective WET reduction measures can be identified, the causes of toxicity in the reclaim water must be addressed. Currently, measures are being adopted by others to improve reclaim water quality.

- **Copper Study (2019):** The objectives of the copper assessment are to: (1) characterize sources of copper; (2) characterize the ability of the Mill WWTS to reduce total and dissolved copper concentrations; (3) characterize the ability of the WWTS to eliminate WET failures; and (4) identify aspects of the WWTS that warrant further evaluation. The copper study conclusions regarding chronic WET are presented below:
  - Intermittent chronic WET test exceedances were observed at outfall EFF-003 during the study period. Copper is not causing WET at EFF-003 because complexing agents are present in the wastewater that reduce bioavailability/toxicity.
  - Only some of the substances with the potential to contribute to WET are being removed with primary and secondary treatment. The residual toxicity may be caused by non-biodegradable, recalcitrant substances that pass through the WWTS.

Attachment 1: Goodfellow WL, Ausley LW, Burton DT, Denton DL, Dorn, PB, Grothe, DR, Heber, MA, Norberg-King TJ, and Rodgers, Jr. JH. 2000. Major ion toxicity in effluents: A review with permitting recommendations. Environmental Toxicology and Chemistry 19:175-182.

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Annual Review

MAJOR ION TOXICITY IN EFFLUENTS: A REVIEW WITH PERMITTING RECOMMENDATIONS

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Abstract—Effluent toxicity testing methods have been well defined, but for the most part, these methods do not attempt to segregate the effects of active ionic concentrations and ion imbalances upon test and species performances. The role of various total dissolved solids in effluents on regulatory compliance has emerged during the last few years and has caused confusion in technical assessment and in permitting and compliance issues. This paper assesses the issue of ionic strength and ion imbalance, provides a brief summary of applicable data, presents several case studies demonstrating successful tools to address toxicity resulting from salinity and ion imbalance, and provides recommendations for regulatory and compliance options to manage discharges with salinity/ion imbalance issues. Effluent toxicity resulting from inorganic ion imbalance and the ion concentration of the effluent is pervasive in permitted discharge from many industrial process and municipal discharges where process streams are concentrated, adjusted, or modified. This paper discusses procedures that use weight-of-evidence approaches to identify ion imbalance toxicity, including direct measurement, predictive toxicity models for freshwater, exchange resins, mock effluents, and ion imbalance toxicity with tolerant/ susceptible text species. Cost-effective waste treatment control options for a facility whose effluent is toxic because of total dissolved solids (TDS) or because of specific ion(s) are scarce at best. Depending on the discharge situation, TDS toxicity may not be viewed with the same level of concern as other, more traditional, toxicants. These discharge situations often do not require the conservative safety factors required by other toxicants. Selection of the alternative regulatory solutions discussed in this paper may be beneficial, especially because they do not require potentially expensive or high-energy–using treatment options that may be ineffective control options. The information presented is intended to provide a better understanding of the role of ion imbalance in aquatic toxicity testing and to provide various recommendations that should be considered in addressing these issues.

Keywords—Whole effluent testing Ion imbalance Salinity Total dissolved solids

INTRODUCTION

Ion imbalance may result from the composition and con- centration of anions and cations making up salinity. Effluent toxicity testing methods have been well defined, but these methods generally do not attempt to segregate the effects of salinity or ionic strength upon test and species performance. This issue has emerged during the last few years and has caused confusion in technical assessment and in permitting and compliance issues. This paper assesses the issue of ion imbalance, provides a brief summary of applicable data, presents several successful technical tools to address toxicity resulting from effluent salinity and ion imbalance, and discusses regulatory and compliance options to manage discharges with salinity/ ion imbalance issues.

Toxicity resulting from ion effects on a test organism is a complex issue in technical assessment and permitting/compliance. Specifically, some effluents are toxic because of imbalances in the ion environment to which the test organisms are exposed. Such imbalances may be the result of mixing

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effluents and receiving waters, yielding a testing solution that is physiologically intolerable for the test species. Dorn and Rodgers [1] and Dorn et al. [2], who identified calcium in a process stream causing toxicity to the mysid shrimp *Mysidopsis bahia,* showed the phenomenon of effluent toxicity due to ion imbalance. Although few literature sources compile the general toxicological responses of various aquatic organisms to anions and cations, a good listing of toxicity of common ions to freshwater and marine organisms can be found in Amer- ican Petroleum Institute [3].

Currently, it is debated whether salinity should be considered a toxicant in all situations. It should also be noted that ion imbalance is accounted not only for high levels of ions but also for situations in which the effluent does not contain sufficient ions (e.g., condensate discharges) and is functionally de-ionized. Toxicity associated with ion imbalance of the effluent occurs when the ion concentrations and molar ratios of the effluent exceed or do not meet the physiological tolerance range of the selected test organism.

This manuscript is an outcome of extensive discussions among the authors, who represent the regulated, regulatory, and academic sectors with expertise in whole effluent toxicity (WET) testing, and incorporates data and other information from the scientific community that addresses this issue. Specific examples from the literature are presented, as are experiences in addressing various aspects of ions in aquatic toxicity testing, to provide further insight into the problem for both the regulated and regulatory communities. The information presented is intended to provide a better understanding of the role of ion imbalance in aquatic toxicity testing and to provide various recommendations that should be considered in ad dressing these issues.

ion. For *C. dubia* and *D. magna,* the toxicity of Cl^- and SO_4^{2-} was reduced in solutions that were enriched with more than one cation. In preliminary applications of the salinity toxicity models to field-collected samples, a high degree of accuracy was observed for the *C. dubia* model, whereas the *D. magna* and fathead minnow models tended to overpredict ion toxicity. For freshwater matrices, a salinity/toxicity relationship pro- gram has been developed [8] based on tests conducted using moderately hard water.

For marine organisms, the major ions influencing toxicity are Cl^-, SO_4^{2-}, HCO $^-$, Br^{2-}, H BO $^-$, F^-, Na^+, Mg^{2+}, Ca^{2+},

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REVIEW OF TOXICITY OF COMMON IONS

The potential effects of the concentrations of common ions and of the combination of ions must be identified; furthermore, we must understand that ion concentrations can increase to levels that are unacceptable to aquatic organisms. Total dis- solved solids (TDS), conductivity, and salinity are often used as measures for the common ions in freshwater. The correlation between increasing TDS or conductivity and toxicity may vary with ionic composition and therefore may not be the best pre- dictor of toxicity. Because cations or anions are not present as individual constituents but rather are in combination with other ions, the individual toxicity of a cation or anion may be masked or inseparably affected by the associated anion or cation. Therefore, effects in effluents, receiving waters, or pro- duced waters must be considered as caused by combinations of ions, with an understanding of the effects of the various ions. In addition, for marine waters, the deficiency of common ions can be as detrimental to aquatic organisms as excessive ions. This deficiency of common ions is also true for freshwater species, but deficiency is not as likely to occur unless there is a total void of ions (e.g., condensate effluents).

