PRELIMINARY REPORT

# USEPA BENCH SCALE WASTEWATER TREATABILITY STUDY
# PULP AND PAPER MILL DISCHARGES
# OF 2378-TCDD and 2378-TCDF

## PROPOSED INTERIM CONTROL MEASURES
## INTERIM NPDES PERMIT STRATEGY

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U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION 5 - ENVIRONMENTAL SCIENCES DIVISION
EASTERN DISTRICT OFFICE
WESTLAKE, OHIO

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# I. INTRODUCTION

The formation of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2378-TCDD) and 2,3,7,8-tetrachlorodibenzofuran (2378-TCDF) in the bleaching of certain kraft pulps and the discharge of 2378-TCDD and 2378-TCDF from certain bleached kraft pulp and paper mills have been well documented (USEPA 1988a). Contamination of native fish collected downstream from a number of pulp and paper mills has also been demonstrated (USEPA 1987). A large-scale cooperative investigative program is underway to determine the formation and discharge of 2378-TCDD and 2378-TCDF at each pulp and paper mill in the United States where bleaching of chemically produced pulps with chlorine and chlorine derivatives occurs (USEPA 1988b). The pulp and paper industry is also conducting independent research directed at the mechanism of formation of 2378-TCDD and 2378-TCDF in pulp bleaching (NCASI 1988a).

From an environmental standpoint, the most effective method to reduce or eliminate wastewater discharges of 2378-TCDD and 2378-TCDF from pulp and paper mills, as well as the discharge of other toxic pollutants, is through process modifications which would eliminate or minimize formation of these materials. The limited information currently available suggests that certain process modifications and bleaching sequences may result in less formation of 2378-TCDD and 2378-TCDF. Each of these technologies results in the use of less elemental chlorine for lignin removal in the first chlorination stage during bleaching of kraft pulps.

The installation of oxygen delignification at a hardwood bleach line at one mill has apparently resulted in less formation of 2378-TCDD and 2378-TCDF (CPI 1987). The industry reports no detectable levels of 2378-TCDD in bleached pulps and effluents from a relatively new softwood mill with oxygen delignification followed by a short bleaching sequence incorporating a high degree of chlorine dioxide substitution in the first bleaching stage (Buckeye Cellulose 1988). Results from the USEPA/Paper Industry Cooperative Dioxin Screening Study, also known as the "five-mill study," showed no detectable levels of 2378-TCDD and relatively low levels of 2378-TCDF in bleached pulp and filtrates from a hardwood bleach line with a $C_DE_OD$ bleaching sequence and 18% chlorine dioxide substitution in the first bleaching stage (USEPA 1988a). Results from the same study demonstrated relatively high rates of formation of 2378-TCDD and 2378-TCDF in another mill with both softwood and hardwood $C_DE_OD$ bleach lines, much higher chlorination rates, and less chlorine dioxide substitution in the first bleaching stages (5-6%). The Swedish Pulp and Paper Research Institute reports that in limited laboratory and full-scale studies the formation of 2378-TCDD and 2378-TCDF was highly dependent upon the charge of chlorine in relation to the lignin content of the unbleached pulp (chlorine ratio). Their work suggests a threshold chlorine ratio of 0.15, below which formation of 2378-TCDD and 2378-TCDF is substantially reduced (STFI 1988). Data from the five-mill study exhibit similar trends (USEPA 1988a). The Expert Committee on Kraft Mill Toxicity, Ontario Ministry of the Environment, estimates reduced organochlorine discharges with increased chlorine dioxide substitution and oxygen delignification (OMOE 1988).

These limited findings suggest that major process modifications such as installation of oxygen delignification systems at existing bleach lines or installation of completely new bleaching sequences may be effective at significantly reducing formation of 2378-TCDD and 2378-TCDF and other toxic pollutants. Also, the degree of chlorination and chlorine dioxide substitution in conventional bleaching lines may significantly affect the formation of 2378-TCDD and 2378-TCDF. Additional research is underway to determine the mechanisms of formation of 2378-TCDD and 2378-TCDF and the process modifications that will be most effective at minimizing or eliminating formation of these compounds. Results from this research are expected during the next few years. Although decisions to install oxygen delignification and other bleaching process modifications have been made for several mills, implementation of process modifications across the industry, if warranted, may be a relatively long-term proposition.

Human health risks associated with formation of 2378-TCDD and 2378-TCDF in pulp and paper mills and the distribution of these materials in the environment through contamination of paper products, wastewater discharges, and wastewater sludges are currently under review. Although most estimates of potential health risks in terms of excess cancers are relatively low, the highest risks are estimated where contaminated materials are directly ingested (e.g., consumption of contaminated fish) (USEPA 1988c,d); or where 2378-TCDD and 2378-TCDF are transferred from a paper product to a material that is ingested (e.g., transfer from coffee filters to coffee) (A.D. Little 1987, NCASI 1988b). The industry estimates that dermal contact with paper products, normal food contact with paper products, and inhalation of paper dusts by paper mill workers do not result in significant health risks (NCASI 1987a,b; 1988c). Also, risks to surfers and swimmers from exposure to pulp mill effluents in the ocean through dermal adsorption and ingestion were estimated to be quite low (Radian 1988).