In WET testing, it is important to consider the ion (or salinity) tolerance of the test organism relative to receiving water considerations. In general terms, it is more important to match the salinity tolerance for chronic versus acute toxicity testing, given the fact that the growth and reproductive end- points are more sensitive to energy-taxing requirements of osmoregulation than is the acute endpoint of survival. The

U.S. Environmental Protection Agency (U.S. EPA) manuals for the performance of WET tests provide appropriate salinity tolerances for the marine tests species [4--6].

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The effects of common ions on three of the most common species used in the freshwater WET testing program in the United States have been studied [3,7]. Studies have shown that Na^+ is not generally a major contributor to aquatic toxicity and that the associated anion, Cl^-, is more toxic than Na^+ [7], although Na^+ has a role in ion deficiency–related toxicity. Sulfate is less toxic than Cl^- and is associated with several cations, including the compounds Na_2SO_4, $CaSO_4$, and $MgSO_4$. Effects of Ca^{2+}, Mg^{2+}, and K^+ are possible, yet only Na_2SO_4 was considered [7]. The toxicity of more than 2,900 ion so- lutions was evaluated using the daphnids *Ceriodaphnia dubia* and *Daphnia magna* and fathead minnows (*Pimephales pro- melas*). The relative ion toxicity was K^+ > HCO $^-$ = Mg^{2+}

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> Cl^- > SO $^{2-}$. Sodium and calcium were not significant variables in regression analysis. For all of the salts tested, *C. dubia* was the most sensitive, compared with *D. magna* and

P. promelas. For certain salts, such as $CaSO_4$, toxicity to the three species is very similar, whereas for others (i.e., NaCl), the difference was great. In addition, the toxicity of Na^+ and Ca^{2+} salts was primarily attributable to the corresponding an-

K^+, and Sr^{2+}. Marine data are sparse in comparison with the amount of data available from freshwater testing, with most of the toxicity information on the same species. Dorn and Rodgers [1], Dorn et al. [2], and Ward [9] have assessed the toxicity of calcium to *M. bahia.* McCulloch et al. [10] and Douglas and Horne [11] evaluated the major essential ions and their associated toxicity to *M. bahia.* Tietge and Mount [12] and Pillard and Evans [13] have investigated the major trace ion toxicity to the saltwater organisms *M. bahia, Cyprinodon variegatus,* and *Menidia beryllina.* Dorn and Rodgers [1] test- ed *M. bahia* and *C. variegatus* to determine the LC50 for Ca^{2+}. Three species of marine organisms were acclimated to seawater with a salinity of 25‰ in culture, and each species was subsequently tested in waters of salinities of near zero to as high as 80‰. *Mysidopsis bahia* showed significant mor- talities at salinities greater than 50‰, and survival was reduced both in solutions with deficient salinity and in those with ex- cessive salinity. *Menidia beryllina* was less tolerant of saline solutions than was *C. variegatus,* but the results were highly variable. *Cyprinodon variegatus* was the most tolerant of low and high ion concentrations, with a high rate of survival in some tests at salinities of 1‰ to 80‰. Calcium and potassium caused significant mortality when present in very low (defi- cient) and high (excess) concentrations. A recent report [14] provides models for predicting toxicity to marine organisms, and a beta version (GRI-MSTR) of software that assists in identifying the potential toxicity and marine waters to three species of marine organisms that are similar to the freshwater matrices salinity/toxicity program is being tested [8].

EXAMPLES OF DISCHARGES CAUSING ION IMBALANCE

One can identify a variety of wastewater-generating pro- cesses that may create ion balance problems for aquatic or- ganisms. Extremes in ion concentrations in wastewater gen- erally arise from one or more of four paths: direct addition of chemicals to water (e.g., salt, lime, alum) in production or treatment processes; ion concentration by other chemical/phys- ical manipulation (e.g., pH modification, reverse osmosis, dis- tillation/evaporation); discharge of wastes initially high in ion content (e.g., seawater, coproduced groundwater/mine dewa- tering, contaminated groundwater remediation); or discharge from facilities with extremely low total dissolved solids in water wastes.

Table 1 lists various discharge types and examples where ion content and balance may influence the WET testing pro- gram. These examples are not meant to be comprehensive but rather to provide the reader with various situations in which ion discharges may require additional consideration as part of the National Pollutant Discharge Elimination System (NPDES) permit.

Table 1. Selected examples of discharges that cause ion imbalance

Discharge types Discharge examples Reported ion concentrations

Reverse osmosis systems Desalination facilities produce reject
water that concentrates salts present in seawater or other natural sources
Hydrostatic testing Pipelines and petroleum storage termi- nals require testing by pressurization with water
Food processing Salinity or pickling process Water recycle
Oil and gas production/refining High-salinity groundwater is often co-
produced with extraction of subsur- face crude oil and natural gas
Various refinery processes
Coal bed methane degasification (CBMD)
Mining operations/ore processing Ground water leachates from forma-
tions/mining operations
Ore processing/washing of mineral product
Textile operations Dying operations use large volumes of water and a variety of salts
Water recycle

pH modifications Many wastewater treatment processes include pH modifications that pro- duce high total dissolved solids (TDS) in waste streams
Metal cleaning and plating processes use significant pH modification

Agricultural runoff Arid western portions of the United States often use highly mineralized groundwater for crops irrigation
Evaporation of irrigation waters and other surface waters
Pharmaceutical Pharmaceutical chemical products have been noted to produce high TDS in effluent
Chemical production Chemical processing and product recov-
ery can yield high total TDS in efflu- ent
pH modification of process streams and waste streams
Groundwater remediation Remediation systems can discharge
high TDS in wastewater
pH modification in treatment process can increase TDS in effluent

Publicly owned treatment works Heavily commercial/industrial signifi-
cant users often increase TDS con- centration of wastewater