Several states have issued fish consumption advisories for segments of streams with paper mill discharges where native fish have been found to be contaminated with 2378-TCDD at levels of concern (Minnesota 1985, Wisconsin 1985, Maine 1985, Louisiana 1987). Other fish consumption advisories are anticipated as additional data become available through USEPA's bioaccumulative pollutant study and monitoring by states. Because of the high incidence of native fish contamination downstream from pulp and paper mills, the U.S. Environmental Protection Agency has determined that, as part of an interim strategy for regulating pulp and paper mills, short-term measures to limit discharges of 2378-TCDD and 2378-TCDF should be taken where possible (USEPA 1988e).

Based upon these considerations, the USEPA Office of Water Regulations and Standards - Industrial Technology Division, in cooperation with Regions 1 and 5, undertook a wastewater characterization and preliminary (bench scale) treatability study at two of the mills from the five-mill study. This study was focused on the distribution of 2378-TCDD and 2378-TCDF in various untreated and treated wastewater samples and possible removal of these materials from the wastewater streams through chemically assisted clarification. While the

industry chose not to participate financially or assist in the design of the study, NCASI did provide assistance to USEPA in identifying current wastewater treatment practices at a number of mills with respect to coagulant and polymer addition for suspended solids control.

This report presents a preliminary summary of the study findings and proposed interim control measures that can be implemented through USEPA's interim regulatory strategy. A more detailed technical report including a full discussion of the field and analytical programs, estimates of sludge generation, and preliminary engineering cost estimates for installation and operation of chemically assisted clarification systems is in preparation.

## II. OBJECTIVES

1. Determine the aqueous and solid phase distribution of 2378-TCDD and 2378-TCDF in untreated and treated wastewaters from two bleached kraft pulp and paper mills with known contamination.

2. Determine whether, or to what extent, 2378-TCDD and 2378-TCDF can be removed from untreated, partially treated, and treated bleached kraft pulp and paper mill wastewaters through chemically assisted clarification.

3. Develop proposed interim (i.e., short-term) control measures for 2378-TCDD and 2378-TCDF that can be implemented through USEPA's interim strategy for regulating dioxins in pulp and paper mills.

## III. FINDINGS AND CONCLUSIONS

1. While some fraction of 2378-TCDD and 2378-TCDF in internal untreated pulp and paper mill bleachery wastewaters (i.e., caustic extraction stage filtrates, combined bleach plant wastewaters) is associated with suspended solids, most of the 2378-TCDD and 2378-TCDF is in the aqueous phase of those wastewaters or in fine colloidal suspensions.

2. After biological treatment at two mills, more than 90% of the 2378-TCDD and 2378-TCDF is associated with suspended solids and subsequently is transferred to the sludge or discharged with the suspended solids in the effluent.

3. Chemically assisted clarification appears to be an effective mechanism for control of 2378-TCDD and 2378-TCDF in internal plant wastewaters. However, as an interim control measure, improved suspended solids controls in existing treatment facilities can more quickly and easily be implemented at less cost.

4. Limited data from the five-mill study, supplemental data obtained as part of this study, and data from Scandinavian studies indicate that chlorine minimization and chlorine dioxide substitution programs in bleacheries can significantly reduce formation of 2378-TCDD and 2378-TCDF.

## IV. STUDY DESIGN

The bench scale wastewater treatability study was conducted in two phases. Phase 1 consisted of screening the effectiveness of various coagulants and polymers for suspended solids removal from samples of caustic extraction stage filtrate, combined bleach plant wastewaters, aeration basin effluent prior to settling, and final effluent, all obtained from Mill E as designated in the five-mill study (see USEPA 1988a). Phase 2 consisted of performing chemically assisted clarification studies on a second set of samples obtained at Mill E and a set of similar samples obtained at Mill A from the five-mill study. Those coagulants and polymers determined to be most effective from the Phase 1 screening program were tested at various dosages and combinations in Phase 2. Caustic extraction stage filtrates and combined bleach plant wastewaters were selected for the treatability study since data from the five-mill study indicated these streams contained the highest levels of 2378-TCDD and 2378-TCDF (USEPA 1988a).

The untreated samples obtained for the Phase 2 program were analyzed for 2378-TCDD and 2378-TCDF in both the aqueous and solid phases as were those treated samples from the bench scale studies exhibiting the best performance with respect to total suspended solids. Samples of gravity settled (in laboratory) aeration basin effluents were analyzed for 2378-TCDD and 2378-TCDF in similar fashion.

## V. FIELD PROGRAM

Samples for the Phase 1 screening program were obtained at Mill E by personnel from the USEPA Region 1 Environmental Services Division (ESD) and E.C. Jordan Co., Portland, Maine. Samples for the Phase 2 program were collected by Region 1 ESD personnel at Mill E and by Region 5 Environmental Sciences Division personnel at Mill A. In each case, four grab samples were collected to prepare an eight-hour composite sample at each sampling site. Samples were shipped or transported to an E.C. Jordan laboratory in Portland, Maine, where the chemically assisted clarification studies were conducted.

Bench scale jar tests were performed to evaluate the effectiveness of various coagulants and polymers for total suspended solids (TSS) and total organic carbon (TOC) removal from the untreated wastewater streams. A six-paddle gang stirrer was used to perform the tests. After flocculation, samples were allowed to settle for 30 minutes, which is less than the detention time in conventional clarification systems for secondary biological solids.

## VI. ANALYTICAL PROGRAM

Analyses for TSS and TOC were performed by E.C. Jordan Co. using USEPA approved analytical methods. Analyses for 2378-TCDD and 2378-TCDF were performed by the Brehm Laboratory - Wright State University using the sample extraction, extract clean-up, and analytical protocol developed for the five-mill study with a modified three-phase column for concurrent isomer-specific determinations of 2378-TCDD and 2378-TCDF. Criteria for identification and quantitation of 2378-TCDD and 2378-TCDF were attained. However, for selected samples with low levels of solid material, the desired analytical detection levels of 0.01 parts per trillion (ppt) were not achieved. This is primarily a function of the requested separate analyses of the solid and aqueous phases of each sample. This problem was particularly evident for the final effluent and treated samples from Mill E. The untreated Phase 2 samples from Mill E had much lower levels of 2378-TCDD and 2378-TCDF than similar samples obtained during the five-mill study. In retrospect, the analytical protocol for aqueous samples used in the five-mill study would have been preferable for attaining lower detection limits. That protocol included combining the extracts from separate extractions of the solid and liquid phases for each sample prior to analysis.

## VII. SUMMARY OF RESULTS

### A. Distribution of 2378-TCDD and 2378-TCDF in Waste ater Samples

Tables 1 and 2 present the analytical results r 2378-TCDD and 2378-TCDF in untreated and partially treated wastewaters and the final effluents from Mills A and E, respectively. Table 3 presents final effluent data for two California pulp mills that have no end-of-pipe treatment and discharge to the ocean. These data show that substantial fractions (60%-70%) of the 2378-TCDD and 2378-TCDF found in caustic extraction stage and combined bleach plant wastewaters are in the aqueous phase. Also, that 35%-50% of the 2378-TCDD and 2378-TCDF found in combined untreated wastewaters from pulping and bleaching are in the aqueous phases of those wastewaters. All of the untreated wastewaters are high in organic content. It is theorized that 2378-TCDD and 2378-TCDF are codissolved with the organic species or are in colloidal suspensions that pass through the fine laboratory filters used to separate the aqueous and solid fractions of each sample.

Of particular importance is the finding that nearly all of the 2378-TCDD and 2378-TCDF in aeration basin mixed liquor suspended solids is found on the suspended solids. It is likely that as other organics are oxidized in biological treatment systems, 2378-TCDD and 2378-TCDF, which are refractory to conventional biological treatment, are taken up with food by the active microorganisms or are adsorbed onto the cell walls. This finding is consistent with the distribution of 2378-TCDD and 2378-TCDF in primary and secondary wastewater treatment sludges observed in the five-mill study (USEPA 1988a) and findings at the Dow Chemical

TABLE 1

MILL A
DISTRIBUTION OF 2378-TCDD AND 2378-TCDF
IN SOLID AND AQUEOUS PHASES

| Wastewater Sample | Sample Number | Phase | 2378-TCDD (%) | 2378-TCDD (pg/gm or ppt) | 2378-TCDF (%) | 2378-TCDF (pg/gm or ppt) |
|---|---|---|---|---|---|---|
| Caustic Extraction Filtrate [TSS 40 mg/L] | DE027804S | Solid | 37 | | 35 | |
| | DE027804W | Aqueous | 63 | | 65 | |
| | | Total | | 0.50 | | 2.15 |
| Combined Bleach Plant [TSS 86 mg/L] | DE027803S | Solid | 32 | | 35 | |
| | DE027803W | Aqueous | 68 | | 65 | |
| | | Total | | 0.20 | | 0.88 |
| Aeration Basin Effluent Prior to Settling [TSS 3700 mg/L] | DE027802S | Solid | >98 | | >99 | |
| | DE027802W | Aqueous | <2 | | <1 | |
| | | Total | | 0.84 - .85 | | 2.63 |
| Final Effluent [TSS 23 mg/L] | DE020801S | Solid | >75 | | 75 | |
| | DE020801W | Aqueous | <25 | | 25 | |
| | | Total | | 0.009-0.012 | | 0.043 |

Notes: (1) Wastewater concentrations of 2378-TCDD and 2378-TCDF are reported as picograms/gram (pg/g) or parts per trillion (ppt).

(2) Where 2378-TCDD or 2378-TCDF were not detected in either the solid phase or aqueous phase of a sample, the distribution between the solid phase and aqueous phase was estimated assuming the analyte was present at the detection level for that fraction of the sample.

(3) Where a range of concentrations is presented, the values reported represent the minimum and maximum concentrations. The minimum concentration was estimated by assuming that the analyte concentration was zero when not detected in a fraction. The maximum concentration was estimated by assuming that the analyte was present at the detection level.

TABLE 2

MILL E
DISTRIBUTION OF 2378-TCDD AND 2378-TCDF
IN SOLID AND AQUEOUS PHASES

| Wastewater Sample | Sample Number | Phase | 2378-TCDD (%) | 2378-TCDD (pg/gm or ppt) | 2378-TCDF (%) | 2378-TCDF (pg/gm or ppt) |
|---|---|---|---|---|---|---|
| Caustic Extraction Filtrate [TSS 240 mg/L] | 88911S | Solid | -- | | 31 | |
| | 88911W | Aqueous | -- | | 69 | |
| | | Total | | ND(0.024) | | 0.069 |
| "B" Bleach Line [TSS 410 mg/L] | 88913S | Solid | -- | | >30 | |
| | 88913W | Aqueous | -- | | <70 | |
| | | Total | | ND(0.014) | | 0.027-0.087 |
| Aeration Basin Effluent Prior to Settling [TSS 840 mg/L] | 88910S | Solid | >92 | | 92 | |
| | 88910W | Aqueous | <8 | | 8 | |
| | | Total | | 0.10 .11 | | 0.74 |
| Final Effluent [TSS 44 mg/L] | 88909S | Solid | -- | | 41 | |
| | 88909W | Aqueous | -- | | 59 | |
| | | Total | | ND(0.044) | | 0.15 |

Notes: (1) Wastewater concentrations of 2378-TCDD and 2378-TCDF are reported as picograms/gram (pg/g) or parts per trillion (ppt).