Nine facilities in Florida observed TDS concentrations of 10– 20‰ [29]

Depending upon the water source, TDS concentrations reported from 1 to >13‰ [3]

A cucumber pickling facility observed with effluent concentra- tions of 1.9 to 14.6‰ (L. Ausley, personal communication)
TDS levels in produced waters can range from 1.7 to 200‰ [30,31]
TDS levels from a CBMD facility observed at 1–10‰ [32] Conductivity of petroleum refinery effluents can vary from
2,940 μS/cm to 3,810 mS/cm [33]

Cl^- concentrations from a coal mine remained relatively stable, with a mean of 18.7‰ [33]
A chromium ore processor reported with Cl^- concentrations of 4.5 to 5‰ [33]
One facility reported that dying bath contained TDS levels of 120‰ (L. Ausley, personal communication)
Another similar facility reports effluents with range of conduc- tivity from 6,000 to 9,000 μS/cm [29]
Textile facilities frequently need to modify surface of fibers through pH modification with subsequent neutralization. Con- ductivity in the range of 2,000 to 3,000 mS/cm are not un- common in this wastewater [29]
A metal plating facility has reported conductivity as high as 8,300 mS/cm resulting from metal surface preparation by acidification and subsequent neutralization [29]
Irrigation drain waters reported from Nevada range as high as 30,000 μS/cm, 3.0‰ SO_4^{2-}, and 11.2‰ Cl^- [34]

A producer of Vitamin C discharges effluent containing conduc- tivity levels averages 15,000 μS/cm to 45,000 μS/cm [29]

A manufacturer of polyphenylene sulfate reported to discharge approximately 12‰ Cl^- [29]
Product recovery during production of various chemicals results in sodium- and calcium-dominated waste streams ranging from 1 to 10‰ [1]
Groundwater remediation systems have been observed with TDS levels ranging from 1 to 15‰, depending on water source [35]
Remediation of groundwater may yield wastewaters that are vir- tually void of ions [36]
Typical values for TDS concentrations in domestic wastewaters have been characterized as 850 mg/L (strong), 500 mg/L (medium), and 250 mg/L (weak) [37]

Combustive/evaporative concen- tration

Combustion of oil, wood products, and coal often yields wastewaters with high mineral ash and other residues
Boiler bottoms and blow downs Cooler tower/recycle

Electric generating utilities have been commonly observed with effluents that range in conductivity values from 1,000 to 3,700 μS/cm [29]

IDENTIFICATION OF ION IMBALANCE AS A SOURCE OF WET IN TOXICITY IDENTIFICATION

A variety of approaches can be used to determine if salinity or ion imbalance is responsible for wastewater toxicity. Initial insight can be obtained by determining the salinity/conductiv- ity of the wastewater. Although most of the toxicological data is presented in parts per million or milligrams per liter, con- ductivity is the most common assessment of general ionic concentrations in WET. As a general screening tool, if the conductivity of a freshwater effluent is above 2,000 μS/cm, the concentration of dissolved solids can be high enough to adversely affect freshwater test species [3]. Correlation be-

tween wastewater toxicity and TDS levels may also indicate that ion imbalance may be responsible for test organism mor- tality. Additional information can be obtained by conducting the U.S. EPA Phase I Toxicity Identification Evaluation (TIE) [15–18]. If the results of the Phase I TIE manipulations on the wastewater with relatively high conductivity indicate that toxicity cannot be eliminated or significantly reduced by any of the treatment steps, the concentration of ions in the effluent may be responsible for toxicity and should be further evalu- ated.

The evaluation process may include a determination of spe- cific inorganic ion concentrations in the wastewater before and

after each step of the Phase I TIE protocol. Specific ions im- portant to freshwater species include Ca^+, K^+, Mg^{2+}, Na^+, Cl^-, HCO_3^-, and SO_4^{2-} [3]. Ions important to marine organisms include the above ions and also Br^-, $B_4O_7^-$, and Sr^{2+} [3]. Measured wastewater concentrations of the ions can be com- pared to literature or to laboratory-derived toxic effect con- centrations to determine if ion concentrations are above toxic effect concentrations. One can also assess the cause(s) of tox- icity of an effluent by evaluating the concentration of the major ions that compose the effluent's TDS, using the freshwater matrices salinity/toxicity program model previously discussed [8]. Although a marine model similar to the freshwater ma- trices salinity/toxicity program presently does not exist, several studies presented earlier may assist investigators in the TIE assessment of the major ions on estuarine and marine organ- isms until this model is developed [1,2,7,9,10–13].

Chemical fractionation schemes can provide additional in- formation on whether inorganic toxic constituents are con- tributing to WET toxicity. Chromatographic columns contain- ing various exchange resins (i.e., cation and anion exchange resins, activated carbon, and silica gel) have been successfully used by a variety of researchers [19–24] to separate wastewater into its inorganic and organic components. The results of the toxicological tests and chemical analyses performed on the wastewater before and after resin treatment are used to deter- mine whether a correlation exists between the removal or re- duction of select chemical constituents and wastewater tox- icity.

A cation exchange methodology currently under develop- ment by Burgess et al. (unpublished data) may also be helpful in determining if inorganic salts are playing a role in toxicity. The methodology employs cation exchange resin (Supelco LC- WCX column, Supelco, Bellefonte, PA, USA or Alltech's Ex- tract-Clean IC-Chelate column, Alltech, Deerfield, IL, USA) to characterize and identify metal toxicity in marine TIEs. Recently, Tucker et al. (unpublished data) used calcium pre- cipitation via sodium sulfate and ion exchange resin (Rohm & Haas Amberlite IR-120Plus cation exchange resin, West Hill, ON, Canada) to determine if marine discharge toxicity is due to an imbalance of essential ions such as calcium or potassium.

Synthetic effluents, which mimic the major ions in the ef- fluent under evaluation, have also proven useful for the as- sessment of TDS toxicity associated with WET toxicity. Al- iquots of the whole effluent are mixed with various amounts of the synthetic effluent (based upon chemical evaluation of the effluent for the major ions) in an effort to determine if the concentration of the measured anions and cations cause tox- icity to the test organism [22]. For example, treatment groups might include the following proportions: 100% effluent from the facility under evaluation; 75% effluent and 25% synthetic effluent; 50% each of effluent and synthetic effluent; 25% effluent and 75% synthetic effluent; and 100% synthetic ef- fluent. The hypothesis of this procedure is that if the effluent is diluted with various amounts of synthetic effluent that con- tain only the salts found in the effluent, then any unknown toxicants potentially in the effluent will also be diluted, re- sulting in a lessened acute or chronic toxicity response of the test organism [22]. However, if TDS were the toxicants of concern in the wastewater, the corresponding acute or chronic toxicity responses would be similar. To help ensure appropriate interpretation of test results, it is important that the test matrix in the synthetic tests be as similar as possible to the natural

effluent (e.g., effluent with toxicity removed, intake water). The above approaches, coupled with test results for synthetic effluents containing suspected toxicants, can be used in a weight-of-evidence approach to ascertain the role of ion im- balance in wastewater toxicity.