(2) Where 2378-TCDD or 2378-TCDF were not detected in either the solid phase or aqueous phase of a sample, the distribution between the solid phase and aqueous phase was estimated assuming the analyte was present at the detection level for that fraction of the sample.

(3) Where a range of concentrations is presented, the values reported represent the minimum and maximum concentrations. The minimum concentration was estimated by assuming that the analyte concentration was zero when not detected in a fraction. The maximum concentration was estimated by assuming that the analyte was present at the detection level.

TABLE 3

CALIFORNIA PULP MILLS
DISTRIBUTION OF 2378-TCDD AND 2378-TCDF
IN SOLID AND AQUEOUS PHASES

| Sample | Sample Number | Phase | 2378-TCDD (%) | 2378-TCDD (pg/gm or ppt) | 2378-TCDF (%) | 2378-TCDF (pg/gm or ppt) |
|---|---|---|---|---|---|---|
| **Mill 1** | | | | | | |
| Final Effluent [TSS 120 mg/L] | 1PGH117 | Solid | 64 | | 50 | |
| | | Aqueous | 36 | | 50 | |
| | | Total | | 0.10 | | 0.63 |
| Final Effluent (duplicate) | 2PGH117 | Solid | 66 | | 56 | |
| | | Aqueous | 34 | | 44 | |
| | | Total | | 0.11 | | 0.63 |
| **Mill 2** | | | | | | |
| Final Effluent [TSS 113 mg/L] | 5PGH117 | Solid | 56 | | 45 | |
| | | Aqueous | 44 | | 55 | |
| | | Total | | 0.36 | | 7.59 |
| Final Effluent (duplicate) | 6PGH117 | Solid | 59 | | 48 | |
| | | Aqueous | 41 | | 52 | |
| | | Total | | 0.36 | | 7.42 |

NOTE:
1. Wastewater concentrations for 2378-TCDD and 2378-TCDF are reported as picograms/gram (pg/g) or parts per trillion (ppt).
2. The final effluents at both mills are comprised of essentially untreated wastewaters from pulping and bleaching.
3. Samples collected November 1987 by USEPA-Region 9.
4. Analyses for 2378-TCDD and 2378-TCDF by Brehm Laboratory, Wright State University, Dayton, Ohio (see Section VI for analytical protocol).
5. Analyses for total suspended solids by Kennedy/Jenks/Chilton, San Francisco, California.

Michigan Division plant at Midland, Michigan (USEPA 1986). These data clearly indicate that effective separation of mixed liquor suspended solids and extended aeration basin suspended solids is a key factor in minimizing effluent discharges of 2378-TCDD and 2378-TCDF.

8. Bench Scale Wastewater Treatability Study

The Phase 2 bench scale results are summarized in Tables 4 and 5, for Mills A and E, respectively. As noted earlier, the unexpectedly low levels of 2378-TCDD and 2378-TCDF in Mill E samples and the low levels of solid material in the treated samples confounded the analytical program and rendered analysis of much of the treatability data for that mill inconclusive.

The results for Mill A demonstrate that more than 95% of the 2378-TCDD and 2378-TCDF present in caustic extraction stage and combined bleach plant wastewaters was removed through chemically assisted clarification. However, the substantial dosages of alum (2000 mg/L) or lime (1500 mg/L) required for treatment would result in generation of large quantities of sludge from the suspended solids removed, any alum or lime in excess of saturation, and dissolved and colloidal materials taken out of solution. In order to implement this type of treatment on a full-scale basis, separate clarification and sludge dewatering facilities would be required at most mills. Estimates of the quantities of sludge generated and preliminary engineering cost estimates will be presented in the final technical report for this study.

For the aeration basin mixed liquor suspended solids, use of a non-ionic polymer (dosage 6.25 mg/L) resulted in improved suspended solids effluent quality over laboratory gravity settling (17 mg/L vs. 70 mg/L). The level of suspended solids attained was also somewhat lower than the full-scale treatment system final effluent discharge of 23 mg/L attained without the use of polymers or settling aids, but with longer settling time. 2378-TCDD and 2378-TCDF were removed to less than detectable levels (detection level 0.016 ppt). The actual final effluent concentration of 2378-TCDD was 0.009-0.012 ppt vs. <0.016 ppt in the "treated" aeration basin effluent sample. The 2378-TCDF in the final effluent was 0.043 ppt vs. <0.016 ppt in the polymer-treated aeration basin effluent. Unfortunately, the prescribed analytical protocol precluded attainment of desired lower detection levels in the "treated" samples. Use of the same polymer at a lower dosage for the Mill E aeration basin effluent appeared to be less effective for removal of TSS, 2378-TCDD and 2378-TCDF.

The final effluent at Mill A was subjected to treatment with alum (200 mg/L) and a cationic polymer (4.0 mg/L). Marginal improvement of effluent quality is indicated with respect to TSS and 2378-TCDF. Results for 2378-TCDD are inconclusive due to the analytical issues noted earlier. Treatment of a final effluent in this manner as an interim measure would not be practical because of the need for installation of additional large-scale clarification facilities.

TABLE 4

MILL A
USEPA BENCH SCALE PAPER MILL WASTEWATER TREATABILITY STUDY
PRELIMINARY SUMMARY OF RESULTS

| | Untreated | Treated | % Removal | Treatment |
|---|---|---|---|---|
| **Caustic Extraction** | | | | |
| TSS | 40 | 18 | 55% | Alum (2000 mg/L) |
| TOC | 290 | 150 | 48% | Nalco 7769 (7.5 mg/L) |
| 2378-TCDD | 0.50 | <0.019 | >96% | (anionic) |
| 2378-TCDF | 2.15 | <0.039 | >98% | |
| **Combined Bleach Plant** | | | | |
| TSS | 86 | 19 | 78% | Lime (1500 mg/L) |
| TOC | 190 | 120 | 37% | Calgon WT 2439 |
| 2378-TCDD | 0.20 | <0.010 | >95% | (5.0 mg/L) |
| 2378-TCDF | 0.88 | <0.011 | >98% | (cationic) |
| **Aeration Basin Effluent** | | | | |
| TSS | 3700 | 70 | 98% | Gravity Settling |
| TOC | 400 | 57 | 86% | No Additives |
| 2378-TCDD | 0.84-0.85 | <0.030 | 96% | |
| 2378-TCDF | 2.63 | 0.091 | 96% | |
| **Aeration Basin Effluent** | | | | |
| TSS | 3700 | 17 | >99% | American Cyanamid |
| TOC | 400 | 48 | 88% | 1906 N (6.25 mg/L) |
| 2378-TCDD | 0.84-0.85 | <0.016 | >98% | (non-ionic) |
| 2378-TCDF | 2.63 | <0.016 | >99% | |
| **Final Effluent** | | | | |
| TSS | 23 | 15 | 35% | Alum (200 mg/L) |
| TOC | 48 | 22 | 54% | Calgon 2136 (4.0 mg/L) |
| 2378-TCDD | 0.009-0.012 | <0.043 | -- | (cationic) |
| 2378-TCDF | 0.043 | <0.02 | >53% | |

Notes: 1. Analytical results for total suspended solids (TSS) and total organic carbon (TOC) are reported in mg/L (or ppm); analytical results for 2378-TCDD and 2378-TCDF are reported in pg/gm (or ppt).
2. Analyses for TSS and TOC by E.C. Jordan Co.

TABLE 5

MILL E
USEPA BENCH SCALE PAPER MILL WASTEWATER TREATABILITY STUDY
PRELIMINARY SUMMARY OF RESULTS

| | Untreated | Treated | % Removal | Treatment |
|---|---|---|---|---|
| Caustic Extraction "B" Bleach Line | | | | |
| TSS | 240 | 16 | 93% | Lime (5000 mg/L) |
| TOC | 550 | 250 | 55% | Calgon WT 2439 |
| 2378-TCDD | <0.024 | <0.014 | -- | (3.0 mg/L) |
| 2378-TCDF | 0.069 | <0.009 | >87% | (cationic) |
| Combined "B" Bleach Line | | | | |
| TSS | 410 | 31 | 92% | Lime (6000 mg/L) |
| TOC | 400 | 200 | 50% | Calgon WT 2439 |
| 2378-TCDD | <0.014 | <0.020 | -- | (8.0 mg/L) |
| 2378-TCDF | 0.027-0.087 | <0.008 | >70% | (cationic) |
| Aeration Basin Effluent | | | | |
| TSS | 840 | 160 | 1% | Gravity Settling |
| TOC | 180 | 110 | 9% | No Additives |
| 2378-TCDD | 0.10-0.11 | <0.030 | 1% | |
| 2378-TCDF | 0.74 | 0.13 | 51% | |
| Aeration Basin Effluent | | | | |
| TSS | 840 | 39 | 95% | American Cyanimid |
| TOC | 180 | 97 | 46% | 1906 N (5.0 mg/L) |
| 2378-TCDD | 0.10-0.11 | <0.054 | >48% | (non-ionic) |
| 2378-TCDF | 0.74 | 0.35 | 53% | |
| Final Effluent | | | | |
| TSS | 44 | 21 | 52% | Alum (400 mg/L) |
| TOC | 93 | 32 | 66% | Nalco 7769 (3.5 mg/L) |
| 2378-TCDD | <0.044 | <0.027 | -- | (anionic) |
| 2378-TCDF | 0.15 | <0.011 | >92% | |

Notes: 1. Analytical results for total suspended solids (TSS) and total organic carbon (TOC) are reported in mg/L (or ppm); analytical results for 2378-TCDD and 2378-TCDF are reported in pg/gm (or ppt).
2. Analyses for TSS and TOC by E.C. Jordan Co.

Although installation of facilities for treatment of internal process wastewaters (caustic extraction stage filtrates, combined bleach plant wastewaters) might appear to be an effective method for reducing effluent discharges, the time required for installation, the additional sludge generated, possible difficulties in sludge dewatering, and the relatively high costs argue against this alternative. A more effective interim measure would be improved suspended solids controls in existing treatment facilities. At most pulp and paper mills, facilities for addition of clarification chemicals (e.g., coagulants, polymers) have been installed or can be installed quickly at relatively low cost. Also, the incremental sludge generated can be handled within the capability of existing sludge dewatering facilities at most mills. Similar treatment should also be feasible at many mills with extended aeration wastewater treatment facilities.