The selection of the species to be used in the initial phase of the TIE should be based upon factors not necessarily the same as those used in the permitting process. For example, for effluents that may have high TDS concentrations, it may be more effective to select a species that is less sensitive to ion concentration, although still considered sensitive to a ma- jority of other toxicants that may be in the effluent, in addition to the test species that triggered the toxic result. Selecting a species that is less sensitive to TDS will allow the investigator to assess if other potential toxicants are present in the effluent. It should be cautioned that organisms that are better ion reg- ulators might also be more tolerant of other divalent cations (e.g., heavy metals). Because the original organisms that dem- onstrated toxicity would not be used in the above scenario, it may be necessary to also perform the following studies with the compliance species during the latter stages of the TRE should chemical characterization of the effluent indicate the potential for metal toxicity.

There is a significant difference in the sensitivities of fresh- water organisms to TDS, and that difference can often provide useful information for the successful performance of the TIE [22–24]. For example, *C. dubia* [11] and *Daphnia pulex* have similar sensitivity to sodium chloride, with 48-h LC50s of 2.4‰ [22]. However, the 48-h LC50 for *D. magna* is approx- imately double that of the other two cladocerans [25]. The most commonly tested freshwater fish, *P. promelas,* is even less sensitive to sodium chloride, with a 96-h LC50 of 10.8‰ [25]. This relative sensitivity pattern is useful when conducting multiple-species testing on an effluent with high concentrations of TDS [22]. If the LC50 for *P. promelas* is lower than the *C. dubia* LC50, it is probably safe to rule out TDS as the dominant toxicants. If the situation is reversed, these results may indicate that TDS may be major toxicants in the effluent. For certain situations, it may even be useful to use an estuarine organism such as *M. bahia* or *C. variegatus* as part of the TIE for highly saline effluents in order to help factor out the effects of the high TDS concentration in the effluent while still as- sessing the role of the other toxicants. Although the examples presented above are for acute toxicity, the same experimental design logic can be made for chronic TIEs.

Although the selection of the species to match the goal and objective of the investigative aspect of the TIE is strongly encouraged in the U.S. EPA TIE procedures [16–18], the latter phases of the TIE (e.g., Phase III [15]) should use the permitted species that initially indicated a potential toxicity problem in the effluent.

REGULATORY APPROACHES TO ADDRESS IONRELATED TOXICITY

Two case studies are presented to illustrate approaches that have been successfully used to identify when high concentra- tions of TDS or imbalances of the molar ratios are the major toxicants observed in the effluent. These case studies have been used during application of WET as part of the NPDES permitting process. Recommendation of potential regulatory approaches to address related toxicity observed in WET tox- icity is also provided.

Case study 1

One of the characteristic features of this chemical manu- facturing facility wastewater was its very high conductivity, which ranged from approximately 10,000 to 25,000 μS/cm [20]. Major components contributing to the high conductivity were Na^+ (1,020 mg/L), Ca^{2+} (3,000 mg/L), and Cl^- (7,310 mg/L). Regulatory toxicity tests with *P. promelas* indicated that the 96-h LC50 value for the wastewater typically ranged from 45 to 80% effluent. Because the water was very saline, it was speculated that salinity could either be directly or in- directly responsible for toxicity due to osmotic stress.

In order to determine if salts were likely responsible for toxicity, two wastewater sources were evaluated for their acute toxicity to the fathead minnow. The first effluent, designated as natural effluent, was obtained directly from the manufac- turing site discharge point. The second effluent, designated as synthetic effluent, was prepared by adding sodium chloride and calcium chloride to well water so that the resulting con- centrations of Na^+, Ca^{2+}, and Cl^- were identical to those found in the natural effluent (i.e., Na^+, 1,020 mg/L; Ca^{2+}, 3,000 mg/ L; and Cl^-, 7,310 mg/L). Fathead minnow acute toxicity tests were performed on each of these samples.

Although the investigation initially focused on Na^+, Ca^{2+}, and Cl^- as potential toxic components, it was discovered that high Ca^{2+} levels were responsible for the toxicity of some manufacturing site wastewater [1]. Specifically, it was deter- mined that wastewater having a Ca^{2+} to Na^+ ratio of 15:1 exhibited high test organism mortality, whereas wastewater with a Ca^{2+} to Na^+ ratio of 1:20 had low test organism mor- tality. Calcium acute effect levels for several aquatic species were determined to be as follows: *D. pulex* 48-h EC50, 499 mg/L; *M. bahia* 96-h LC50, 927 mg/L; *P. promelas* 96-h LC50, 266 mg/L. Based upon this test data, Ca^{2+} levels in the natural effluent were determined to be above acute effect levels for the fathead minnow. Consequently, Ca^{2+} levels were mea- sured in the toxicity test solutions to determine if a correlation existed between Ca^{2+} levels and test organism mortalities [20]. Chemical fractionation tests were performed on the waste- water to determine if the specific factors responsible for waste- water toxicity could be isolated and identified. Samples of wastewater were passed through four separate resin treatments (i.e., granular activated carbon, cation exchange resin, anion exchange resin, and cation exchange resin followed by anion exchange resin). Total organic carbon and inductively coupled plasma analyses were performed on wastewater samples before and after resin treatment.

The results of the natural and synthetic effluent toxicity tests showed a strong correlation between toxicity and Na^+, Ca^{2+}, and Cl^- concentrations. Analysis of test organism mor- tality and corresponding Ca^{2+} concentrations in the test con- centrations revealed that the concentration of Ca^{2+} at the *P. promelas* 96-h LC50 level (i.e., 2,400 mg/L) was similar to the 96-h LC50 reported by Dorn and Rodgers [1] for *C. var- iegatus* (2,766 mg/L). Chemical analyses also revealed that the concentrations of Ca^{2+} and Cl^- in the undiluted natural wastewater were two to four times higher than the 48-h EC50 value. No test organism mortalities were observed in natural or synthetic wastewater treated sequentially with cation and anion exchange resins. Confirmation of Ca^{2+} and Cl^- as the primary toxicants in the wastewater was determined via the use of a synthetic effluent containing only Na^+, Ca^{2+}, and Cl^-. The dose response curves for both wastewater sources were very similar.

An evaluation of potential sources of Ca^{2+} and Cl^- at the manufacturing facility revealed that most of the Ca^{2+} in the wastewater came from lime (calcium oxide) that was used for product recovery and plant influent neutralization. In order to reduce Ca^{2+} levels in the discharge, the manufacturing facility implemented a caustic conversion project that substituted so- dium hydroxide for lime in product recovery processes and wastewater neutralization. This resulted in the formation of sodium chloride in the discharge, which is significantly less toxic than calcium chloride. The toxicity of the effluent was eliminated as a result of this change.

Case study 2

A TIE for a specialty chemical manufacturing facility was performed using the synthetic effluent method described earlier [22]. Phase I TIE tests performed over several months indi- cated that acute toxicity was not removed by any of the frac- tionating procedures. An ion scan was performed to determine the major anions and cations in the effluent. The analysis in- dicated that Na^+ and Cl^- were the major ions in the effluent. An aliquot of whole effluent from this facility was diluted with an increasing percentage of deionized water and adjusted to the original salinity concentration using a stock synthetic ef- fluent of sodium chloride. The resulting 48-h LC50s for *C. dubia* ranged from 61.2 to 66.0% effluent.

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A second effluent sample from this facility was evaluated in more detail for the major ions. This second sample was immediately analyzed for anions, including HCO^-, CO^{2-}, Cl^-, F^-, NO^-, NO^-, PO^{-3}, and SO^{2-}, as well as cations, including Ca^{2+}, Mg^{2+}, K^+, and Na^+. A synthetic effluent was prepared to mimic the major anions and cations identified in this effluent sample. Using a similar experimental design as the previous experiments, the effluent and synthetic effluent were combined in varying proportions. The resulting *C. dubia* 48-h LC50s were virtually the same, ranging from 58.7 to 61.2% effluent.

These results indicated that the toxicity of this effluent was caused by TDS, as evidenced by the similar acute toxicity values for all of the tests. The most likely major toxicants causing toxicity in the effluent were Na^+ and Cl^-. If the toxicity of the effluent had decreased as the synthetic effluent mixture increased in concentration, these results would have indicated that something other than the TDS in the effluent caused the acute toxicity. Because the TDS of the effluent was determined to be the major toxicant and the effluent was discharged to an inland receiving stream with naturally high salinity, the reg- ulatory situation for this discharge was that *C. dubia* was not reflective of the receiving system; only *P. promelas* was used in future WET testing.

The potential for major ion toxicity should be considered in the management of effluent wastewaters that contain wastes with various ionic strengths and compositions. This issue would be minimal if balancing, removal, or treatment of ions was inexpensive. However, treatment processes, such as re- verse osmosis, ion exchange, or distillation, have high capital equipment, operating and maintenance costs, and energy re- quirements and may not be justified in terms of risk reduction to aquatic biota. Source control and pollution prevention ap- proaches are usually much less costly in capital and in oper- ating and maintenance costs and energy requirements. In ad- dition, as water conservation and reuse become more impor- tant, the problem of concentration of ions in effluents may become exacerbated. Substitution of process chemicals to

modify ion content, although usually the first option, may not prove to be cost effective for many facilities. As proximity to seawater increases, the influence of marine and estuarine water either on the discharge or on the interaction of the discharge with the receiving stream increases. When major ion toxicity is initially suspected, the regulatory review should proceed cautiously.

Current regulatory approaches

Most state-specific water quality standards contain some consideration of effects of high ion content, typically as Cl^-, SO_4^{2-}, hardness, salinity, or TDS. However, numeric TDS cri- teria do not account for ion balance, which should be kept in mind when generic criteria are used for the protection of aquat- ic life.

Various regulatory attempts have been made to address WET and subsequent noncompliance issues with permit lim- itations caused by ion imbalance problems. Some regulatory programs do not make a distinction between different causes of toxicity in applying regulatory control, because they assume that the permittee's sole responsibility is remaining in com- pliance. This approach is consistent with the approach used for most other limited parameters (e.g., specific toxic sub- stances or conventional pollutants). Several state programs have prescribed approaches that require toxicant characteriza- tion within the compliance cycle to establish that ion imbalance is causing toxicity. Organisms less sensitive to ion imbalance have been substituted in the testing process for those indicating noncompliance [26].

Water quality–based solutions and approaches

States may, at their discretion, adopt policies affecting the application and implementation of their criteria. For example, policies concerning mixing zones and water quality standard variances may be adopted with U.S. EPA approval. Following applicable guidance does not always require meeting all water quality criteria within the discharge pipe to protect the integrity of the entire body of water. Recognizing these factors, the U.S. EPA described permitting procedures that would consider ex- posure duration and magnitude as well as frequency; these procedures are to be used in establishing water quality–based limits [27]. As is the case with any other toxicants, biological effects caused by common ions are a matter of exposure (with consideration of magnitude, duration, and frequency). A con- clusion of the Pellston WET conference was: ''WET testing is an effective tool for predicting receiving system impacts when appropriate considerations of exposure are considered'' [28]. The Technical Support Document [27] recommends that wastewater discharge permits be written to protect sensitive aquatic species from toxic effects; therefore, permits must con- sider exposure. The Technical Support Document also de- scribes use of a three-compartment conceptual model to char- acterize exposure conditions in establishing limits for short- term and longer-term exposures, describing mixing zones that allow the discharge to enter the stream, a zone where no acute toxicity is allowed, and a zone outside of which no chronic toxicity is allowed.

Current regulatory problems with NPDES permits derived to protect against the toxicity of discharges to surface waters probably suffer most significantly from lack of, or insufficient consideration of, exposure, such as effluent dilution studies [28]. This is true not only of ion imbalance–related toxicity, but also of other effluent constituents or characteristics. It is

unlikely that the permitting process will be able to selectively deal with controlling the effects of ion imbalance versus effects of other toxic conditions, because of the complexity and timing of identification of toxicants in the permitting cycle and be- cause of relatively understaffed regulatory agencies. It is ques- tionable whether this distinction is desirable in the context of NPDES WET permitting because its intent is protection against measurable adverse effects, regardless of cause.