C. Other Observations

Tables 6 and 7 present comparisons of results obtained in the five-mill study with results obtained in the treatability study for Mills A and E, respectively. These comparisons illustrate two important points: (1) significantly lower levels of 2378-TCDD and 2378-TCDF were found in bleach plant wastewaters with lower bleaching rates (application of chlorine and chlorine derivatives) in C-stages and across the bleach lines; and (2) significantly lower effluent discharges of 2378-TCDD and 2378-TCDF were observed with improved suspended solids control. Bleach plant operating data for Mill E for the treatability study have been claimed confidential and have not been presented here. Notwithstanding, the degree of bleaching at each mill during the treatability study sampling as measured by unbleached, partially bleached, and fully bleached pulp characteristics (K, CEK, final brightness) was about the same as that measured during the five-mill study. Based upon limited results from other studies (OMOE 1988, USEPA 1988a, STFI 1988), the changes in levels of 2378-TCDD and 2378-TCDF presented in Tables 6 and 7 are believed to be principally related to changes in bleaching practice rather than laboratory, random process, or chemical reaction rate variability. These limited data indicate that an effective interim strategy for minimizing effluent discharges of 2378-TCDD and 2378-TCDF should include both chlorine minimization and improved suspended solids controls.

## TABLE 6

MILL A
FIVE-MILL STUDY AND TREATABILITY STUDY
CHLORINATION PRACTICE AND 2378-TCDD/2378-TCDF LEVELS

| | | Five-Mill Study June 1986 | Treatability Study[1] December 1987 |
|---|---|---|---|
| **Chlorination Practice[2]** | | | |
| C-Stage (softwood) | | 75 lbs/ton | 60 lbs/ton |
| Bleach Line (softwood) | | 235 | 121 |
| C-Stage (hardwood) | | 66 | 41 |
| Bleach Line (hardwood) | | 128 | 75 |
| Bleach Plant[3] | | 162 | 99 |
| **Pulp Characteristics** | | | |
| Softwood | | | |
| K (unbleached) | | 19.6 | 20.3 |
| CEK | | 3.0 | 2.8 |
| Final Brightness | | * | * |
| Hardwood | | | |
| K (unbleached) | | 11.8 | 12.4 |
| CEK | | 2.9 | 3.0 |
| Final Brightness | | * | * |
| **Wastewater** | | | |
| Caustic Extraction Stage (softwood) | 2378-TCDD | 1.8 ppt | 0.50 ppt |
| | 2378-TCDF | 33 | 2.2 |
| Combined Bleach Plant[4] | 2378-TCDD | 0.44 | 0.20 |
| | 2378-TCDF | 7.6 | 0.88 |
| Aeration Basin Effluent Prior to Settling | 2378-TCDD | NA[5] | 0.84-0.85 |
| | 2378-TCDF | NA | 2.6 |
| Final Effluent | 2378-TCDD | 0.12 | 0.009-0.012 |
| | 2378-TCDF | 2.2 | 0.043 |
| | TSS | 104 ppm | 23 ppm |

NOTES:
1. Treatability study results for 2378-TCDD and 2378-TCDF (December 1987) are for native wastewater samples prior to addition of any coagulants or polymers.
2. Chlorination practice expressed as lbs $Cl_2$EQOX/ton of air-dried brownstock pulp (see USEPA 1988a, pp. 92-103).
3. Bleach plant chlorination practice is production weighted for hardwood and softwood bleach lines.
4. Combined bleach plant sample for the five-mill study represents mathematical composite of softwood and hardwood bleach line filtrates. Combined bleach plant sample for treatability study represents field composite sample obtained from combined bleach plant sewer.
5. NA - Not analyzed in five-mill study.

* Data not presented.

TABLE 7

MILL E
FIVE-MILL STUDY AND TREATABILITY STUDY
CHLORINATION PRACTICE AND 2378-TCDD/2378-TCDF LEVELS

| | | Five-Mill Study January 1987 | Treatability Study[1] December 1987 |
|---|---|---|---|
| **Chlorination Practice[2]** | | | |
| $C_D$-Stage (hardwood) | | 98 lbs/ton | ** lbs/ton |
| Bleach Line (hardwood) | | 156 " | ** " |
| Bleach Plant[3] | | 193 " | ** " |
| **Pulp Characteristics** | | | |
| Softwood | | | |
| K (unbleached)(PN) | | 18.8 | ** |
| CEK | | 3.0 | ** |
| Final Brightness | | * | ** |
| Hardwood | | | |
| K (unbleached)(PN) | | 16.7 | ** |
| CEK | | 2.8 | ** |
| Final Brightness | | * | ** |
| **Wastewater** | | | |
| Caustic Extraction Stage (hardwood) | 2378-TCDD | 3.6 ppt | <0.024 ppt |
| | 2378-TCDF | 14 | 0.069 |
| B Bleach Line[4] | 2378-TCDD | 2.1 | <0.014 |
| | 2378-TCDF | 5.80 | 0.027 to <0.087 |
| Combined Bleach Plant[5] | 2378-TCDD | 1.3 | NA[6] |
| | 2378-TCDF | 5.8 | NA |
| Aeration Basin Effluent Prior to Settling | 2378-TCDD | NA[6] | 0.10-0.11 |
| | 2378-TCDF | NA | 0.74 |
| Final Effluent | 2378-TCDD | 0.09 | <0.044 |
| | 2378-TCDF | 0.42 | 0.15 |
| | TSS | 89 ppm | 44 ppm |

NOTES: 1. Treatability study results for 2378-TCDD and 2378-TCDF (December 1987) are for native wastewater samples prior to addition of any coagulants or polymers.
2. Chlorination practice expressed as lbs $Cl_2$EQOX/ton of air-dried brownstock pulp (see USEPA 1988a, pp. 92-103).

TABLE 7

MILL E
FIVE-MILL STUDY AND TREATABILITY STUDY
CHLORINATION PRACTICE AND 2378-TCDD/2378-TCDF LEVELS

NOTES: continued...

3. Chlorination practice for bleach plant is production weighted for hardwood and softwood bleach lines.
4. "B" bleach line sample for treatability study represents field composite of "B" bleach line (hardwood) filtrates. "A" bleach line (softwood) was down during treatability study sampling program. "B" bleach line sample for five-mill study represents mathematical composite of "B" bleach line filtrates.
5. Combined bleach plant sample for five-mill study represents a mathematical composite of "A" bleach line and "B" bleach line filtrates.
6. NA - Not analyzed in five-mill study or not sampled in treatability study.

* Data not presented.
** Bleach plant chemical application and pulp [illegible]a claimed confidential.

## VIII. PROPOSED NPDES PERMIT SPECIAL CONDITIONS

To date, there is only one industrial discharger in the United States with a specific process wastewater discharge limitation for 2378-TCDD or 2378-TCDF. In May 1984, the Michigan Water Resources Commission issued a Final Order of Abatement for the Dow Chemical - Michigan Division plant at Midland, Michigan, and a concurrent NPDES permit (MWRC 1984 a,b). The NPDES permit prohibited the discharge of detectable levels of 2378-TCDD and prohibited the discharge "... at a level which is or may become injurious ... ." The Final Order established a final water-quality based effluent limitation of 0.3 parts per quadrillion (ppq) 2378-TCDD and interim effluent limitations of 50 ppq effective until December 31, 1985, and 10 ppq for the period January 1, 1986 to June 30, 1988. The Final Order also set out a dioxin minimization program including a requirement for an interim control program consisting of the installation of a mixed-media wastewater effluent filter. Upon installation of the effluent filter, the discharge from Dow Chemical has consistently contained less than 10 ppq 2378-TCDD. Recently, the Michigan Water Resources Commission issued an amended Final Order of Abatement and reissued the NPDES permit for Dow Chemical (MWRC 1988 a,b). By these actions the state has revised the final water-quality based effluent limitation from 0.3 ppq to 0.1 ppq 2378-TCDD, and the interim effluent limitation from 10 ppq to 8 ppq. Further dioxin minimization programs and treatability studies have also been required. This approach has resulted in significantly reduced effluent discharge levels and reduced 2378-TCDD levels in native fish collected from the receiving water (USEPA 1988d).

USEPA's interim strategy for regulation of pulp and paper mill dioxin discharges requires development of appropriate best professional judgment (BPJ) best available technology (BAT) effluent limitations and suggests the use of chlorine minimization and improved suspended solids control programs as mechanisms to develop those limitations (USEPA 1988e). Current data indicate that both of these mechanisms are likely to result in reduced effluent discharges of 2378-TCDD and 2378-TCDF. Attachments 1 and 2 present proposed NPDES permit special conditions for chlorine minimization and improved suspended solids control, respectively. These conditions can be applied in permits as they are reissued or as modifications to permits currently in effect. The specific requirements should be tailored to site-specific conditions at each mill, taking into account any recent progress that may have been made through programs initiated by the paper companies.

The treatability data presented in this report are the result of bench scale studies at two pulp and paper mills. While the treatability data and supplemental information presented here provide insight into approaches for interim measures to reduce effluent discharges of 2378-TCDD and 2378-TCDF, the data are too limited to establish interim BPJ BAT NPDES permit effluent limitations for these compounds at all bleach kraft pulp and paper mills. Until such time as more data become available, approaches that establish near-term target levels in the range of 10 ppq (0.01 ppt) are suggested. The data from

the five-mill study, data from other researchers, and the limited data presented here indicate that discharges of 2378-TCDD and 2378-TCDF in the range of 10 ppq are attainable with changes in bleaching practice and improved suspended solids controls. The interim control programs are reasonable interim measures that can be taken to work toward attainment of water-quality based effluent limitations that may be applicable to many pulp and paper mills.

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## ATTACHMENT 1

## Proposed Interim Chlorine Minimization Program

1. Within 30 days from the effective date of this permit, the permittee shall conduct a 72-hour composite sampling program of the following points at each bleach line for the purpose of establishing the rates of formation of 2378-TCDD and 2378-TCDF (lbs/ton of air-dried brownstock pulp) with current bleaching practice:

   Pulps

   a. Brownstock pulp fed to first stage chlorination
   b. Fully bleached pulp after last bleaching stage.

   Wastewaters

   a. Combined bleach plant wastewaters exclusive of noncontact cooling waters, process wastewaters from pulping, chemical recovery, paper machines, utilities or other nonbleach plant sources to the extent possible.

   b. If it is not possible to sample combined bleach plant wastewaters separately, individual bleach line filtrates and other bleach plant wastewaters shall be sampled for the 72-hour sampling period. The permittee may analyze each filtrate separately or prepare a flow-weighted composite sample of filtrates and other bleach plant wastewaters for analyses, insuring that the composite sample is representative in terms of flow and composition of each wastewater.