Case-specific considerations of toxicants in the permit-writ- ing phase may prove feasible but will likely be costly. Making these permit distinctions may also require the development of new operational strategies for NPDES permitting, including development of thorough TIE information prior to issuance of a permit. Several states, such as Texas and Maryland, make this assessment a part of the TRE. If toxicity is observed initially, and if ion imbalance is observed as the causative toxicant, then additional consideration may be applied. Having the flexibility to address issues such as ion imbalances as part of the TRE and as a receiving water assessment will likely reduce the instances in which additional treatment controls are prescribed but risks to the aquatic community are not apparent.

Deficiency of necessary ions must also be acknowledged in the permitting process when adverse effects are observed in WET tests. However, recent decisions in Texas acknowl- edged that adding ions such as Mg^{2+} to chemical effluent dis- charge to remove an ion deficiency was not a prudent regu- latory judgment or a cost-effective solution to effluent toxicity [25]. Several states and regions will allow the addition of hardness as part of the WET tests for very low hardness ef- fluents, particularly when chronic tests are to be performed. Appropriate modeling and application of exposure conditions that occur in the receiving streams represent the best permitting solutions to protect the aquatic community against adverse effects when ion-related toxicity or ion imbalances are sus- pected.

Permitting problems are likely to be encountered for efflu- ents with ion-related toxicity when there is no alteration of the inorganic ion balance or concentration or when that balance is disrupted. In such situations, compliance will be determined by the difference between the effluent ion composition and the receiving water ion composition relative to the test species' tolerance to those differences. In discharge situations where the limit is effluent dominated, the ion imbalance of the effluent has the highest potential to be problematic. States that do not allow mixing and zones of initial dilution are placed in the situation of requiring compliance with NPDES permit require- ments at the end of the pipe and are therefore severely limited in their options for solutions. We recommend that regulatory agencies and permittees alike should consider whether this represents cost-effective environmental management.

Where mixing zones are allowed, two points of compliance in the receiving water body are generally defined by: (1) a zone of allowable acute toxicity, and (2) a zone outside of which no chronic toxicity is acceptable. This should be true whether ion imbalance or absolute ion concentration presents toxic potential. Direct consideration of exposure becomes crit- ical to provide realistic protection of the receiving water and achieving compliance goals.

Toxicity testing methodology applications and considerations

A distinction that seems to become muddled in this regu- latory debate is the line between compliance with WET limits

and further testing to characterize toxicants. The goal of the water quality–based permitting approach is the protection of receiving water biota. The organisms selected for testing and the exposure conditions should be representative of the re- ceiving water conditions. Attempts to segregate ion effects of freshwater/low salinity discharges to estuaries or to the marine environment can be seen in required testing methodologies that dictate balance of salinity or ions between control/diluent and the wastewater being tested. Where the effluent salinity is lower than that tolerated by the test species, then sea salts or hypersaline brine may be added to increase salinity. The regulator should also consider whether some invertebrates and plants are amenable to testing at the high effluent concentra- tion, such as 100% effluent if the test method does not rec- ommend sea salts. Another factor to be considered is the ability of the sea salts added to the effluent to reach chemical equi- librium. In these situations, freshwater test species might be used if the permit limit or trigger is greater than the highest concentration that can be effectively tested as the initial round of testing. However, if toxicity is observed, the discharger must be allowed to evaluate the effluent under the discharge con- ditions that are actually occurring.

The use of additional species to identify toxicants present in a wastewater has previously been discussed as an effective scientific tool. The modification of the permit to test species less sensitive to observed effects circumvents the intent of the analysis. However, species that are not matched to the more applicable discharge conditions are often not predictive of the situation in the receiving waters.

SUMMARY AND RECOMMENDATIONS

Effluent toxicity resulting from inorganic ion imbalance is pervasive in permitted discharges from many industrial pro- cesses and municipal discharges where process streams are concentrated, adjusted, or modified. Procedures using weight- of-evidence approaches to identify ion imbalance toxicity in- clude direct measurement, predictive toxicity models for fresh- water, exchange resins, mock effluent, and ion imbalance test- ing with tolerant/susceptible test species comparisons. Devi- ations in concentration or proportion of the dominant ions usually indicate a potential ion imbalance problem. The ion composition of freshwater varies greatly relative to that of seawater. However, certain cations (Ca^{2+}, Mg^{2+}, Na^{+}, and K^{+}) and anions (HCO_3^-, CO_3^{2-}, SO_4^{2-}, and Cl^-) are typically pres- ent.

Estuarine and brackish waters also pose challenges for many discharge situations. For example, low-salinity effluents entering a higher-salinity receiving stream or, conversely, high- salinity effluents entering a low-salinity receiving water can be problematic to species selection and to the assessment of true toxicity rather than artifactual toxicity. Environmental re- quirements and tolerance of aquatic organisms to salinity or ion composition vary and should be recognized. Ion regulation problems arise when marine organisms are moved rapidly from full-strength seawater to lower salinity. Thus, WET testing requirements for marine and freshwater organisms vary be- cause of different osmoregulatory requirements that must be considered when selecting test species to identify sources of toxicity due to ion imbalance. Groundwater discharges to sur- face waters often pose special cases of ion imbalance because they are not mature water sources such as surface waters; instead, they are often dominated by a few anions and cations. Cost-effective waste treatment control options for a facility

whose effluent is toxic because of TDS or specific ion(s) are scarce at best. However, depending on the discharge situation, TDS toxicity may not be viewed with the same level of concern as other toxicants. These discharge situations often do not require the conservative safety factors that other toxicants do. Regulatory solutions to ion imbalance toxicity when no other toxicants are present may include modifications to the site- specific exposure through discharge modification, use of al- ternative models (e.g., dynamic models), exposure-specific toxicity tests, or alternate mixing zones for TDS or specific ion(s). These approaches may be beneficial, as opposed to those that require potentially expensive or high-energy–using treatment options that are often ineffective treatment controls for the removal or addition of ions. This is especially true for the case of those ions that do not pose human health concerns or significant ecological hazards and that are rapidly diluted or assimilated in the receiving water body.