2. The permittee shall retain all bleach plant operating logs for the period beginning 24 hours prior to initiation of sampling and lasting until 24 hours after completion of sampling. A minimum of 24 grab samples shall be taken at approximate equal-time intervals to make up each 72-hour composite sample.

3. The samples shall be analyzed for 2378-TCDD and 2378-TCDF in accordance with the analytical protocol set out in Appendix C of USEPA/Paper Industry Cooperative Dioxin Screening Study (EPA 440/1-88-025, March 1988) or other equivalent analytical protocol approved by USEPA.

4. Within 90 days from the effective date of this permit, the permittee shall submit a report including the results of the sampling program, the rates of formation of 2378-TCDD and 2378-TCDF, and a description of the bleaching practice followed (e.g., all chemical additions in lbs/ton of air-dried brownstock pulp, Kappa number, CEK, and all data necessary to compute the Kappa factor or chlorine ratio).

5. Beginning 30 days from the effective date of this permit and lasting for 120 days, the permittee shall review bleach plant operating practices and develop operating practices to minimize, to the maximum extent practical without compromising product specifications, the use of elemental chlorine for pulp bleaching. Operating practices may include control of chlorine application, greater substitution of chlorine dioxide for chlorine where possible, improved mixing of bleach chemicals, and other operating practices which would result in lower chlorine use.

6. Within 120 days from the effective date of this permit and lasting until the expiration date, the permittee shall implement those practices that are feasible. Within 150 days from the effective date of this permit, the permittee shall submit a report describing the results of its chlorine minimization efforts.

7. Within 180 days from the effective date of this permit, the permittee shall conduct a second bleach plant monitoring program (see paragraphs 1 to 4 above). The permittee shall submit a report of that monitoring program not later than 240 days from the effective date of this permit.

8. Beginning 12 months after the effective date of this permit and continuing at six-month intervals, the permittee shall submit a report describing any further actions it has taken to minimize chlorine use in pulp bleaching including, but not limited to, changes in operating practices, process modifications, and process substitutions.

9. Based upon the results of this program, the permitting authority may reopen this permit for modification, as appropriate.

ATTACHMENT 2

Proposed Interim Effluent Suspended Solids Minimization Program

1. Within 30 days from the effective date of this permit, the permittee shall initiate laboratory scale screening studies for the purpose of determining what coagulants, polymers, or other materials or additives, may be most effective for minimizing the discharge of total suspended solids from the final effluent. For mills with biological treatment systems including secondary clarifiers, the testing shall be conducted on samples of biological treatment system effluent (aeration basin) prior to addition of any treatment chemicals and prior to settling in secondary clarifiers. For mills with aerated stabilization basins without secondary clarifiers, the testing shall be conducted at the entry or influent to the final settling zones prior to discharge. The testing shall include as a control a gravity settled sample of the secondary clarifier or final setting zone influent including any treatment chemicals currently used at dosages reflecting current practice.

2. Within 30 days from the effective date of this permit, the permittee shall obtain 72-hour composite samples of the final effluent and the biological treatment system effluent (aeration basin) prior to addition of any treatment chemicals and prior to settling in secondary clarifiers or final settling zones as noted above. The aeration basin sample shall be analyzed for 2378-TCDD and 2378-TCDD in both the solid fraction (laboratory filtered) and the liquid fraction in accordance with the analytical protocol set out in Appendix C of USEPA/Paper Industry Cooperative Dioxin Screening Study (EPA 440/1-88-025, March 1988) or equivalent analytical protocol approved by USEPA. The final effluent sample 2378-TCDD and 2378-TCDF analytical results shall be reported on a total sample basis (i.e., separate extraction of solid and liquid fractions, but analysis of combined extracts in accordance with the above analytical protocol).

3. Within 60 days from the effective date of this permit, the permittee shall report the results of the laboratory-scale screening studies and the analyses of the final effluent and aeration basin effluent for 2378-TCDD and 2378-TCDF. The permittee shall also submit a study plan for pilot plant or full-scale verification of the laboratory-scale screening study.

4. Within 75 days from the effective date of this permit, the permittee shall initiate pilot-scale or full-scale total suspended solids wastewater treatability studies for the purpose of validating the results of the laboratory-scale screening studies. If pilot-scale treatability studies are conducted, the studies shall be conducted at a scale that would permit implementation of the results on a full-scale basis.

5. Within 150 days from the effective date of this permit, the permittee shall submit a report of the pilot-scale or full-scale total suspended solids wastewater treatability studies. The report shall include the complete study results; estimates of the increased amounts (volume and mass) of wastewater sludge generated; estimates of expected total suspended solids effluent quality; estimates of the investment and annual costs associated with improved suspended solids controls; and a proposed construction schedule should additional facilities be required.

6. Beginning 150 days after the effective date of this permit and lasting until expiration, the permittee shall implement the most effective interim effluent total suspended solids controls derived from the above studies within the capability of existing wastewater treatment and sludge dewatering facilities. Installation and operation of chemical or other material addition facilities shall be considered within the scope of "existing wastewater treatment," if those facilities are not in place, or if modification of existing chemical or other material addition facilities is required.

7. Based upon the results of this program, the permitting authority may reopen this permit for modification, as appropriate.