Additional regulatory or technical solutions may be pos- sible if ions are identified as the only responsible toxicant. In situations where ions and another toxicant (e.g., ammonia) are identified, the initial responsibility should be to address the other toxicant. After that toxicant is effectively dealt with, then the ion-specific toxicity can be addressed. Potential manage- ment and regulatory options that are recommended are con- sideration of waste reduction and pretreatment options, use of appropriate/alternative mixing zones and alternative dilution models (e.g., dynamic modeling), predilution of effluent with receiving waters or other plant process waters prior to final discharge, development of site-specific ion or TDS limits, and use of bioassessment techniques in addition to the WET pro- cedures to further evaluate the discharge situation.

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Exhibit 2 to Consent Order OGC Case No.: 19-1453

Mitigation Project

a. Within 60 days of the Effective Date of the Order, Respondent shall submit, by certified mail, a detailed mitigation project proposal (Proposal) to the Department for evaluation. The Proposal shall include a summary of benefits, proposed schedule for implementation and documentation of the estimated costs which are expected to be incurred to complete the project. These costs shall not include those incurred in developing the proposal or obtaining approval from the Department for the Proposal.

b. If the Department requests additional information or clarification due to a partially incomplete Proposal or requests modifications, Respondent shall submit, by certified mail, all requested additional information, clarification, and modifications within 30 days of receipt of written notice.

c. If upon review of the Proposal, the Department determines that the project cannot be accepted due to a substantially incomplete proposal or due to substantial deficiencies, Respondent shall be notified, in writing, of the reason(s) which prevent the acceptance of the Proposal. Respondent shall correct and redress all the matters at issue and submit, by certified mail, a New Proposal within 60 days of receipt of written notice. If the New Proposal is not approved by the Department, Respondent shall make cash payment of $1,000,000.00 to the Department, within 30 days of Department notice.

e. Within 180 days of obtaining Department approval for the Proposal or in accordance with the approved schedule submitted pursuant to paragraph (a) above, Respondent shall complete the entire project, unless the Department approves a longer time based on the Proposal or New Proposal.

f. During the implementation of the project and thereafter, Respondent shall not post any sign(s) at the site indicating that the reason for the project was anything other than a Department enforcement action.

g. In the event Respondent fails to timely submit any requested information to the Department, fails to complete implementation of the project or otherwise fails to comply with any provision of this paragraph, the entire amount of civil penalties shall be due from the Respondent shall make cash payment of $1,000,000.00 to the Department within 30 days of Department notice.

h. Within 15 days of completing the project, Respondent shall notify the Department, by certified mail, of the project completion and request a verification letter from the Department. Respondent shall submit supporting information verifying that the project was completed in accordance with the approved proposal and documentation showing the actual costs incurred to complete the project. These costs shall not include those incurred in developing the proposal or obtaining approval from the Department for the project.

i. If upon review of the notification of completion, the Department determines that the project cannot be accepted due to a substantially incomplete notification of completion or due to substantial deviations from the approved project, Respondent shall be notified, in writing, of the reason(s) which prevent the acceptance of the project. Respondent shall correct and redress all the matters at issue and submit, by certified mail, a new notification of completion within 60 days of receipt of the Department's notice. If upon review of the new submittal, the Department determines that the project is still incomplete or not in accordance with the approved proposal, Respondent shall pay the Department $1,000,000.00 within 30 days of Department notice.

Exhibit 3 to Consent Order OGC Case No: 19-1453

Salt Ion Composition Study

Goodfellow et al (2000), in the critical review of major ion toxicity in effluents, proposed a strategy for assessing whether ions were the primary toxicant in the effluent. They proposed a series of assessments using a combination of TIE methods and mock effluents experiments to evaluate their impacts. Using these recommendations, Texas DEQ presented the following methods for determining whether dissolved salts were the primary cause of toxicity (TDEQ 2012). They describe that a permittee should use a combination of the following techniques to show that dissolved salts are the primary cause of toxicity:

- Perform measurements of high levels of dissolved salts in the effluent.
- Perform measurements of the ionic component of the dissolved salts.
- Conduct WET tests using an alternative species along with *C. dubia* that is more tolerant of dissolved salts.
- Conduct side-by-side WET tests using the toxic effluent as well as a mock effluent formulated to mimic the ionic composition of the effluent.

Following this approach, we propose to perform a set of experiments and analyses. We will further evaluate the ionic composition of the effluent by performing anionic and cationic scans of the principal salt ions. We will also evaluate the ionic composition of the laboratory's control water, dilution water, and water used for culturing Ceriodaphnia dubia within the laboratory. Using this chemical characterization information, we will create a mock effluent that represents the salt ions in the effluent (without any other potential toxicant(s) in the effluent). A set of chronic toxicity experiments will be performed with *C. dubia*. These chronic tests will be on samples of 100% effluent, 75% effluent/25% mock effluent, 50% effluent/50% mock effluent, 25% effluent/75% mock effluent, and 100% mock effluent. Given the formulation complexity of creating the mock effluent, we will be using one sample for the entire duration of the chronic testing, rather than the three samples used for regular compliance testing. If the toxicity is the similar in all the chronic toxicity tests and do not show a noticeable trend based on the mock effluent assessments, this indicates that the dissolved salts are the principal toxicant and not another unidentified toxicant. Additional to the mock effluent study, we will also perform a side-by-side chronic exposure assessment with an alternative species that is less sensitive to dissolved salts, but similarly sensitive to other toxicant(s) that may be in the effluent.

The evaluation of all these additional studies will provide a weight-of-evidence as to the role of dissolved salts in the effluent and whether an additional toxicant(s) other than dissolved salts are in the effluent.

Laboratory performance, qualifications and experience for multiple bioassay labs are being evaluated to determine their ability to meet the pertinent requirements in EPA-821-R-02-013 and the capabilities to respond immediately by performing a TIE on toxic samples, when appropriate. These evaluations include a desktop review of all laboratory documents, including reports, bench sheets, QA/QC tolerances, etc. This review follows laboratory audits conducted on January 16 and 20, 2020 by an expert in the field of bioassay testing protocols and requirements. Following selection of the most appropriate and qualified laboratory to meet the above requirements, we will be performing chronic toxicity testing on a split sample (provided to the laboratory as two independent blind samples for testing). The validity of the results will be assessed using the following assessment criteria:

The chronic testing will be performed on 5 concentrations (100%, 75%, 50%, 25%, and 12.5% effluent) and a control. This will provide more definitive resolution of the marginally toxic effluent.

If both tests have IC25 ≥ 100% effluent, the sample will be deemed non-toxic.

If both tests have IC25 < 100% effluent, the sample will be deemed toxic.

If one IC25 ≥ 100% and the other IC25 < 100%, the chronic testing will be determined as unverified, since the variability of the test results would be in question because the split samples provided conflicting information. In this instance, the effluent would be re-evaluated with fresh samples within 14 days using the same testing regime.

Toxicity Identification Evaluation

The EPA provides a definition of marginal toxicity as a

very slight (but statistically significant) effect at a dilution that contains a high proportion of effluent (e.g., above 80 percent). For a TIE to successfully identify and confirm toxicants as part of a TRE, toxicity must be present in a sample. Expecting a TIE to immediately follow a single or infrequent event of WET noncompliance is unrealistic (EPA 2001).

Further evaluation of chronic bioassay failure will be conducted using an USEPA chronic Phase I toxicity identification evaluation (TIE). Before conducting the TIE, a screening test will be conducted by the laboratory. The laboratory will confirm the presence of (1) a strongly-linear dose-response, and
(2) a 25% inhibition concentration (IC25) at least one test concentration below the critical effluent dilution (using the revised test concentrations described above). If the toxic signal is not strong, prospects for a successful TIE are diminished and the Phase I TIE will not be conducted. Should the samples be sufficiently toxic, the full suite of TIE manipulation steps will be conducted consistent with USEPA (1991) guidance: Initial toxicity (unaltered effluent); Baseline toxicity (unaltered effluent); pH adjustment (pH 3 and 11); Filtration/pH adjustment (pH 3 and 11); Aeration/pH adjustment (pH 3 and 11); C18 solid phase extraction (SPE)/pH adjustment (pH 3 and 11); Sodium additions; Ethylenediaminetetraacetate (EDTA) additions; and Graduated pH adjustments.

1. The permittee shall comply with the following requirements to evaluate chronic whole effluent toxicity of the discharge from outfall D-003.

   a. Effluent Limitation

      (1) In any routine or additional follow-up test for chronic whole effluent toxicity, the 25 percent inhibition concentration (IC25) for reproduction shall not be less than 100% effluent. [Rules 62-302.530(61) and 62-4.241(1)(b), F.A.C.]

      (2) For acute whole effluent toxicity, the 96-hour LC50 shall not be less than 100% effluent in any test. [Rule 62-302.500(1)(a)4. and 62-4.241(1)(a), F.A.C.]

   b. Monitoring Frequency

      (1) Routine toxicity tests shall be conducted once every month, the first starting within 30 days of the effective date of this Consent Order and lasting for the duration of this Consent Order.

      (2) If a test within the sequence of the consecutive compliant test is deemed invalid based on the acceptance criteria in EPA-821-R-02-013, but is replaced by a repeat valid test initiated within 14 days after the last day of the invalid test, the invalid test will not be counted against the requirement for consecutive compliant valid tests for the purpose of evaluating the number of consecutive compliant tests.

   c. Sampling Requirements

      (1) For each routine test conducted, a total of three flow proportional 24-hour composite samples of final effluent shall be collected and used in accordance with the sampling protocol discussed in EPA-821-R-02-013, Section 8.

      (2) The first sample shall be used to initiate the test. The remaining two samples shall be collected according to the protocol and used as renewal solutions on Day 3 (48 hours) and Day 5 (96 hours) of the test.

      (3) Samples for routine tests shall not be collected on the same day.

   d. Test Requirements

      (1) Routine Tests: All routine tests shall be conducted using a control (0% effluent) and a minimum of five test dilutions: **100%, 75%, 50%, 25%**, and **12.5%** final effluent.

      (2) The permittee shall conduct a daphnid, **Ceriodaphnia dubia**, Survival and Reproduction Test.

      (3) All test species, procedures and quality assurance criteria used shall be in accordance with **Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms**, 4th Edition, EPA-821-R-02-013. Any deviation of the bioassay procedures outlined herein shall be submitted in writing to the Department for review and approval prior to use. In the event the above method is revised, the permittee shall conduct chronic toxicity testing in accordance with the revised method.

      (4) The control water and dilution water shall be moderately hard water as described in EPA-821-R-02-013, Section 7.2.3.

   e. Quality Assurance Requirements

      (1) A standard reference toxicant (SRT) quality assurance (QA) chronic toxicity test shall be conducted with each species used in the required toxicity tests either concurrently or initiated no more than 30 days before the date of each routine or additional follow-up test conducted. Additionally, the SRT test must be conducted concurrently if the test organisms are obtained from outside the test laboratory unless the test organism supplier provides control chart data from at least the last five monthly chronic toxicity tests using the same reference toxicant and test conditions. If the organism supplier provides the required SRT data, the organism supplier's SRT data and the test laboratory's monthly SRT-QA data shall be included in the reports for each companion routine or additional follow-up test required.

(2) If the mortality in the control (0% effluent) exceeds 20% for either species in any test or the "test acceptability criteria" are not met, the test for that species (including the control) shall be invalidated and the test repeated. Test acceptability criteria for each species are defined in EPA-821-R-02-013, Section 13.12 (**Ceriodaphnia dubia**). The repeat test shall begin within 14 days after the last day of the invalid test.

(3) If 100% mortality occurs in all effluent concentrations for either test species prior to the end of any test and the control mortality is less than 20% at that time, the test (including the control) for that species shall be terminated with the conclusion that the test fails and constitutes non-compliance.

(4) Routine tests shall be evaluated for acceptability based on the observed dose-response relationship as required by EPA-821-R-02-013, Section 10.2.6., and the evaluation shall be included with the bioassay laboratory reports.

f. Reporting Requirements

(1) Results from all required tests shall be reported on the Discharge Monitoring Report (DMR) as follows:

(a) Routine Test Results: The calculated IC25 for reproduction for each test species shall be entered on the DMR.

(2) A bioassay laboratory report for each routine test shall be prepared according to EPA-821-R-02-013, Section 10, Report Preparation and Test Review, and uploaded to EzDMR to the Department at the address below within 30 days after the last day of the test.

(3) Data for invalid tests shall be included in the bioassay laboratory report for the repeat test.

(4) The same bioassay data shall not be reported as the results of more than one test.

(5) All bioassay laboratory reports shall be uploaded to EzDMR using the DEP Business Portal at http://www.fldepportal.com/go/

g. Test Failures

(1) A test fails when the test results do not meet the limits in 1.a.(1).
[62-4.241, 62-620.620(3)]